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Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Volume I

GL Smith
AP Poloski
MW Rinker
RL Demmer
AW Etchells, III
BE Lewis Jr.
SL Marra

July 2009

Pacific Northwest NATIONAL LABORATORY

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AW Etchells, III[2]
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SL Marra[4]

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Pacific Northwest National Laboratory
Richland, Washington 99352

(1) Idaho National Laboratory, Idaho Falls, Idaho
(2) AWE3 Enterprises, Philadelphia, Pennsylvania
(3) Oak Ridge National Laboratory, Oak Ridge, Tennessee
(4) Savannah River National Laboratory, Aiken, South Carolina

Executive Summary

The U.S. Department of Energy's Office of Engineering and Technology, Office of Environmental Management sponsored a slurry-handling workshop that was coordinated by Pacific Northwest National Laboratory, Savannah River National Laboratory, Oak Ridge National Laboratory, and Idaho National Laboratory with the help of experts from commercial slurry-processing companies. The focus of the workshop was on the retrieval, transport, and mixing of waste slurry. The objectives of the workshop were to: 1) focus on the risks associated with slurry retrieval, mobilization, pipeline plugging, and stratification of slurries in vessels; 2) provide technical education and expert commentary on slurry handling; and 3) generate a technical report documenting slurry transport to include mobilization and transport technologies. The purpose of this report is to document the workshop and its follow-on activities. This report includes aspects of slurry-characterization consensus methods and design of slurry mobilization and transport that will allow for comparable results and system designs among DOE sites.

A critical responsibility of DOE's Office of Environmental Management is the processing of radioactive-waste slurries and designing facilities to handle them for safe long-term disposal. This responsibility is vital to the environment in several states that contain legacy nuclear waste from nuclear weapons programs. This is of keen interest at the Hanford Site in Washington State and the Savannah River Site in South Carolina. Other sites, including Idaho Falls, Idaho, and Oak Ridge, Tennessee, have waste-slurry issues, although the problems are smaller in magnitude.

The presence of solids in waste streams can severely reduce expected throughput and yield, and increase startup time while also increasing investment. The purpose of the Slurry Retrieval, Pipeline Transport & Plugging, and Mixing Workshop and of this report is to share experiences in the nuclear-waste and commercial-process industries to improve the performance of nuclear-waste processing facilities and to avoid problems in new and planned facilities for handling nuclear waste. The handling of two-phase mixtures and, in particular, solid-liquid mixtures (slurries) is not typically taught in undergraduate engineering schools. To this end, the first two days of the workshop were devoted to a tutorial by two experts in the field of slurry handling, particularly transport and mixing.

Several examples of slurry handling from all the primary DOE sites were provided at the workshop, covering retrieval of waste from underground storage tanks to waste treatment, and are documented in this report. This includes specific information for the Hanford Site, the Savannah River Site, the Idaho Site, and the Oak Ridge Site. Presentations touched on the highly variable slurry physical properties, tank waste retrieval, waste transport and waste processing. Many commonalities were also noted. The DOE-Site and Industry-expert presentations highlighted the importance of good chemical and physical characterization of the actual waste being processed, from as-received through all unit operations, to underpin the design basis.

Key Messages from Industry/Academia Experts:

The key messages conveyed by the industry-academia experts, based upon in-depth studies of commercial process industries, are that:

- Handling solids is many times more difficult than handling gases or liquids, particularly if the solids are unrefined as in the minerals processing industry, which is similar to the variety of wastes to be

handled in the DOE complex. Handling slurries can severely reduce expected throughput and yield, and it can greatly increase startup time and cost.

- Innovative process steps may have apparently significantly low investments but are very dangerous to the success of processes.
    - Experience demonstrates that low-cost innovation in process steps places success at high risk.
    - Even the best projects and most successful commercial companies only have successful innovative process steps about one-half the time. Too many innovative steps can lead to disaster, so contingency plans are necessary.
- The keynote industry-academia expert presentations highlighted the importance of good chemical and physical characterization of the actual waste slurries being processed, from as-received through all unit operations, to underpin the design basis.
- A common industrial practice to prevent unrealistic designs is the third-party review, which for maximum success must be scheduled to occur several times along the project timeline.

Three concurrent technical breakout sessions were conducted during the workshop. The subjects were waste retrieval, slurry transport, and slurry processing. Participants were charged with obtaining information on best practices, lessons learned, and successful or unsuccessful efforts in their subject areas. In addition to the general sessions, four special sessions were held. Those special sessions were: mobilization to overcome shear strength, transport related to open-channel flow and critical velocity with viscosity adjustments, slurry processing with pulse-jet mixers, and use of computational fluid dynamics in slurry modeling. The Breakout sessions discussed how to prevent problems on DOE projects and suggested paths forward. The breakout sessions were used to understand and disseminate lessons learned and best practices in the areas of waste slurry retrieval, transport, and mixing.

One of the goals of DOE's Office of Engineering & Technology, EM-20, is to help establish best practices to share across the DOE complex. As discussed at the workshop, use of standardized guidelines is valuable for ensuring comparable results from all actual radioactive-waste and simulant-based tests conducted within the DOE complex. Therefore, three sets of technical guidelines for use by engineers and scientists working on slurry issues were documented and recommended for use within the DOE complex as best-practice guidelines: 1) Waste Slurry Sampling and Measurement Techniques; 2) Simulant Development, Approval, Validation, and Documentation; and 3) Performing Chemical, Physical, and Rheological Properties Measurements . Use of these guidelines will help ensure standardized, comparable results from all tests on simulated or actual waste slurries conducted across the DOE complex.

The participants responded very favorably to the format and value of the slurry handling workshop. They recommended that an ongoing effort to develop and maintain a slurry-handling core competency within the DOE Complex is required. This could be accomplished through a community-of-practice environment.

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1.0 Introduction and Key Results

The U.S. Department of Energy's Office of Engineering and Technology, Office of Environmental Management sponsored a slurry-handling workshop that was coordinated by Pacific Northwest National Laboratory, Savannah River National Laboratory, Oak Ridge National Laboratory, and Idaho National Laboratory with the help of experts from commercial slurry-processing companies. The focus of the workshop was the retrieval, transport, and mixing of waste slurry. The objectives of the workshop were to: 1) focus on the risks associated with slurry retrieval, mobilization, pipeline plugging, and stratification of slurries in vessels; 2) provide technical education and expert commentary on slurry handling; and 3) generate a technical report documenting slurry transport to include mobilization and transport technologies. The workshop environment helped to identify technical vulnerabilities and reduce risks relative to waste-slurry handling and to understand and disseminate lessons learned and best practices in the areas of waste-slurry retrieval, transport, and mixing. The purpose of this report is to document the workshop and its follow-on activities. One of the important follow-on activities stemmed from discussions at the workshop regarding the use of common slurry-handling guidelines to help ensure comparable results from all actual radioactive-waste and simulant-based tests conducted within the DOE complex. Therefore, this report also includes three sets of technical guidelines for use by engineers and scientists working on waste slurry issues which are: 1) Waste Slurry Sampling and Measurement Techniques; 2) Performing Chemical, Physical, and Rheological Properties Measurements; and 3) Simulant Development, Approval, Validation, and Documentation. Use of these guidelines will allow comparable results and system designs among DOE sites.

1.1 Background

A critical responsibility of DOE's Office of Environmental Management (EM) is the design, construction, and operation of equipment and facilities to process legacy radioactive-waste slurries for safe long-term disposal. This responsibility is vital to completing the safe cleanup of the environment in several states that have legacy nuclear waste from nuclear weapons development and production and Government-sponsored nuclear energy research. This is of keen interest at the Hanford Site in Washington State and the Savannah River Site in South Carolina; other sites, including Idaho Falls, Idaho, and Oak Ridge, Tennessee, have waste-slurry concerns, although of less magnitude.

The mission of DOE's Office of Engineering and Technology (EM-20) is "To Identify Vulnerabilities and to Reduce the Technical Risk and Uncertainty of EM Programs and Projects."[1] To further this mission, EM-20 sponsors technical workshops and exchanges on specific areas of technical concern to the DOE complex. Collaboration and sharing of technical expertise and lessons learned between national and international laboratories, the private industry sector, and academia help to reduce risk and technical uncertainties within DOE programs. For these reasons, DOE EM-20 sponsored a slurry-handling workshop to understand and disseminate lessons learned and best practices in the areas of waste-slurry retrieval, transport, and mixing, and to review information on innovative technologies. The slurry-handling workshop provided an opportunity for education, a forum for discussing the current state of slurry-handling technology and identifying technical gaps within the DOE complex and for sharing

[1] U.S. Department of Energy Office of Engineering & Technology, Applied Research and Technology Development and Deployment. March 2008. *Integrated Multi-Year Program Plan (FY 2008 – FY 2010).* Accessed December 8, 2008 at http://www.em.doe.gov/pdfs/Integrated%20MYPP%20Final%20_3-20-08_.pdf

participants' lessons learned experiences, and a means to integrate DOE slurry-handling approaches complex-wide. The purpose of the Slurry Retrieval, Pipeline Transport & Plugging, and Mixing Workshop and this report is to share experiences in the nuclear-waste and commercial-process industries to improve the performance of nuclear-waste processing facilities and to avoid problems in new and planned facilities for handling nuclear waste.

Approximately seventy engineers and scientists from radioactive-waste storage sites in North America and beyond, as well as representatives from commercial firms and academic institutions, attended the workshop. All the major DOE Sites (Hanford, Savannah River, Oak Ridge, Idaho), national laboratories, academia, directed institutions, private industry, and international participants (UK, Canada, and Australia) were represented. The workshop was held over five days from January 14 to 18, 2008, in Orlando, Florida. As handling of two-phase mixtures, and in particular, solid-liquid mixtures (slurries) is not typically taught in undergraduate engineering programs, a two-day short course on specialized slurry mixing and handling was conducted by private-sector experts: Dr. Art Etchells, retired DuPont Fellow, taught about slurry mixing; and Dr. Nigel Heywood, BHR Group, taught about slurry rheology and transport. The subsequent three-day slurry handling workshop included DOE-site presentations on nuclear-waste physical and rheological properties and methods used to retrieve, transport and process the waste, presentations and discussions on best practices and lessons learned from all attendees, and invited talks from eminent private-sector and academic keynote speakers. The workshop also included breakout sessions specific to the retrieval, transport, and processing of nuclear-waste slurries to discuss best practices and lessons learned from all attendees.

The presence of solids in waste streams can severely reduce expected throughput and yield and it can greatly increase plant startup time and cost. To help avoid problems in new and planned facilities for handling nuclear waste, the first two days were devoted to a tutorial by two private-industry experts in transport and mixing of slurries. Dr. Art Etchells covered the following topics: What is Mixing and Why It Matters; Process Result - Mixing Equipment; Mixing Concepts – turbulence, power, flow patterns, settling, blending liquids, liquid-liquid dispersions; Liquid Blending – yield stress materials; Solid-Liquids Mixing – off-bottom and distribution and attrition; and CFD (Computational Fluid Dynamics) to Solve Single Phase and Solid-Liquid Mixing Problems. Dr. Nigel Heywood covered the following topics: Viscosity and the Flow Curve; Flow Curve Measurement; Physical Basis of Dispersion Rheology; Pumps and Pumping; Yield Stress Measurements; Chemical Basis of Dispersion Rheology; Pipeline Design for Non-Settling Slurries; and Pipeline Design for Settling Slurries. The slides used in the short courses are provided in Appendix A.2.

The three-day slurry-handling workshop included the following:

1) DOE-site presentations: several examples of slurry handling, from retrieval of waste from underground storage tanks to waste treatment at all the primary DOE nuclear waste sites, were provided at the workshop and are documented in this report. This includes information specific to the Hanford Site, the Savannah River Site, the Idaho Site, and the Oak Ridge Site (Appendix A.3). Presentations touched on the highly variable slurry physical properties, tank-waste retrieval, waste transport and waste processing; many commonalities were noted. These presentations and discussions were used to get all the attendees on a common basis regarding the issues and challenges facing DOE.

2) Eminent keynote speakers from academia and the private sector to disseminate lessons learned and best practices in the areas of slurry handling (Appendix A.4): Dr. Rick Bockrath, DuPont, presented

lessons learned with regard to third-party reviews of slurry-processing projects and their importance as an industrial best practice; Dr. David Gottschlich, Independent Project Analysis Inc., spoke on the problems with using new technology for solids handling; Prof. David Boger, University of Melbourne, spoke on rheology and surface chemistry of slurries, reviewing several aspects of rheology that have been recognized as problematic by the minerals industry; and Dr. Robert Cooke, Patterson & Cooke, spoke on slurry-retrieval and transport lessons learned from the minerals industry and the design of slurry pipelines.

3) Waste-slurry handling specific breakout sessions covering waste-slurry retrieval, transport, and processing best practices and lessons learned (Appendix A.6). In addition, special breakout sessions were conducted and moderated by keynote speakers. A Retrieval session discussing "Mobilization to Overcome Shear Strength" was moderated by Professor Boger; a Transport session discussing "Open-Channel Flow" and "Critical Velocity w/ Viscosity Adjustment" was moderated by Dr. Cooke; and a Processing session discussing "Pulse-Jet Mixers" was moderated by Dr. Etchells. Based upon the interested generated during the short course, an *ad hoc* Computational Fluid Dynamics (CFD) Modeling session was convened, moderated by Thomas Michener;

4) On the last day of the workshop, selected talks in the areas of retrieval, transport, and processing from private sector, academia, DOE sites, and UK experience (Appendix A.5).

1.2 Key Best Practices and Lessons Learned

Key Messages from Industry/Academia Experts:

The key messages conveyed by the industry-academia experts, based upon in-depth studies of commercial process industries, are:

- Handling solids is many times more difficult than handling gases or liquids, particularly if the solids are unrefined, as in the minerals processing industry, which is similar to the variety of wastes to be handled in the DOE complex (Appendix A.4). Handling slurries can severely reduce expected throughput and yield, and it can greatly increase startup time and cost.
- Innovative process steps may have apparently significantly low investments but are very dangerous to the success of processes (Appendix A.4). [Note: use of the term "innovative" is based upon David Gottschlich's keynote talk[1] (Appendix A.4): "New process technology is any of the following: Process chemistry that has not been used commercially; Incorporation of major equipment that is commercially unproven; New match of feed and equipment."]
  - Experience demonstrates that low-cost innovation in process steps places success at high risk.
  - Even the best projects and most successful commercial companies only have successful innovative process steps about one-half the time. (Appendix A.4) Too many innovative steps can lead to disaster, so contingency plans are necessary.
  - A recent example of innovative process equipment use is pulse-jet mixers which, though demonstrated on some waste feeds, had never been demonstrated on the waste feeds of interest or the scale required (Appendix A.4 and A.6).

[1] See slide number 12 from the presentation by Dr. David A. Gottschlich, Independent Project Analysis, Inc., titled *New Technology and Solids: A Difficult Combination*, January 17, 2008, in Appendix A of this report.

- The keynote industry-academia expert presentations highlighted the importance of good chemical and physical characterization of the actual waste slurries being processed, from as-received through all unit operations, to underpin the design basis. Process design cannot be based on average slurry properties but must cover a robust range of variable properties of slurries.
- A common industrial practice to prevent unrealistic designs is the third-party review, which for maximum success must be scheduled to occur several times along the project timeline. Third-party reviews should involve senior technical people not involved with the project who will be able to speak freely. These should be mainly people from industry rather than only those from the academic community (Appendix A.4).

Three concurrent technical breakout sessions were conducted during the workshop (Chapter 3). The subjects were waste retrieval, slurry transport, and slurry processing. Participants were charged with obtaining information on best practices, lessons learned, and successful or unsuccessful efforts in their subject areas. In addition to the general sessions, four special sessions were held. Those special sessions were: mobilization to overcome shear strength, transport related to open-channel flow and critical velocity with viscosity adjustments, slurry processing with pulse-jet mixers, and use of computational fluid dynamics in slurry modeling. The Breakout sessions discussed how to prevent problems on DOE projects and suggested paths forward. The breakout sessions were used to understand and disseminate lessons learned and best practices in the areas of waste slurry retrieval, transport, and mixing.

Key Messages from Slurry Retrieval Breakout Sessions:

- There are many varying types of waste that need to be retrieved; physical properties can vary significantly even within a given tank. The ability to design and field effective retrieval equipment depends upon knowing and understanding the chemical, physical and rheological properties of the waste slurries.
- Waste retrieval equipment utilized today has little ability to control properties of the resulting slurries that are conveyed out of the tanks.

Key Messages from Slurry Transport Breakout Sessions:

- An aggressive slurry physical and rheological characterization campaign is needed for proper design of slurry-transport systems.
- Laminar-flow regimes should be avoided in the design of slurry pipelines.
- Typical mineral-industry transport velocity correlations, while similar, are not appropriate for the particle property ranges encountered in DOE cleanup applications. Recent data show that correlations are different for Newtonian and non-Newtonian slurries.
  - The mineral-industry correlations focus on particles larger than 100 micrometers while DOE slurries are typically less than 100 micrometers in size with a broad distribution of particle densities. The transport velocity correlation from Saskatchewan Research Council (SRC), led by Dr. R.G. Gillies, is viewed as the best available.
  - The Hanford Waste Treatment Plant (WTP) has recently commissioned a set of pipeline transport tests. Since the WTP expects to process large quantities of non-Newtonian slurries, PNNL testing investigates the deposition-velocity calculations for both Newtonian and non-Newtonian slurries. Results of the PNNL tests indicate that a design approach for critical velocity of both Newtonian

and non-Newtonian fluids should include at least three criteria: 1) the critical deposition boundary, 2) the transitional deposition boundary, and 3) the laminar deposition boundary. These boundaries define the transitions between stable and unstable slurry-flow regimes for systems with non-Newtonian flow properties. Correlations for these boundaries can be found in the draft PNNL report, WTP-RPT-175 Rev A.

Key Messages from Slurry-Processing Breakout Sessions:

- Paper studies are inadequate for slurry processing; thorough cold testing of unit operations can obviate costly upgrades.
- It is crucial to processing success to understand how various processing unit operations affect slurry chemical and physical properties, and to what extent.

Pulse-Jet Mixers (PJMs):

- Pulse-jet mixing is an innovative process technology that was not as fully developed as many believed and, as often happens with innovative processes, needed much more development work and redesign than had been expected. Such failures of innovation are common with slurries in the process industries.
- Equipment manufacturers frequently do not have extensive knowledge of process applications. Such knowledge often gets lost over time with loss of key technical people.
- PJMs are not designed to mix tall tanks unassisted.
- Because they use high velocities, PJMs must be checked experimentally for erosion potential.
- Devices handling raw solids such as tank-farm waste will be subject to a wide variety of feeds and must be designed based on worst-case conditions rather than average properties.
- Early review of key technology assumptions by technical people from the process industries would probably have uncovered the shortcomings of the PJMs; such third-party reviews are common in the process industries.

Key Messages from Slurry Computational Fluid Dynamics (CFD) Modeling Breakout Session:

- There is a role for Computational Fluid Dynamics in slurry handling; however, it must be validated and applied correctly. It has not been proven that CFD is capable of accurate predictions when applied to multi-phase, chemically reacting mixtures with complex rheologies, such as non-Newtonian.
- While current CFD modeling is improving, it can be dangerously misleading if conducted improperly. Current CFD codes do not have all the necessary correct physics, chemistry, and numerical modeling capability to handle multi-phase flow problems and will need to take advantage of high performance computing capabilities to be able to model complex slurry interactions in a timely manner.
- CFD validation against known data is crucial. The ability of any CFD model to predict slurry behavior prior to actual experiments is the true test; blind validation, or in other words, pre-test predictions, must be demonstrated to match test results without any additional data manipulation.
- More research is needed on multiphase-flow turbulence and how to model it.

Best Practices to Share Across the DOE Complex:

One of the goals of DOE's Office of Engineering & Technology, EM-20, is to help establish best practices to share across the DOE complex. As discussed at the workshop, use of standardized guidelines is valuable to ensure comparable results from all actual radioactive waste and simulant-based tests conducted within the DOE complex. Therefore, three sets of technical guidelines for use by engineers and scientists working on slurry issues were documented and recommended for use within the DOE complex as best-practice guidelines: 1) Waste Slurry Sampling and Measurement Techniques; 2) Performing Chemical, Physical, and Rheological Properties Measurements; and 3) Simulant Development, Approval, Validation, and Documentation. Use of these guidelines will help ensure standardized, comparable results from all tests on simulated or actual waste slurries conducted across the DOE complex.

Sampling of high-level waste tanks at DOE sites has been accomplished by several different methods. Obtaining samples of high-level waste created during the reprocessing of spent nuclear fuels presents unique challenges due to the necessity of sampling remotely and the requirements for shielding during handling, transport, and storage of samples. Treatment processes in the tanks often produce multiphase wastes that are heterogeneous and contain significant concentrations of dissolved salts, frequently resulting in the formation of salt-cake and/or crusts. High-level waste tanks may include liquid, slurry and sludge layers as well as these salt-cake and crust layers. Appendix B includes descriptions and additional details about sampling methods and important parameters to consider when designing a sampling plan.

Standardized guidelines for measurement of chemical, physical, and rheological properties are provided in Appendix C; these are useful across the DOE complex and a valuable starting point for a consensus document on property-measurement guidelines. Use of these property-measurement guidelines is essential to ensure standardized, comparable results from all tests on simulated or actual waste slurry conducted across the DOE complex. This document brings together chemical, physical, and rheological property-measurement guidelines for the characterization of both actual and simulated wastes.

A guideline for simulant development, approval, validation, and documentation was developed and is provided in Appendix D of this report; it details the instructions necessary for research and technology project staff to develop, approve, validate, and document simulants for use within a slurry project. The use of this guideline will facilitate a consistent methodology for development, preparation, and validation of simulants for use within DOE-complex projects.

After the slurry-handling workshop, a questionnaire was sent to all participants to gauge the effectiveness of the workshop in helping participants understand and disseminate lessons learned and best practices in the areas of retrieval, transport, and mixing of nuclear-waste slurries. The participants responded very favorably to the format and value of the slurry-handling workshop. The following recommendations come from these interactions:

- An ongoing effort to develop and maintain a slurry-handling core competency within the DOE Complex is required. This could be accomplished through a community-of-practice environment.
- Slurry-handling collaboration between national and international laboratories, private industry, academia, and DOE sites through participation in the technical short course and workshop is valuable and helps to ensure DOE mission success.

- Slurry-handling workshops should be held every two years. These technology-exchange meetings will help disseminate best practices and lessons learned in slurry handling from both DOE-EM- and private-industry-funded applied research and technology development, demonstration, and deployment activities.

2.0 Industrial Experience in Slurry Handling

The keynote talks were opened by Dr. Steven Krahn, the director of Waste Processing for the DOE Office of Engineering and Technology. Dr. Krahn stated that the mission of the Engineering and Technology Program is "to identify vulnerabilities and to reduce the technical risk and uncertainty of Environmental Management programs and projects." Accomplishing this mission entails several activities including:

- Implementation of roadmap initiatives
- Completion of Technology Readiness Assessments (TRAs)
- Completion of external technical reviews
- Review of risk-management plans
- Technical workshops and exchanges.

Dr. Krahn stressed the importance of technical workshops and exchanges and concluded by saying that broader collaboration through technical exchanges, such as this slurry-handling workshop, is needed to ensure mission success. Dr. Krahn's full presentation can be found in Appendix A.1 of this report.

Industry experts at the slurry workshop pointed out that their in-depth experience and study of commercial process industries have shown that handling solids is much more difficult than handling gases or liquids, particularly if the solids are unrefined, which is the case with the variety of wastes required to be handled in the DOE complex. Each of the four experts provided technical insights garnered from years of work with slurries as well as projects with significant technology components. Summaries of each of their discussions are provided below, and their presentation materials can be found in Appendix A.4.

2.1 Projects with High Technology Content – Dr. Richard E. Bockrath

Dr. Bockrath addressed the topic of managing technical difficulties at DuPont. He began by discussing DuPont's long history of commercial innovation. He gave examples of successful and unsuccessful projects at DuPont: the unsuccessful projects often have what is termed "high technology content." Dr. Bockrath provided several indicators for projects with high technology content; several of the indicators apply to DOE-EM cleanup projects involving slurry handling. DuPont has managed the high-technology projects through aggressive use of third-party reviews. A detailed process for third-party reviews identifies, ranks, and tracks technical issues throughout the life of the project. These reviews are performed by internal staff who are not working directly on the project. A formal set of roles, responsibilities, and accountabilities have been established for the review staff. The reviews occur "early and often," with several held before the pre-project authorization stage. This strategy has been used by DuPont over the past 6 years with much success. Dr. Bockrath's full presentation can be found in Appendix A.4.

2.2 Projects with New Technologies and Slurries – Dr. David A. Gottschlich

Dr. David Gottschlich of Independent Project Analysis, Inc. (IPA) spoke about project success when combining new technology with systems involving solids or slurries. IPA has a database of over 12,000 projects ranging in size from $15K to $10 billion including about 150 DOE projects between 1970 and 2000. IPA's database is used to find statistical relationships between the project background and the actual plant performance. Metrics for plant performance include plant startup time and the actual plant throughput relative to plant nameplate capacity. The statistics indicate that the combination of new technology and solids handling results in a substantial reduction in plant throughput and increase in plant startup time. These issues can be exacerbated by inadequate fundamental technical data on the slurries to be processed and the unit operations within each specific process. Dr. Gottschlich stated that these problems may be overcome by a highly capable project team consisting of strong project management, technical direction, and facility operations; the team must have good continuity and communication, and the team roles and responsibilities must be clearly identifiable. Dr. Gottschlich concluded his presentation with a statistical comparison of a DOE project with new technology and solids handling versus a standard project without new technology or solids handling. The $100 million DOE project had a startup time of about 20 months compared to one month for the standard industry project.
Dr. Bockrath's full presentation can be found in Appendix A.4.

2.3 Rheology and Surface Science of Colloids – Dr. David V. Boger

Professor David Boger of the University of Melbourne gave a presentation on the rheology and surface science of colloids. DOE-EM slurry retrieval, transport, and waste-processing applications are all dependent on rheology. Dr. Boger reviewed several aspects of rheology that have been recognized as significant by the minerals industry; he discussed problems with transport and disposal of mineral tailings, then described the critical role of yield stress in determining the best approach for slurry transport. Professor Boger explained several techniques used to measure the yield stress of fluids, including concentric-cylinder rheometry, vane rheometry, and slump tests. Vane rheometry and slump tests are particularly attractive: the vane system can be inserted directly into a large volume of process fluid; the slump test, on the other hand, takes a relatively small amount of material and very simple techniques. Professor Boger referred to his slump test as a "fifty-cent rheometer." He explained that rheological properties vary with the degree of attractive and repulsive forces between the particulates and also with solids concentration, particle diameter, particle shape, aqueous and surface chemistry, and zeta potential. Professor Boger provided several mechanistic relations for each of these effects. Professor Boger's full presentation can be found in Appendix A.4.

2.4 Pipeline Transport in the Minerals Industry – Dr. Robert Cooke

Dr. Robert Cooke of Paterson & Cooke in Denver, CO, discussed pipeline transport in the minerals industry. Dr. Cooke explained how the yield stress of a slurry dictates the equipment and approach taken in pipeline transport. He defined a "slurry" as material having a yield stress of less than 20 Pa, "thickened tailings" as having a yield stress of about 20-100 Pa, and "pastes" and "cakes" as having yield stresses exceeding 100 Pa. Dr. Cooke then provided several examples of pipeline transport in the minerals industry. In one example, he explained that chemistry has a significant effect on the pressure drop

required for pipeline transport. Pressure drop was reduced by nearly half when the pH of a mineral slurry was reduced from 6.6 to 5.6 by adding lime. Dr. Cooke explained that laminar flow should be avoided in the design of slurry pipelines. Several examples were given of pipelines that were blocked during transport of yield-stress slurries in laminar flow. Dr. Cooke explained that laminar-flow transport can be implemented if the pressure gradient is large enough to push particles through the pipe. This usually requires equipment different from that typically used for slurry transport. Understanding the conditions needed for laminar-flow transport is a subject of ongoing research. Dr. Cooke said that an aggressive slurry rheology-characterization campaign is needed for proper design of these systems. Several additional examples were provided of improper design leading to centrifugal pump explosions.
Dr. Cooke's full presentation can be found in Appendix A.4.

3.0 DOE Site Experience with Slurry Handling and Technical Breakout Sessions

3.1 Summary of DOE Site Discussions

Staff from each DOE site provided presentations regarding recent and ongoing activities in waste retrieval, slurry transport, and slurry processing. Their presentations are provided in Appendix A.3.

3.2 Technical Breakout Sessions

Three concurrent technical breakout sessions were conducted during the workshop. The subjects were waste retrieval, slurry transport, and slurry processing. Participants were charged with obtaining information on best practices, lessons learned, and successful or unsuccessful efforts in their subject areas. A summary of each of the sessions is provided in this section; detailed information is found in Appendix A.6.

In addition to the general sessions, four special sessions were held. A session on mobilization to overcome shear strength was moderated by David Boger. A session on transport related to open-channel flow and critical velocity with viscosity adjustments was moderated by Robert Cooke. A session on slurry processing with pulse-jet mixers was moderated by Dr. Arthur W. Etchells of AWE3 Enterprises. A session on computational fluid dynamics was moderated by Thomas E. Michener of Pacific Northwest National Laboratory. These four sessions are all described in Section 3.3, "Special Breakout Sessions."

3.2.1 Waste Retrieval

The waste-retrieval discussions were focused on DOE-EM cleanup projects at each site where slurry retrieval was used in the past or will be used in the future. Detailed information and the input provided by the sites can be found in Appendix A.6.

The objective of the first morning's discussion was to define success as it pertains to waste retrieval. It was deemed important to discuss this as we work toward lessons learned from both successful and unsuccessful efforts. The main criterion for success in waste retrieval was defined as regulatory agreement to close a tank or tanks; however, it does not adequately define what degree of cleanliness is sufficient. Another mark of retrieval success is the ability to reuse the tank after it has been emptied; an example of this was Tank 48 at Savannah River. While safety is always paramount in all of our activities, it was concluded that we need to state that success includes the ability to operate safely in the tank farms with no harm to the workers or the environment during operations. Finally, ensuring that we do not create new waste streams or issues resulting from our operations is another component of success.

The group also discussed important technology and attributes that need to be considered in all retrieval operations. The 2000-2001 federal facility agreement requires that the Hanford Site contractors perform "hot demonstrations" of new technologies for cleaning out tanks as well as determine whether new technologies were capable of meeting retrieval needs at the site. Waste in different tanks has different properties; one technology does not suit all needs. In many cases, the physical and chemical

properties of the waste are not well understood prior to retrieval operations; it is important to gain this understanding. The retrieval discussions were centered on various functional aspects of retrieval, including bulk mixing, heel retrieval, transfer, and characterization and monitoring.

Mixing – At Savannah River, bulk mixing is achieved by the use of slurry pumps, including the standard Lawrence pump, the quad-volute pump, the advanced-design mixer pump, and the submersible mixer pump. In general, all worked for the applications used, but some modifications were necessary. At Hanford, the use of standard mixer pumps is the baseline, and the only recent activity with mixer pumps was the mixing in Tank SY-101 several years ago. Multiple mixer pumps were also used at the West Valley Site in New York to remove the heel from their tanks after bulk mixing was completed; this mixing required large volumes of water. In the United Kingdom, pulse-jet mixers have been used for waste streams over the past twenty years. At Oak Ridge, Tennessee, pulsed-jet mixers were used in their horizontal (W and C) tanks as well as the capacity-increase tanks. Russian-style mixers were also deployed in the Gunite tanks at Oak Ridge as a demonstration of other technologies.

Sluicing – Sluicing has been a method of mixing and retrieval through many deployments at Savannah River and Hanford. It was used extensively at the Savannah River Site (SRS) during the 1970s and 1980s. At Hanford, 20-30 single-shell tanks have been sluiced over the years. Most recently, there were several campaigns to sluice tank C-106 to remove the waste and the heel. Sluicing was used at the Fernald site in Ohio within the past 10 years to remove the waste from silos, and in some of the tanks at Oak Ridge.

Agitator-Based Mixers – These types of mixers have been used recently at SRS during extensive testing as well as removal of the zeolite heel in Tank 19. Oak Ridge also used one of these mixers. The results varied, but in general agitator-based mixers were not as effective as mixer pumps.

Air Sparging – Air sparging was used at Hanford via air-lift circulators for agitation but not retrieval. Spargers were used in the UK for waste homogenization. Oak Ridge deployed a variant of air sparging with pulsed-air mixers. In all cases, this technology is not viewed as a strong candidate for waste retrieval.

Heel / Residual Material Removal – Many methods have been tested, demonstrated, or used with moderate degrees of success. At Oak Ridge, a robotic arm and a remote crawler have been utilized in tandem to remove the waste from the Gunite tanks. This was highly successful, although the cost would likely be prohibitive for use at other sites. Currently at Hanford, a simple articulated arm with a vacuum retrieval system is being used. A crawler also is being used, but with limited success. A high-pressure, low-flow-rate Salt Mantis has been deployed in one tank at Hanford and SRS is considering their use in Tanks 18 and 19. At SRS, a wall crawler to be deployed in the annulus is being developed to remove salt nodules. In the UK, an arm-based and crawler-based system was deployed in a tank and in a waste basin. A common method for heel retrieval that has been deployed at several sites is the use of high-pressure water nozzles and water lances. High-pressure systems were utilized at K Basin at Hanford. A borehole miner was used at Oak Ridge to remove the heels from horizontal tanks at the Old Hydrofracture Facility. Another unique method for heel removal in the UK involves the use of ultrasonic technology, although it was not clear whether it has been deployed. Chemical cleaning has been used successfully in Tank 16 at SRS, and more chemical cleaning deployments are planned. The Russians have also utilized chemicals for cleaning their tanks. They have provided technical information to SRS regarding the chemical cleaning processes they use. At Hanford, high-molarity caustic has been utilized for heel removal, and at

Idaho, acid cleaning was used in stainless steel waste tanks. In the UK, spray washing has been a popular technique for removing waste from the floors and sidewalls of tanks. At Oak Ridge, a Gunite scarifying end effector was utilized to remove contaminants from the tanks. At Idaho, a sprayball system was used to get around the coils inside their tanks.

Transfer Out of Tanks – Waste is usually transferred out of the tanks with pumps; each site has significant experience with pumps. Generally, commercially available pumps are used, although some radial pumps were deployed at SRS. Pumps that have been used typically include rotodynamic (centrifugal and turbine) pumps, positive-displacement pumps, fluidic pumps, and pneumatic pumps. There was some discussion on the development of mechanical methods, such as augers and clamshells, but those were not viewed as acceptable methods of transfer.

Characterization & Monitoring – One of the primary methods of monitoring is the use of still and video camera systems. These are essential to retrieval operations but are problematic. While some radiation-hardened cameras have been developed, they are expensive; it may be more cost effective to use commercial systems and replace them regularly. Volumetric measurement, or mapping, is a very important aspect of retrieval; it helps in determining how effective retrieval operations have been. There are many volumetric mapping techniques, including tomography, volume displacement, mass balance, laser range finders, structured-light scanning, profilometry, sludge sounding, visual inspection based upon known items in the tanks, and radar systems.

At Hanford, it is more useful to measure waste through volumetric measurements than mass balance. The use of photos is extremely important in working with the regulators. Hanford is currently using video scaling techniques and beginning to use gamma cameras. At SRS, the volume and radionuclide content of tanks are tied to the performance assessment. Visual techniques with landmarks such as cooling coils and plates on the floor are utilized. Oak Ridge and Fernald also use landmarks to monitor remaining tank-waste volume.

Another characterization tool being used is the turbidity meter, a suspended-solids profiler which is in use at SRS for transfer of supernate rather than slurry, but was ineffective at Hanford. Various densitometers based upon ultrasound or coriolis effects are being used at Hanford.

Fernald used microwave technologies and gamma attenuation for radon control.

SRS does not have an in-tank characterization and monitoring system.

Radiation monitoring at Hanford includes a prototypical gamma-assay system deployed to evaluate mixing, although there were problems when this was deployed during the AZ-101 mixing tests. There are two in-line probes on a transfer line at Hanford for real-time cesium estimates. There was a gamma scan for salt on the tank walls at SRS.

Physical Samples (both in-situ & grab samples): at Hanford, there is no in-situ sampling. All samples are removed from the tanks, and they have used many devices including augers, clamshells, and the “alligator” and “aardvark” dip samplers for supernate and core samples before retrieval. Hanford has a Raman probe that has not yet been deployed. At SRS, a core-type sampler was utilized in Tank 5. In

Tanks 18/19, a clamshell-type sampler was deployed on their crawler. Dip samples are common including 3-, 5-, and 25-liter samplers, but these are for processing and not retrieval. SRS is also working on a Raman probe.

3.2.2 Slurry Transport

The discussions were focused on DOE-EM cleanup projects at each site where slurry transport was used in the past or will be used in the future. Notes on this information exchange were taken in the form of meeting minutes and are provided in Appendix A.6. During this discussion, industry experts made the following important points:

- For proper design of slurry transport systems, an aggressive campaign of slurry physical and rheological characterization is needed.
- Laminar-flow regimes should be avoided in the design of slurry pipelines.
- Typical mineral-industry correlations do not cover the particle-property ranges encountered in DOE cleanup applications. The mineral-industry correlations focus on particles larger than 100 micrometers while DOE slurries are typically less than 100 micrometers with a broad distribution of particle densities. The correlation from Saskatchewan Research Council (SRC), led by Dr. R.G. Gillies, is viewed as the best available; attendees of their course receive software and training.

In addition to the discussion component of the breakout session, the group had knowledge of several past and ongoing projects related to several DOE sites. Written submissions were requested from the breakout-session attendees. The written components are intended to be a compilation of slurry-transport information significant to the DOE-EM cleanup process and are provided in Appendix A.6. A short summary of the written components follows.

Information on activities at the Fernald site were provided by C. Phil McGinnis. The Fernald Closure Project was supported by a DOE technology-assistance effort. Mr. McGinnis led a team of engineers and chemists from ORNL, PNNL, INL, Mississippi State University, The University of North Dakota Energy and Environmental Research Center, and other industries to test a piping flow loop. Testing of the design in an existing flow loop at ORNL demonstrated that the surrogate (provided by PNNL) would not stay suspended and caused plugs in the line. Equipment-evaluation tests were conducted using several pumps and slurry instrumentation. Results were documented in a series of reports culminating in a final report. The executive summary of the report is included in Appendix 6; the full report can be sent electronically upon request.

Savannah River Site staff provided information on three projects at SRS: the Defense Waste Processing Facility (DWFP), the Saltstone Production and Disposal Facility, and vacuum retrieval and transport. The DWFP discussion includes two summaries on SRS tank-farm sludge rheology from Terri L. Fellinger and Jonathan Bricker. Erich Hansen provided a summary of transport issues related to the Saltstone process. This included a discussion about transport of grout mixtures using large-scale peristaltic pumps. Lastly, Daniel Krementz provided a summary of three-phase-flow transport issues that have arisen when using a vacuum retrieval and pneumatic transport process at SRS. These contributions can be found in Appendix A.6.

Rick Demmer of Idaho National Laboratory provided a written summary of INL slurry-transport issues, which is in Appendix A.6. The summary includes issues associated with the transport of INL liquid waste slurries as well as pneumatic transport of calcined waste.

John Connor and Mark N. Hall of Bechtel's Waste Treatment Plant project provided a written summary of transport issues from the Hanford tank farms, which is included in Appendix A.6. The summary presents information on the pumps and instrumentation that have been used at the Hanford Site. Line-plugging and -unplugging issues at Hanford are discussed. The K-Basin cleanup project at Hanford also faced several challenging transport issues; Gary Hofferber provided information on a set of hose-in-hose simulant-transport tests that were conducted in support of this project. The lessons learned from these tests were compiled in a conference paper, the abstract of which is provided in Appendix A.6.

Robb Burk reported that the Hanford Waste Treatment Plant (WTP) has recently commissioned a set of pipeline-transport tests, since the WTP expects to process large quantities of non-Newtonian slurries. Testing at PNNL investigates the deposition-velocity calculations for both Newtonian and non-Newtonian slurries. The PNNL test results indicate that a design approach for maintaining critical velocity of both Newtonian and non-Newtonian fluids should include at least three criteria: 1) the critical deposition boundary, 2) the transitional deposition boundary, and 3) the laminar deposition boundary. These boundaries define the transitions between stable and unstable slurry-transport flow regimes for systems with non-Newtonian flow properties. Correlations for these boundaries can be found in the draft PNNL report, WTP-RPT-175 Rev A. Appendix A.6 includes the testing summary from this report.

Lastly, Dr. David Roelant of Florida International University's Applied Research Center served as principal investigator for a pipeline-unplugging test with technology from NuVision Engineering. Results of these recently-completed tests were compiled into a report, the executive summary of which is provided in Appendix A.6.

Emerging Findings for Waste Slurry Pipeline Transport Critical Velocity Correlations

Recent and ongoing tests performing pipeline transport critical-velocity measurements using waste slurry physical simulants have yielded new results. Test results are summarized by Poloski et al. (March 2009). Critical velocity is defined as the point where a moving bed of particles begins to deposit those particles on the bottom of a straight horizontal pipe during slurry-transport operations. Deposition testing was performed on slurries containing particles with well-characterized size and density suspended in fluids with known rheological properties. The test results provide information to evaluate the conditions that can lead to plugging in waste-slurry pipeline-transport operations.

The simulant test particles ranged in density from 2.5 to 8 g/cc while the nominal particle size ranged from 10 to 100 μm. Seventeen tests were conducted with these test particles suspended in three carrier fluids with target Bingham-plastic yield-stress values of 0, 3, and 6 Pa.

An experimental flow loop was constructed of 3-inch schedule 40 stainless steel piping with a mixing tank, slurry pump, and instrumentation for determining flow rate and pressure gradient. At the beginning of a test, the slurry flow velocity was nominally set to 8 ft/sec. The flow was then incrementally decreased and a steady-state pressure gradient was obtained at each flow condition. A rise in pressure

gradient as the flow rate dropped indicated that the pipe cross-sectional area had filled with moving sediment. This point is referred to as the "critical velocity." The laminar-to-turbulent transition velocity is referred to as the "transition velocity."

Since typical critical-velocity correlations are limited to Newtonian fluids with particles greater than 100 micrometers in size, and nuclear-waste slurry processing will include non-Newtonian slurries with these properties, the testing investigates deposition-velocity calculations for non-Newtonian slurries. The test results indicate that a design approach for critical velocity of both Newtonian and non-Newtonian fluids should consist of at least three criteria: 1) the critical deposition boundary, 2) the transitional deposition boundary, and 3) the laminar deposition boundary. These boundaries define the transitions between several slurry-transport flow regimes. Four of the major regimes are shown schematically in Figure 3.1.

The three boundaries between these four regimes are strong functions of slurry rheological properties. By plotting yield stress on the abscissa and flow velocity on the ordinate, a "stability map" showing these stability regimes can be obtained. An example stability map is shown in Figure 3.2. Point W on the abscissa (i.e., rheological properties equivalent to water) represents the Newtonian critical deposition velocity for turbulent flow. This point can be predicted by the Oroskar and Turian (1980) and Shook et al. (2002) equations. As the non-Newtonian character of the slurry increases, adding yield stress, the increased apparent viscosity of the slurry adds drag on the particles and reduces the flow velocity required for suspending the particles in turbulent flow. The boundary between the stable turbulent and unstable turbulent flow regimes is referred to as the critical deposition boundary and is depicted by path WX (shown in blue).

However, the flow velocity required to reach turbulent flow increases with slurry rheological properties because viscous forces dampen the formation of turbulent eddies. At point X, the flow becomes dominated by viscous forces rather than turbulent eddies. Since the turbulent eddies necessary for particle transport are not present, the particles will settle when flow velocity is below this boundary, called the transitional-deposition boundary. Along path XY, the stable turbulent and unstable laminar flow regimes are defined.

As the yield stress continues to increase, the required pressure gradient for flow increases. At point Y, the required pressure gradient is adequate to push the particles through the pipeline even along the pipeline wall. Often yield stress forces are large enough to suspend the particles in the stagnant core region of a non-Newtonian flow. Along path YZ, the rheological properties continue to increase, which lowers the flow velocity required to achieve the pressure gradient required to push the particles through the pipeline. Hence, for particles in slurries to remain suspended, **pipelines must be operated in Regime A or Regime D**. The test report presents the equations that define where these boundaries fall with respect to particle and rheological properties. Testing has been completed using more complex waste simulants over a wider range of Bingham plastic yield-stress values to better define the laminar deposition boundary for physical and rheological properties relevant to nuclear-waste slurries Poloski et al. (July 2009). Further testing on the transport of particles in non-Newtonian suspensions flowing through complex piping systems consisting of elbows, tees, PUREX connectors, reducers, and vertical rises are described in Yokuda et al. (May 2009). This report also includes an investigation of the transport of particles in non-Newtonian suspensions in partially filled sloped piping to simulate gravity-driven drains.

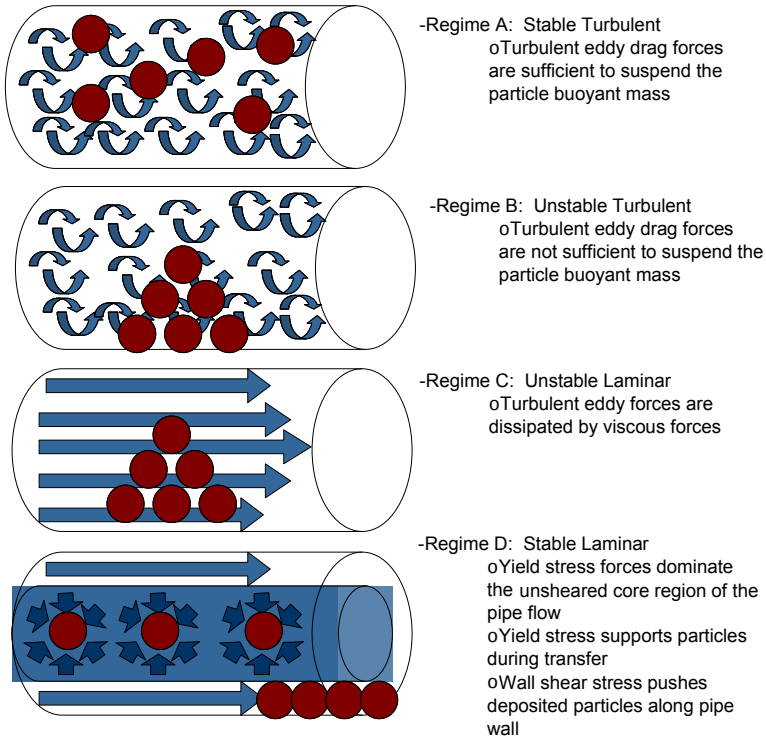

Figure 3.1. Four Slurry-Transport Flow Regimes

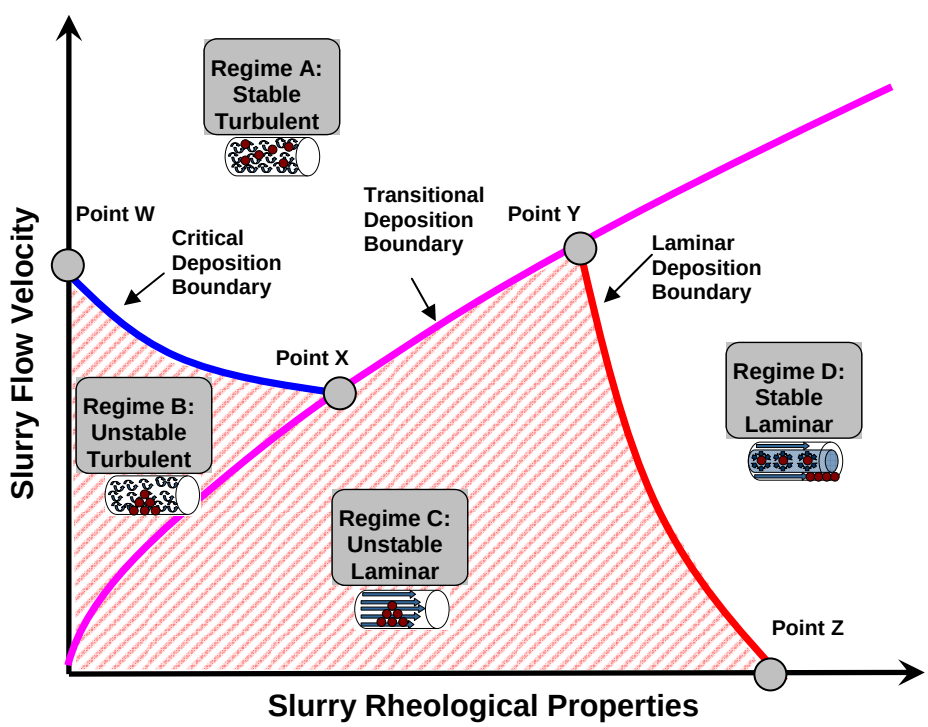

Figure 3.2. Example of a Stability Map

3.2.3 Slurry Processing

The Unit Operations breakout sessions of the Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop met twice. This was a voluntary section devoted to discussing the issues, problems, and lessons arising from processing slurries. Attendees agreed that the format for this session would follow the overall headings of the chemical-processing unit operations with their specific applications within the DOE sites' processing systems. The details of the session are included in Appendix A.6

In the first session, the following divisions were determined to be the major DOE-applicable unit operations for discussion and further development:

- Mixing – homogeneous mixing, specialized sampling (e.g., accountability, process control). Uniform transfer (e.g., melter feed to melter, whole batches), heterogeneous mixing (well stirred, off the bottom), and computational fluid-dynamics modeling (this mixing section overlapped with the breakout section on pulse-jet mixers).
- Solid-Liquid Separations – centrifugation and filtration
- Waste Immobilization – grouting, vitrification, and steam reforming
- Chemical Reactions – leaching, oxidation, reduction, dissolution, and precipitation
- Evaporators – scaling, precipitation, and foaming
- Process Control – instrument line unplugging in processes, solids interference with instrumentation for liquids, on-line assay and non-destructive analysis (NDA)
- Ventilation (off-gas processing) – gas generation, retention and release (H_2, CO_2, volatile organic compounds), aerosolization, and ties with process chemistry and retrieval.

Discussion in selecting these divisions was significant and covered most of the unit operations involving slurries throughout the DOE complex. Attendees were assigned "homework" to identify key DOE sites providing information for these operations; the homework was to identify design criteria the sites used when processing slurries in these operations, identify particular operations that were in process or were successfully applied at the DOE sites, state lessons that had been learned and what guidance references (i.e., DOE Orders) were available.

The next breakout session dealt with providing a summary of slurry-processing activities that had taken place at each site. It was a frank and open discussion of general difficulties that the process engineers had working with slurries and some improvements that could be made. Lessons that have been learned from the DOE sites also were highlighted. A great deal of the discussion centered on the frustration of rushing systems into operation without adequate development. While research and development can be an expensive investment, the challenge of working with slurries dictates more development for successful startup and operation. Rushing complicated systems into operation without sufficient development usually results in costly upgrades.

Another area of discussion among process engineers was where to turn for understanding of the historical and technical accomplishments within the DOE complex. This was expressed as the desire to develop an "encyclopedia" of processes that produced the waste mixtures we are trying to treat. A good

start would be to gather the descriptive training manuals for processes such as the PUREX method that was used at Hanford to recover plutonium and uranium. A knowledge center that collects process information and identifies Subject Matter Experts for future reference, such as the D&D folks are putting together with Florida International University, would be another good step. The sessions concluded with the agreement that the DOE sites have technical challenges in processing slurries, and that workshops such as this were tremendously valuable for instruction from slurry experts, and certainly for the interaction and discussion among various site engineers.

3.3 Special Breakout Sessions

3.3.1 Retrieval – Mobilization to Overcome Shear Strength

On day two, a special session moderated by Professor David V. Boger of the University of Melbourne dealt with mobilization needed to overcome shear strength. The understanding, or lack thereof, of physical properties of waste was the topic of much of the discussion. One of the questions posed by Professor Boger was, "What properties of the waste should you know before waste removal begins?"

This question resulted in much discussion about the reality of tank-waste retrieval at the Hanford and SRS sites today. The premise of the question was that if we know more about the waste and its physical behavior, then we will be more successful in fielding appropriate technologies for removing it from underground storage tanks for transport and processing it for waste disposal. The general conclusion was that most of the difficulties come from roughly 10% of the waste volume, in terms of waste retrieval without major concerns regarding waste transport or waste processing and treatment. These statements were based upon experiences at the sites during sluicing of single-shell tanks during the era of operations. As noted, there has not been significant experience with tanks that have been emptied and closed. The reality of funding priorities at the sites is that until significant problems occur, the cost of obtaining representative samples (or determining properties either *in-situ* or via core samples) is prohibitive.

The site baselines have been selected, and if there were additional sampling, or if it were affordable to sample now, it would likely have minimal impact on current retrieval baseline technologies. The overlying feeling of the site staff was that getting the waste out of the tanks is difficult, and if that can be accomplished, then other things can be done to ensure that waste can be transported to the next step of the process. One of the problems with knowing more about the waste is that representative samples would have to be taken and analyzed to understand its properties. Costs of representative sampling are prohibitive, and any samples taken would not tell what the *in-situ* properties of the waste truly are. In general, the sites continue to move in a direction of not initiating major sampling campaigns until there is a problem that could stop retrieval operations. While the consensus of the group was that additional physical properties would be beneficial, there is minimal support for performing these property measurements.

3.3.2 Transport – "Open Channel Flow" and "Critical Velocity w/ Viscosity Adjustment"

A special breakout session on gravity-flow systems was moderated by Robert Cooke. Mark Hall described the design guide for determining slopes for process-drain/overflow lines within the Waste Treatment and Immobilization Plant (WTP). He began with a review of process lines affected by the

design guide as well as 0anticipated waste properties. He explained that three slopes are to be implemented in the WTP with rise-to-run ratios of 1:100, 1:50, and 1:20. He then outlined the technical approach for determining system flow rates and velocities before determining an effective pipe diameter. The diameter and slurry properties (e.g., particle size, density, and viscosity) are then used in equations for conventional pipeline transport; the result is a prediction of the critical velocity of the open-channel system.

3.3.3 Processing – Pulse-Jet Mixers (PJM)

A special breakout session on pulse-jet mixers was moderated by Art Etchells. The session was attended by 16 workshop participants.

Perry Meyer of PNNL described the PJM mixing tests at PNNL for Bechtel and the Waste Treatment Plant (WTP) project at Hanford. He began with a review of the work on non-Newtonian fluids and the correlation derived for getting complete mobility in a tank. This could be achieved by PJMs alone, but the cost of additional air-handling equipment was prohibitive. A hybrid design, with PJMs mixing the lower part of the vessel and sparged air mixing the upper part of the vessel, was developed and will be implemented. He discussed current work on the suspension of discrete particulate solids, looking at off-bottom suspension and vertical distribution as measured by cloud height. This presentation is included in the proceedings.

Representatives from NuVision Engineering, the current suppliers of PJM technology, said they knew of no equivalent information from their history.

Sam Shicks of the Parsons engineering firm gave a preview of his talk on re-suspending a bed of fine, fluffy solids with devices similar to the PJMs that are called air pulse agitators (APA) by Parsons. This work was done in support of the proposed Salt Waste Processing Facility (SWPF) at Savannah River. He was able to use the actual salt waste material in two scale tanks and measured the time it took to achieve uniform concentration of the solids. Correlations of velocity with various geometric parameters were obtained. Attendees from Savannah River National Laboratory (SRNL) will work with Parsons to better characterize the settling bed and the final slurry. Shicks's work was presented later in the workshop and is included in the proceedings.

There was a general discussion of what data and models are available for various process results for PJMs and the need to develop consistency and a good technical basis. Some existing information is considered proprietary by the possessors.

The only action item that came out of the session was the conclusion that a workshop on PJM mixing involving expertise from both the UK and the USA would be most useful. Etchells is to follow up on organizing such a workshop in 2009.

3.3.4 Computational Fluid Dynamics

The special session on computational fluid dynamics was moderated by Tom Michener. The first portion of this unplanned, *ad hoc* session was an opportunity to discuss the number of staff working as near-full-time CFD modelers at each company, with the intent of getting an order-of-magnitude feel for what is going on in CFD for the attendees of the workshop.

The remainder of the discussion was of the role of CFD in supporting the slurry transport and mixing issues that DOE is facing while dealing with the transport and mixing processes involved as part of nuclear-waste-vitrification efforts. Private-industry attendees working with non-nuclear slurries also provided their input on CFD and multi-phase modeling at their companies.

Estimates of number full-time staff performing CFD

- SRNL has 1-2 applied-CFD staff
- Nexia Solutions in the UK has ~10 people, a mixture of modelers and developers
- Dow has ~10 people running commercial codes with enhancements in some cases
- Bechtel National, Inc. has 3-5 CFD modelers
- DuPont has 3-4 CFD modelers and 1 developer
- PNNL has ~20 staff performing CFD, of which ~6 are developers.

General discussion

The discussion started with comments regarding the impact on the credibility of CFD when applied improperly, whether it is:

- inappropriate grids
- application of the software outside the bounds of the assumptions for the constitutive equation
- presentation of predictions when the code has not been verified or validated for the specific type of application.

The majority opinion among the attendees was that multi-phase and non-Newtonian CFD capabilities are still weak. Examples of difficult problems that are not generally solved correctly are the slumping problem with low-tech rheometers, and accurately modeling particle distributions in mixers. The problems with non-Newtonian flow typically arise from a problem with the code in some of the popular software, while the complex physics of the turbulent interaction on drag coefficients in multi-phase systems is usually inadequately modeled. Thus multi-phase modeling needs extensive experimental validation.

Another example of CFD limitations is the free-surface problem: correctly predicting the vortex that forms during certain types of mixing conditions is still problematic.

There was a general consensus that CFD would be of the most value if modelers worked more closely with the experimentalist process Engineer. The more the modeler knows about fluid flow in general, the better the models will be. Validation against good and meaningful experimental data is a necessity.

There will be an enormous amount of data coming from the Hanford M1 (pipeline transport) project. This data will provide an opportunity to evaluate the multiphase modeling capabilities of existing CFD codes and provide a challenge for software currently in development, such as PNNL's lattice-Boltzmann-based multiphase CFD code. The initial data from M1 will provide data on critical flow velocities and the differing flow regimes for various particle-concentration loadings. Eventually, more complex simulants and non-horizontal pipeline geometries will also be investigated.

In addition to the M1 project, BNI/PNNL has an effort, (M3), to look at mixing of multiphase mixtures in the different tank designs using pulse-jet mixers (PJMs). The M3 project is tasked with:

- identifying tanks as containing Newtonian or non-Newtonian wastes
- identifying the tanks with suspected problems in mixing and defining the limiting rheologies
- providing PJM operational guidance

While it is not the intention of the "M" test series to provide data for the modelers, it is an opportunity for modelers to get access to data to perform model calibration and validation of existing and new approaches. Perhaps this data could become part of a standard suite of non-Newtonian data to validate CFD codes.

4.0 Slurry-Handling Protocols for DOE Projects

As discussed at the workshop, use of standardized guidelines is valuable to ensure comparable results from all actual radioactive-waste and simulant-based tests conducted within the DOE complex. Therefore, three sets of technical guidelines for use by engineers and scientists working on slurry issues were documented and recommended for use within the DOE complex as best practice guidelines: 1) Waste Slurry Sampling and Measurement Techniques; 2) Performing Chemical, Physical, and Rheological Properties Measurements; and 3) Simulant Development, Approval, Validation, and Documentation. Use of these guidelines will help ensure standardized, comparable results from all tests on simulated or actual waste slurries conducted across the DOE complex.

4.1 Waste Slurry Sampling

Obtaining samples of high-level waste created during the reprocessing of spent nuclear fuels presents unique challenges due to limited access to the waste, requiring remote sampling and shielding for sample handling, transport, and storage. Treatment processes in the tanks often produce heterogeneous multiphase wastes that contain significant concentrations of dissolved salts, frequently resulting in the formation salt-cake and/or crusts. High-level-waste tanks may contain liquid, slurry and sludge layers as well as these salt-cake and crust layers. Organic layers exist in some waste tanks, creating additional heterogeneity in the wastes. Vapors in the head space of high-level waste tanks are also important in characterization of these wastes, but vapor sampling is not included in this document because it does not significantly impact slurry retrieval, pipeline transport, plugging, or mixing.

Sampling of high-level waste tanks at Department of Energy (DOE) sites has been accomplished by a number of different methods, which are summarized in Table 4.1. Additional details about these methods and important parameters to consider when designing a sampling plan are included in Appendix B.

Table 4.1. High-Level Waste Tank Sampling Methods

Technique	Material Type			Notes
	Solids Li	quids	Gases	
Bottle on a String		X		Dip sample
Sample Thief (Bacon Bomb)		X		
Thief and Trier	X	X		Similar to core drilling.
Soft Sludge Sampler	X	X		Thin walled pipe
Sample Cup	X			Salt-cake sampler
Auger X				
Hydraulic Scoop	X			
Cylinder with retractable nose cone	X			
Robotic Arm	X	X	X	
SUMMA Canister			X	Evacuated canister
Sorbent Trap			X	Aerosol sampler
Cryogenic			X	Head-space vapor sampler

4.2 Organization of Chemical, Physical, and Rheological Guidelines

Standardized chemical, physical, and rheological measurement guidelines are valuable for use across the DOE complex. The property measurement guidelines, provided in Appendix C, were developed based upon work at the Waste Treatment and Immobilization Project (WTP). They are a valuable starting point for a consensus property-measurement guidelines document. Use of these property-measurement guidelines is essential to ensure standardized, comparable results from all actual and simulant-based tests conducted across the DOE complex.

This document brings together chemical, physical, and rheological property-measurement guidelines for the characterization of both actual and simulated wastes. This document is organized into four main sections:

- Introductory Text – These sections contain a table (see Table 1) indicating data that is to be obtained to properly characterize both actual and simulated wastes. All data is to be reported in a hard copy and an accompanying electronic Excel® file (see Table 12).
- Chemical Properties Measurements – This section contains guidelines for obtaining the necessary chemical composition data. The specific analytical procedures, minimum reportable quantities (MRQ), etc., will be agreed upon by the DOE project and the performer of the work via project-approved documentation, e.g., a test-specification/test-plan process. Only instructions for determining pH are given in this section.
- Physical Properties Measurements – This section integrates the steps required for determining each physical property. The physical property measurements are for density of bulk slurry (g/mL), density of settled solids (g/mL), density of centrifuged solids (g/mL), density of supernatant liquid (g/mL), settling rate (vol%/min), particle-size distribution, vol% centrifuged solids, vol% settled solids after 72 hours, wt% undissolved solids, wt% dissolved solids, wt% centrifuged solids, wt% total oxides, wt% oven-dried solids, and wt% total dried solids.
- Rheological Properties Measurements – This section provides a standardized approach to determination of important rheological properties for slurries and melter feeds. The rheological property measurement procedures provided include shear strength, shear stress vs. shear rate, and yield stress.

The property-measurement protocols detailed in the Appendix C guidelines are applicable for research and technology (R&T) testing within DOE projects. This is intended to be a "living" document and will be updated as other DOE tasks determine what additional testing methods need to be added. After the additional specific measurement procedures are developed, reviewed, and approved for use, this guideline document will be revised. The user is responsible for confirming that he/she is using the latest version of this document.

4.3 Simulant Development, Approval, Validation, and Documentation

The guideline provided as Appendix C provides instructions necessary for research and technology project personnel to develop, approve, validate, and document simulants for use within their project. The simulant development guideline, which is described in detail in Appendix D, applies to new and revised simulant-development activities and their associated project documents, such as test specifications, test

plans, test exceptions, simulant-preparation procedures, and test reports. Following the directions in this document will facilitate a consistent methodology for development, preparation, and validation of simulants for use within DOE-complex projects.

This guideline directs how research and technology project personnel:

1. define simulant use;
2. define simulant composition or range of compositions;
3. define simulant-design requirements;
4. review and approve simulant development activities;
5. verify and validate that simulant meets design requirements;
6. verify that simulant is consistent with flowsheet predictions; and
7. document simulant-development activities and preparation procedures.

A brief outline of the guideline documents follows:

1. Scope Statement:
   a. Determine what the simulant is to resemble, e.g., only precipitation of Strontium and transuranics, or all pretreatment-unit operations.
   b. Define the simulant composition: actual waste characterization data will be used as the starting point along with a check against historical waste data; after the best available information on the waste stream is determined, process flowsheet models, etc., can be used to ensure that the correct additional stream compositions are addressed along with any other planned operations (e.g., adjustment of actual waste composition data to reflect future waste-feed delivery activities, such as dissolution of salt cake, to arrive at the optimum composition.
2. Simulant Design Requirements:
   a. Determine which properties are “necessary and sufficient” to measure for each affected unit operation; these should be the same for both actual and simulant waste.
   b. Determine, with cognizant engineers/scientists of affected unit operations, "How close is close enough?" for the "necessary and sufficient" properties of the important analytes, properties, etc. These will then become acceptance criteria for the simulant eventually prepared, to 'validate' the simulant-preparation procedure.
3. Review and Approval:
   a. Review and approval will be performed by individuals who are technically cognizant within all affected unit operations, including appropriate subcontractor personnel.
4. Develop preparation procedure and document that the simulant meets established acceptance criteria. [Note: if, after preparation and properties measurement of the simulant, the original acceptance criteria values cannot be met, an iterative process may be required to come to closure on the “How close is close enough?” question for the analytes, properties, etc; the iterative process would include appropriate project personnel.]

5. Document Simulant Development Activities and Preparation Procedure:
    a. Simulant Preparation Procedure documentation to include:
        i. Simulant Designation
        ii. Simulant waste stream / unit operation usage / requirements
        iii. Actual simulant preparation procedure:
            1. chemicals to use
            2. order of chemical addition
            3. precautions
            4. all other important information needed and recommendations for correct preparation
        i. Key characteristics, limitations, etc., of simulant
        ii. Validation that procedure was followed; documentation of the steps delineated above, e.g.:
            1. chemical composition
            2. "necessary and sufficient" properties
            3. baseline flowsheet design basis (does the simulant fall within the design basis?)
            4. acceptance criteria met
            5. all other important information and steps needed for validation
        i. Technical/Peer-review comments, issues, and such have been addressed
        ii. all other pertinent comments
        iii. Reference for development organization, contact name, etc.

5.0 Conclusions and Recommendations

5.1 Programmatic Conclusions and Recommendations from Industry Experts

The main programmatic conclusions from industry experts at the workshop are:

1. In-depth study of commercial process industries have shown that handling solids is many times more difficult than handling gases or liquids, particularly if the solids are unrefined, which is the case with the variety of wastes to be handled in the DOE complex. Innovative process steps may appear to have significantly low investments but put at risk the success of the processes.
2. It was also shown by the commercial experts that the "best of the best" innovative process steps succeed only about half the time. Too many innovative steps can lead to disaster, so contingency plans for innovative processing are a must. To prevent unrealistic designs, a common industrial practice is third-party review which, for maximum success, must occur several times along the project timeline. Third-party reviews should involve senior technical people who are not involved with the project and will be able to speak freely. They should be mainly people with an industrial background, not just those from the academic community.
3. Projects must have good chemical and physical characterization of the actual waste slurries being processed, from as-received through all unit operations, to underpin the design basis. Processes cannot be designed based on average slurry properties but must be designed over a robust range of variable properties of slurries.
4. An ongoing effort to develop and maintain a slurry-handling core competency within the DOE Complex is required. This could be accomplished through a community-of-practice environment.

5.2 Recommendations on the establishment of Technical Guidelines for Slurry Handling

Three sets of technical guidelines for use by engineers and scientists working on slurry issues were provided and are recommended for use within the DOE Complex. They are:

1. Guidelines for Waste-Slurry Sampling and Measurement
2. Guidelines for Simulant Development, Approval, Validation, and Documentation
3. Chemical, Physical, and Rheological Characterization of Waste Slurry

5.3 EM-20 and the Importance of External Independent Reviews

The National Academies of Science (NAS) 2007 report, *Assessment of the Results of External Independent Reviews for U.S. Department of Energy Projects,*[1] concluded that projects benefit from

[1] Committee on Assessing the Results of External Independent Reviews for U.S. Department of Energy Projects, National Research Council. 2007. *Assessment of the Results of External Independent Reviews for U. S. Department of Energy Projects,* National Academies Press, Washington, D.C. Available from http://books.nap.edu/catalog.php?record_id=11887

preparing and conducting external independent reviews and independent project reviews and that this benefit increases as the size, complexity, and inherent risks of the project increase.

External and internal reviews can provide pertinent information for DOE-EM to assess technical risk associated with projects, develop strategies for reducing that risk, and provide technical information needed to support critical project decisions. Technical-risk reduction increases the probability of successful implementation of technical scope.

5.4 Workshop Conclusions

The key message from Industry experts at the workshop was that in-depth studies of commercial process industries have shown that handling solids is many times more difficult than handling gases or liquids, particularly if the solids are unrefined, which is the case with the variety of wastes required to be handled in the DOE complex (Appendix A.4).

Experts from the mineral industry, which is a good process analogue to radioactive-waste processing, discussed their experience and provided the following additional key insights:

- Innovative process steps may appear to have significantly lower investments but put at risk the success of processes (Appendix A.4). A recent example discussed was the pulse-jet mixers which, though demonstrated on some materials, had never been demonstrated on the materials of interest or on the scale required (Appendix A.4 and A.6).
- Even the best projects only have successful innovative process steps about half the time (Appendix A.4). Too many innovative steps can lead to disaster, so contingency plans are needed for innovative processing steps.
- To prevent unrealistic designs, a common industrial practice is third-party review, which for maximum success must occur several times along the project timeline. Third-party reviews should involve senior technical people who are not involved with the project but will be able to speak freely. They should be mainly people with an industrial background, not just those from the academic community. (Appendix A.4)
- The DOE and the contractor(s) hired to design, procure, and build processing facilities must have a shared project strategy that is DOE driven. The contracting team must balance the production-engineering attitude ("Let's get the plant designed and built") and the process-engineering attitude ("Let's make sure we understand everything"), coupled with a successful plant commissioning and a focus on ease of operation.

Breakout sessions discussed how to prevent problems on DOE projects and suggested paths forward (Chapter 3).

<u>Slurry Retrieval:</u>

- There are many types of waste to be retrieved. In many tanks, the variability of physical properties within a given tank can be significant. The ability to design and field retrieval equipment depends on knowing and understanding the rheological properties.
- Waste-retrieval equipment used today has insufficient control of the slurries from the tanks.

Slurry Transport:

- An aggressive campaign of slurry physical and rheological characterization is needed for proper design of slurry-transport systems.
- Slurry pipelines should be designed to avoid laminar-flow regimes.
- Typical correlations from the mineral industry do not cover the ranges of particle properties encountered in DOE cleanup applications; mineral-industry correlations focus on particles greater than 100 micrometers, while particles in DOE slurries are typically less than 100 micrometers in size with a broad distribution of particle densities. The correlation from Saskatchewan Research Council (SRC), led by Dr. R.G. Gillies, is viewed as the best available; attendees of their course receive software and training. Since the Hanford Waste Treatment Plant (WTP) expects to process large quantities of non-Newtonian slurries, the WTP has recently commissioned a set of pipeline transport tests. Testing at PNNL investigates the deposition-velocity calculations for both Newtonian and non-Newtonian slurries. Results of the PNNL tests indicate that a design approach for critical velocity of both Newtonian and non-Newtonian fluids should consider at least three criteria: 1) the critical deposition boundary, 2) the transitional deposition boundary, and 3) the laminar deposition boundary. These boundaries define the transitions between stable and unstable slurry-transport flow regimes for systems with non-Newtonian flow properties. Correlations for these boundaries can be found in the draft PNNL report, WTP-RPT-175 Rev A.

Slurry Processing:

- Paper studies are inadequate for slurry process design; thorough cold testing of unit operations can obviate costly upgrades.
- It is crucial to processing success to understand how various processing unit operations affect chemical and physical properties of slurry and to what extent.

Pulse-Jet Mixers:

- Pulse-jet mixing is an innovative process technology that was not as fully developed as many believed and, as often happens with innovative processes, needed much more development work and redesign than expected. Such failures of innovation are common with slurries in the process industries.
- Equipment manufacturers frequently do not have extensive knowledge of process applications. Such knowledge often gets lost over time with loss of key technical people.
- PJMs are not designed to mix tall tanks unassisted.
- Because they use high velocities, PJMs must be tested for erosion potential.
- Devices handling raw solids such as tank-farm waste will be subject to a wide variety of feeds and must be designed based on worst-case conditions rather than average properties.
- Early review of key technology assumptions by technical people from the process industries would probably have uncovered the shortcomings of the PJMs; such third-party reviews are common in the process industries.

Computational Fluid Dynamics (CFD) Modeling:

- There is a role for Computational Fluid Dynamics in slurry handling; however, it must be validated and applied correctly. It has not been proven that CFD is capable of accurate predictions when applied to multi-phase, chemically reacting mixtures with complex rheologies, such as waste slurries.
- While current CFD modeling is improving, it can be dangerously misleading if conducted improperly. Current CFD codes do not have all the necessary correct physics, chemistry, and numerical modeling capability to handle multi-phase flow problems and will need to take advantage of high performance computing capabilities to be able to model complex slurry interactions in a timely manner.
- CFD validation against known data is crucial. The ability of any CFD model to predict slurry behavior prior to actual experiments is the true test; blind validation, or in other words, pre-test predictions, must be demonstrated to match test results without any additional data manipulation.
- More research is needed on multiphase-flow turbulence and how to model it.

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Note: PDF versions of WTP reports can be found at the following URL: http://www.pnl.gov/rpp-wtp/

Appendix A.1

Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Meeting Program

Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Monday – Friday, January 14 – 18, 2008
Marriott Courtyard Orlando Lake Buena Vista in the Marriott Village, Orlando, Florida

MEETING PROGRAM

Sponsored by the U.S. Department of Energy
Office of Engineering and Technology
Office of Environmental Management

Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Monday – Friday, January 14 – 18, 2008
Marriott Courtyard Orlando Lake Buena Vista in the Marriott Village, Orlando, Florida

Workshop Organizers:

Gary L. Smith, Adam P. Poloski, and Michael W. Rinker
Pacific Northwest National Laboratory

Sharon L. Marra
Savannah River National Laboratory

Rick Demmer
Idaho National Laboratory

Benjamin E. Lewis Jr.
Oak Ridge National Laboratory

Arthur W. Etchells, III
AWE3 Enterprises

Sponsored by the U.S. Department of Energy
Office of Engineering and Technology
Office of Environmental Management

INTRODUCTION
Slurry Retrieval, Pipeline Transport & Plugging, and Mixing Workshop

The DOE Office of Engineering and Technology, Office of Environmental Management is sponsoring a slurry handling workshop which is being coordinated by Pacific Northwest National Laboratory, Savannah River National Laboratory, Oak Ridge National Laboratory, and Idaho National Laboratory with help from commercial slurry processing companies. The workshop will focus on slurry retrieval, transport, mixing, etc. The objective of this effort will be to generate a manual/report documenting slurry transport to include mobilization and transport technologies focusing on the risks associated with slurry retrieval, mobilization, pipeline plugging, and stratification of slurries in vessels. The manual/report will also include aspects of slurry characterization consensus methods and design of slurry mobilization and transport that will allow for comparable results and system designs among DOE sites. The meeting will be attended by over fifty people from radioactive waste storage sites in North American and beyond as well as both commercial firms and academic institutions. The workshop will be held in Orlando, Florida the week of Monday, January 14, 2008. The first two days will be a short course on slurry mixing and handling; the last three days will be presentations and workshops on best practices, lessons learned, etc. (Requests for further information should be directed to Gary L. Smith, (509) 372-1957, Gary.L.Smith@pnl.gov)

SLURRY RETRIEVAL, PIPELINE TRANSPORT & PLUGGING, and MIXING WORKSHOP

Workshop Agenda:

Monday, January 14, 2008

8:00 am	Short Course
9:30 am	Break
12:00 pm	Lunch on Own
1:00 pm	Short Course
2:30 pm	Break
4:30 pm	End First Day Short Course

Dinner on Own

Tuesday, January 15, 2008

8:00 am	Short Course
9:30 am	Break
12:00 pm	Lunch on Own
1:00 pm	Short Course
2:30 pm	Break
4:30 pm	End Short Course

6:30 pm Group Dinner at Landry's Seafood

Wednesday, January 16, 2008

8:00 am	Workshop Welcome and Introduction	Steve Krahn
8:10 am	Workshop Purpose and Format	Gary L. Smith
8:30 am	Hanford Site Presentation a. Waste Slurry Physical Properties – Adam Poloski b. Tank Waste Retrieval – Rick Raymond c. Waste Transport – Rick Raymond d. Waste Processing – Adam Poloski	
10:00 am	Break	
10:30 am	Savannah River Site Presentation – Sharon Marra a. Sludge Waste Removal and Tank Closure - Noel Chapmen b. Work Supporting Removal of Slurries from Waste Tanks, Transport of Slurries in Pipelines, and Separation of Solids from Liquids at SRS – Michael Poirier	

c. Overview of the Design Basis, Operating Experience, and the Application of Laboratory Data to Support the Transport and Mixing of Sludge Slurries in the Defense Waste Processing Facility - Jonathan Bricker

12:00 pm Lunch on Own

1:00 pm Idaho Site Presentation – Rick Demmer
a. Waste Slurry Physical Properties
b. Tank Waste Retrieval
c. Waste Transport
d. Waste Processing

1:30 pm Oak Ridge Site Presentation – Ben Lewis
e. Waste Slurry Physical Properties
f. Tank Waste Retrieval
g. Waste Transport
h. Waste Processing

2:00 pm Break

2:30 pm Break-Out Sessions: What do we need for final product (report/manual)?
a. Retrieval
b. Transport
c. Processing

4:00 pm Feed Back and Report Out from Break-Out Session Chairs

4:30 pm Key Note Speaker "Third Party Reviews – An Industrial Best Practice" Rick Bockrath

5:30 pm Adjourn Workshop

Dinner on Own

<u>Thursday, January 17, 2008</u>

8:00 am Key Note speaker on "The Problem with Solids" David Gottschlich

9:30 am Break

10:00 am Break-Out Sessions: How to prevent problems on DOE projects?
a. Retrieval
b. Transport

c. Processing

11:30 am Feed Back and Report Out from Break-Out Session Chairs

12:00 pm Lunch on Own

1:00 pm Discussion of Path Forward – Report from the Workshop

2:00 pm Break

2:30 pm Break-Out Sessions: Path Forward

a. Retrieval – Mobilization to Overcome Shear Strength, David Boger Moderator
b. Transport – "Open Channel Flow" and "Critical Velocity w/ Viscosity Adjustment", Robert Cooke Moderator
c. Processing – Pulse Jet Mixers (PJM), Art Etchells Moderator
d. Computational Fluid Dynamics – Tom Michener Moderator

3:30 pm Feed Back and Report Out from Break-Out Session Chairs

4:00 pm Key Note Speaker on "Rheology and Surface Chemistry of Slurries" David Boger

5:30 pm Adjourn Workshop

Dinner on Own

Friday, January 18, 2008

8:00 am Key Note Speaker on "Slurry Retrieval & Transport" Robert Cooke

9:00 am Break-Out Sessions: Short talks on various topics:

- Overview of Advanced Design Mixer Pump Program – Robert Leishear
- Impeller Diameter Reduction of a Radioactive Sludge Transfer Pump – Tom Caldwell

10:00 am Break

10:30 am Break-Out Sessions: Short talks on various topics:

- Examples of Slurry Retrieval, Pipeline Transport & Plugging and Mixing issues at Sellafield in the UK – Tim Tinsley & Dominic Rhodes
- Lessons Learned in Operating the Hose-In-Hose System for Transferring Sludge at Hanford's K Basins - Gary Hofferber & Curt Miska
- On the Formation and Treatment of Trisodium Phosphate Plugs – Jeff Lindner et al.

12:00 pm Lunch on Own

1:00 pm Break-Out Sessions: Short talks on various topics:

- Tomography to Investigate Closed Systems – Tim Tinsley & Dominic Rhodes
- Mixing Within the Salt Waste Processing Facility [SWPF] – Air Pulse Agitators (APA's) – Sam Shicks
- Scale-Up Of Solids Suspension Agitation – Dave Dickey

2:30 pm Closing Discussion and Assignments

3:00 pm Close the Workshop

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ENERGY

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352
1-888-375-PNNL (7665)
www.pnl.gov

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Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Volume II

GL Smith
AP Poloski
MW Rinker
RL Demmer
AW Etchells, III
BE Lewis Jr.
SL Marra

July 2009

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Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

GL Smith
AP Poloski
MW Rinker
RL Demmer[1]
AW Etchells, III[2]
BE Lewis Jr.[3]
SL Marra[4]

July 2009

Prepared for
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Pacific Northwest National Laboratory
Richland, Washington 99352

(1) Idaho National Laboratory, Idaho Falls, Idaho
(2) AWE3 Enterprises, Philadelphia, Pennsylvania
(3) Oak Ridge National Laboratory, Oak Ridge, Tennessee
(4) Savannah River National Laboratory, Aiken, South Carolina

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Appendix A.4 – Keynote Talks

Improving Technical Success in Projects at DuPont by Rick Bockrath A.4.1

New Technology and Solids by Dr. David A. Gottschlich A.4.37

The Rheology and Surface Chemistry of Slurries by Dr. David V. Boger A.4.65

Slurry Transportation: An Overview by Dr. Robert Cooke A.4.189

Appendix A.5 – Other Special Topic Presentations

Gravity Flow Systems by Mark Hall A.5.1

Critical Velocity 2/Viscosity Adjustment by Mark Hall A.5.10

Mixing Sludges & Slurries with Pulsed Jets, by Perry Meyer A.5.19

Predicting Slurry Pump Performance by Robert Leishear A.5.57

Impeller Diameter Reduction of a Sludge Transfer Pump by Thomas. Caldwell A.5.85

Examples of slurry issues at Sellafield by Tim Tinsley & Dominic Rhodes A.5.106

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Treatment of Trisodium Phosphate Plugs by J.S. Lindner A.5.171

Imaging and Tomography at Sellafield by Dominic Rhodes & Tim Tinsley A.5.193

Mixing Within the Salt Waste Processing Facility (SWPF) by Sam Shicks A.5.262

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Appendix A.6 – Contributions from Workshop Breakout Sessions

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Slurry Retrieval A.6.5

Slurry Transport A.6.69

Slurry Processing A.6.100
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Volume IV
Appendix B – Sampling of Radioactive Wastes B.1
Appendix C – Guidelines for Performing Measurements of Chemical, Physical and Rheological Properties C.1
Appendix D – Guideline for Simulant Development, Approval, Validation and Documentation D.1
Appendix E – Slurry Rheology E.1

Appendix A.2

Workshop Short Course Materials

Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Monday – Friday, January 14 – 18, 2008
Marriott Courtyard Orlando Lake Buena Vista in the Marriott Village, Orlando, Florida

SLURRY MIXING, RHEOLOGY, AND HANDLING SHORT COURSE

Monday – Tuesday, January 14 – 15, 2008

Dr. Art Etchells Lectures

Book 1 of 2

Sponsored by the U.S. Department of Energy
Office of Engineering and Technology
Office of Environmental Management

Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Monday – Friday, January 14 – 18, 2008
Marriott Courtyard Orlando Lake Buena Vista in the Marriott Village, Orlando, Florida

Workshop Organizers:

Gary L. Smith, Adam P. Poloski, and Michael W. Rinker
Pacific Northwest National Laboratory

Sharon L. Marra
Savannah River National Laboratory

Rick Demmer
Idaho National Laboratory

Benjamin E. Lewis Jr.
Oak Ridge National Laboratory

Arthur W. Etchells, III
AWE3 Enterprises

Sponsored by the U.S. Department of Energy
Office of Engineering and Technology
Office of Environmental Management

MIXING, RHEOLOGY AND SLURRY HANDLING COURSE

Dr.Nigel Heywood – BHRG

Dr. Art Etchells – AWE3 entp.

January 14 and 15 2008

INTRODUCTIONS

The instructors

BACKGROUND

- Arthur Etchells
- BS and MS chemical engineering U of Pennsylvania
- PhD chemical engineering University of Delaware
- 2 years with Rohm and Haas
- 39 years with Dupont
- Last 29 years as an internal consultant

BACKGROUND

- Internal consultant in mixing and fluid flow in Dupont Engineering
- Served all businesses as a consultant
  - Manufacturing and process development and plant design
  - Polymers to slurries
  - Includes design of DWPF at SRS
- Retired in 2002 as Dupont Fellow – highest technical position in company
- Private consulting since
  - WTP at Hanford for Bechtel through Dupont

BACKGROUND

- Founding council member and former president and award winner of North American Mixing Forum an affiliate of the AIChE
- Adjunct teaching at University of Delaware and Rowan University
- Co-author of Handbook of Industrial Mixing Wiley 2002.

INTRODUCTIONS

The attendees

OTHER EXPERTS

- Professor David Boger – U of Melbourne
  - Rheology and slurry handling

- Dr. David Dickey – Mixtech – consultant on mixing processes and equipment

COURSE OUTLINE

- Two instructors – alternating
- Mixing and slurry flow
- Not often given this way
- Usually separate courses
- Lots of synergy
- Both topics demand same physical properties of the slurries
- Neither is covered in conventional engineering education

Mixing of Slurries

Arthur William Etchells III
AWE3 Enterprises
January 12 and 15 2008
DOE workshop

What will not be covered

- This is basically a 7 hour course
- Typical mixing courses are 3-4 days or 13 weeks
- Many things are being skipped
  - Gas liquid
  - Mass transfer
  - Heat transfer
  - Chemical reactions
  - High viscosity mixing – polymers
  - Workshops, sample calculations and problem solving

Ask questions anytime

Some time at end and during the rest of the workshop to ask questions and discuss problems

WHAT IS MIXING and WHY IT MATTERS

Introduction

M2

DEFINITION OF MIXING

The use of a mechanical device to generate a fluid motion to achieve a process result

DEFINITION OF MIXING

- Mechanical devices
  - There is a mixer

- Fluid motion
  - A branch of fluid mechanics
  - Will not discuss solids solids dry mixing
    - Does not depend on fluid mechanics or the Navier Stokes equations
- PROCESS RESULT
  - Mixing is a means to an end not an end itself.

PROCESS RESULTS

M3

PROCESS RESULTS

- Typical Duties/ Process Results
- Blending of miscible fluids
  - storage and blending
  - chemical reaction
    - high and low viscosity
- Contacting immiscible liquids
  - emulsification
  - extraction

PROCESS RESULTS

- Suspending Solids
  - reactors
  - precipitators
  - crystallizers
- Dispersing Solids
  - Slurry and product makeup
  - pastes

PROCESS RESULT

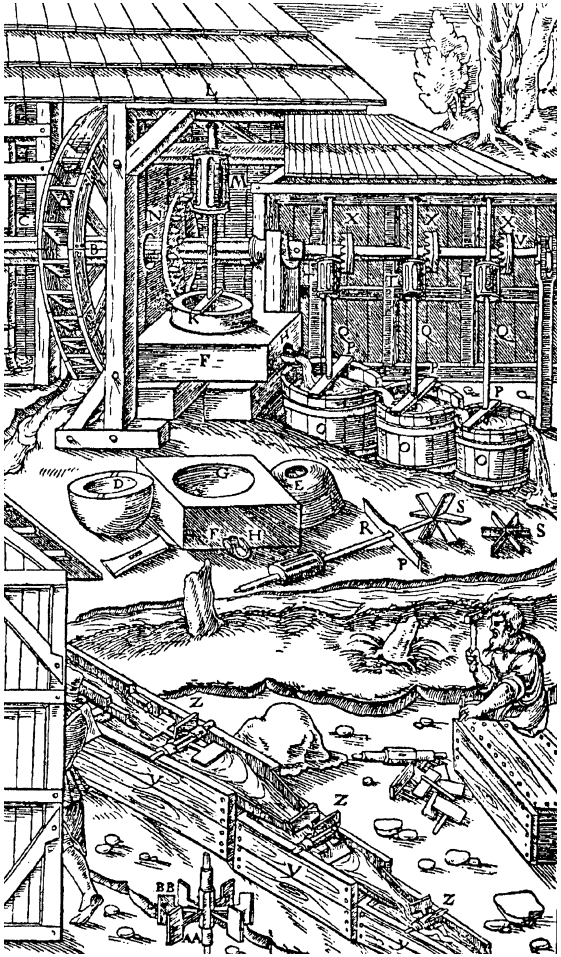
- Dispersing gases into liquids
  - reactors
  - fermentors
- Heating and cooling of liquids

- Other e.g. settled solids mobilization or gas release

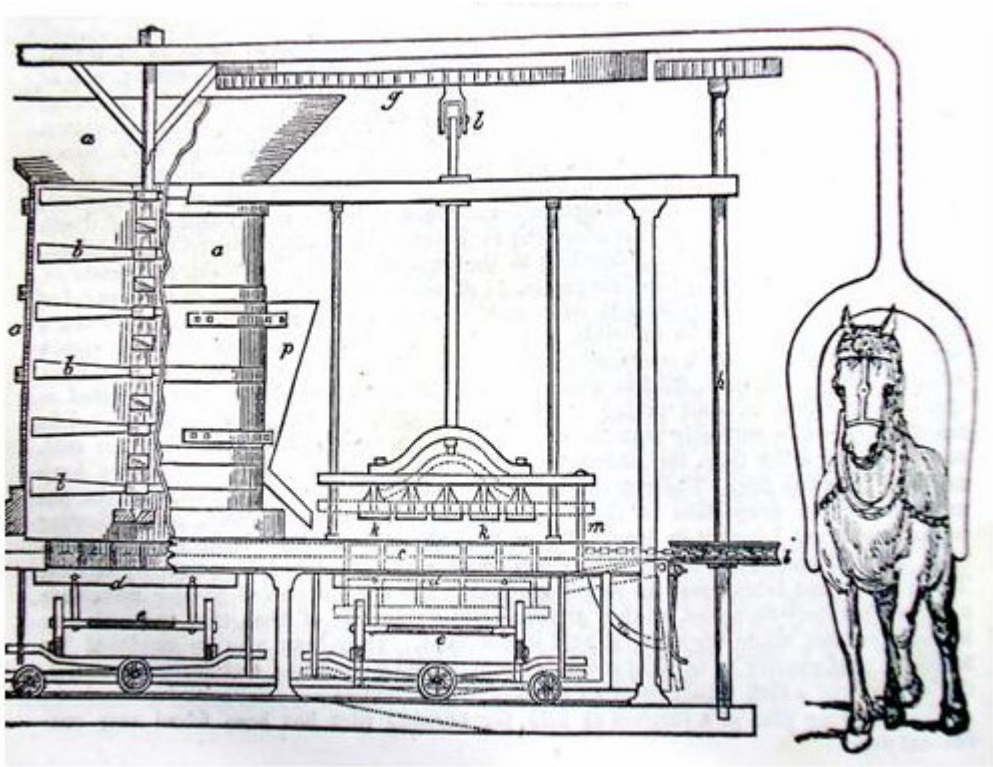
MIXING EQUIPMENT

EARLY MIXING

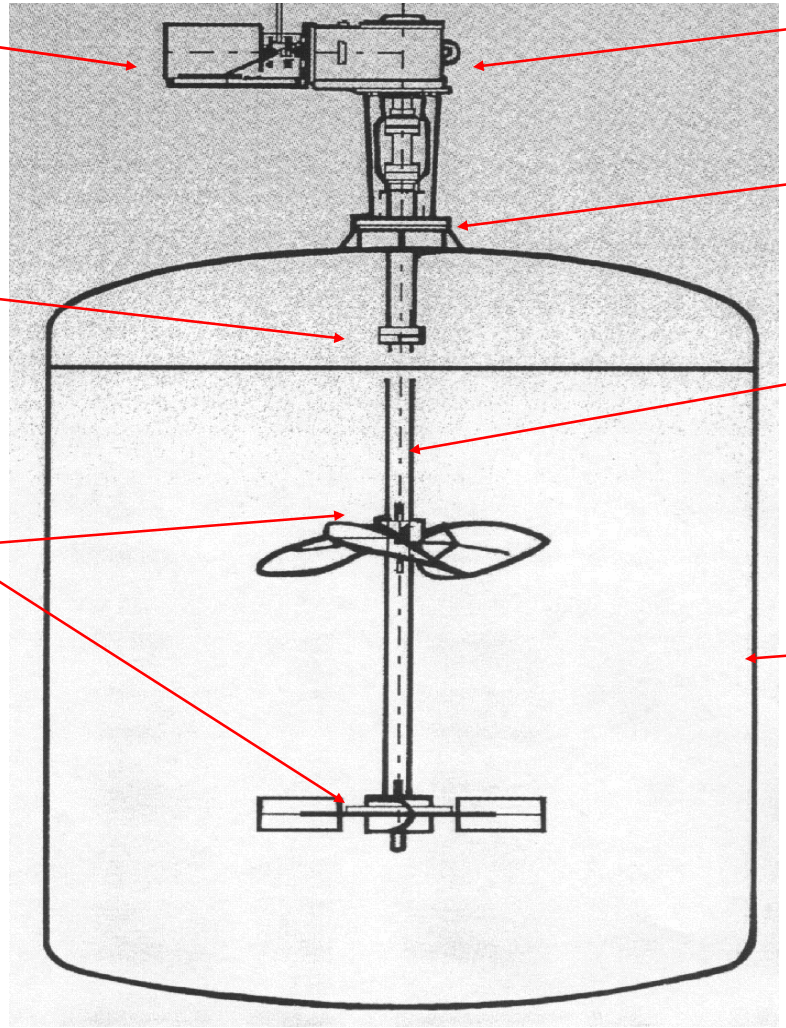
MODERN MIXING



ONE HORSEPOWER



Typical Mixing Equipment



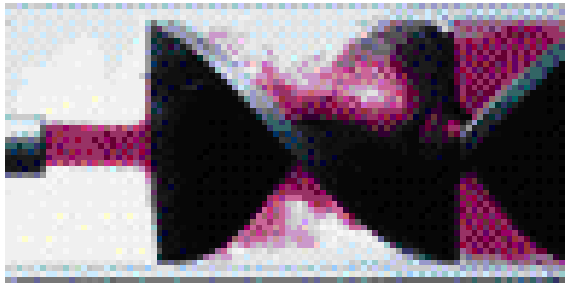
MIXING EQUIPMENT types

- Mechanical agitators in vessels
  - vertical
  - angled or side mounted
- Horizontal
  - side entering - mechanical
  - jets
- Gas mixed

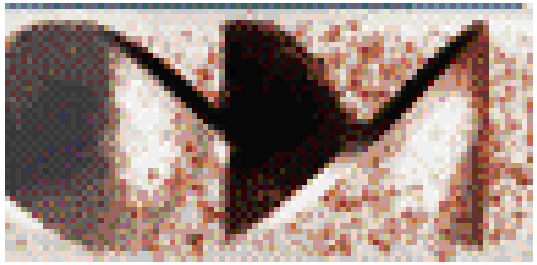
MIXING EQUIPMENT types

- Pipelines
  - motionless mixers
  - mechanical
- High Speed Dispersers
  - blades
  - rotor stator
  - in line and in tank
- Extruders

Motionless Mixers

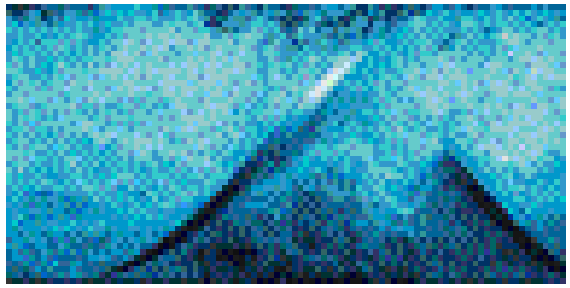


Turbulent Blending



Liquid-liquid dispersion

Laminar Blending



Gas-liquid dispersion

Dynamic In-line Mixers

An inefficient pump

COMMENT ON PICTURES

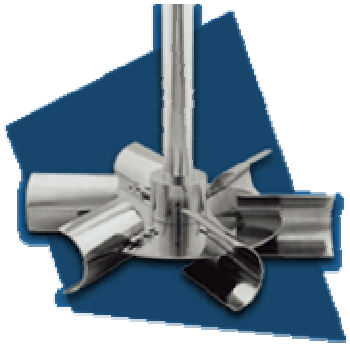
- Many pictures developed by Computational Fluid Dynamics – a mathematical solution to the equations of motion presented visually
- CFD can produce pictures that are very hard to get experimentally
- Not all CFD gives correct pictures as will be discussed later on
- When we show CFD pictures they will be correct and better than anything experimental
- LDA gives similar pictures but experimental

MIXING EQUIPMENT

- VESSELS
- Right cylinders
  - Z/T = 1.0
- Ponds and lagoons
  - Z/T <<<<<< 1.0

MIXING EQUIPMENT
impellers

- Propellors
- Paddles
- Turbines D/T 0.25 to .60
  - flat blades - Rushton turbines with disk
  - angled blades
  - hydrofoils
- Anchors D/T 0.9 to 0.95
- Helical Ribbons and Screws D/T .95-.98



RADIAL IMPELLERS

* RUSHTON

* CD-6 SMITH

* CHEMINEER -B-6

EVOLUTION OF THE HYDROFOIL

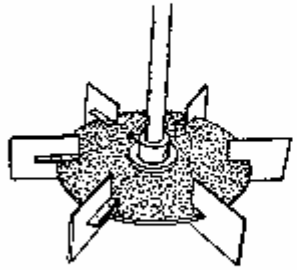
PROPELLOR

PBT - A200 - MFT - etc.

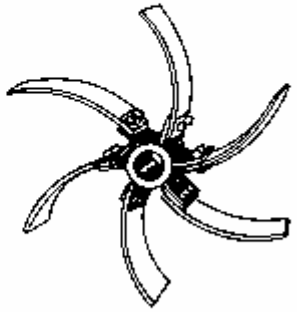
A310

A420

Radial Flow Impellers

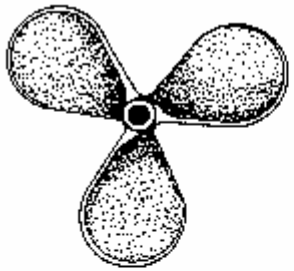


Disk Style Flat Blade Turbine
Commonly Referred to as
the Rushton Impeller

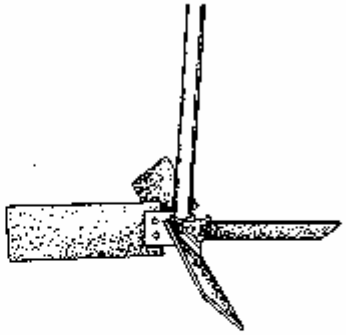


Sweptback or Curved Blade Turbine
(a Spiral Turbine)

Axial Flow Impellers



Propeller



45° Pitched Blade Turbine

Figure 1.4 Turbulent impellers.

IMPELLER CHARACTERISTICS

- Describe impellers by a set of numbers
- Power Number
- Flow Number
- Zweitering constant for solids suspension
- Cavitation number for gas liquid
- Etc.
- These numbers often involve tank geometry parameters in addition

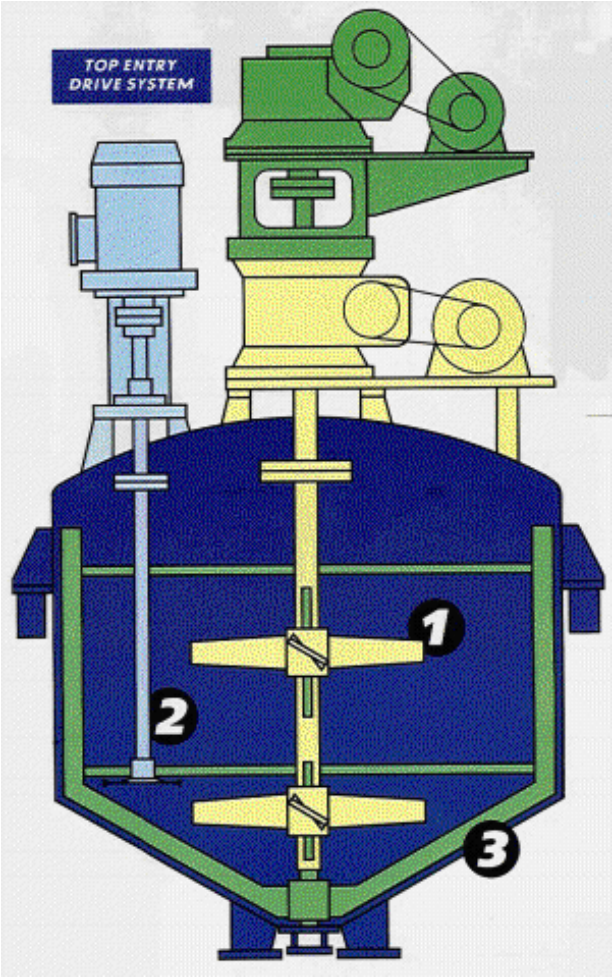
IMPELLER CHARACTERISTICS

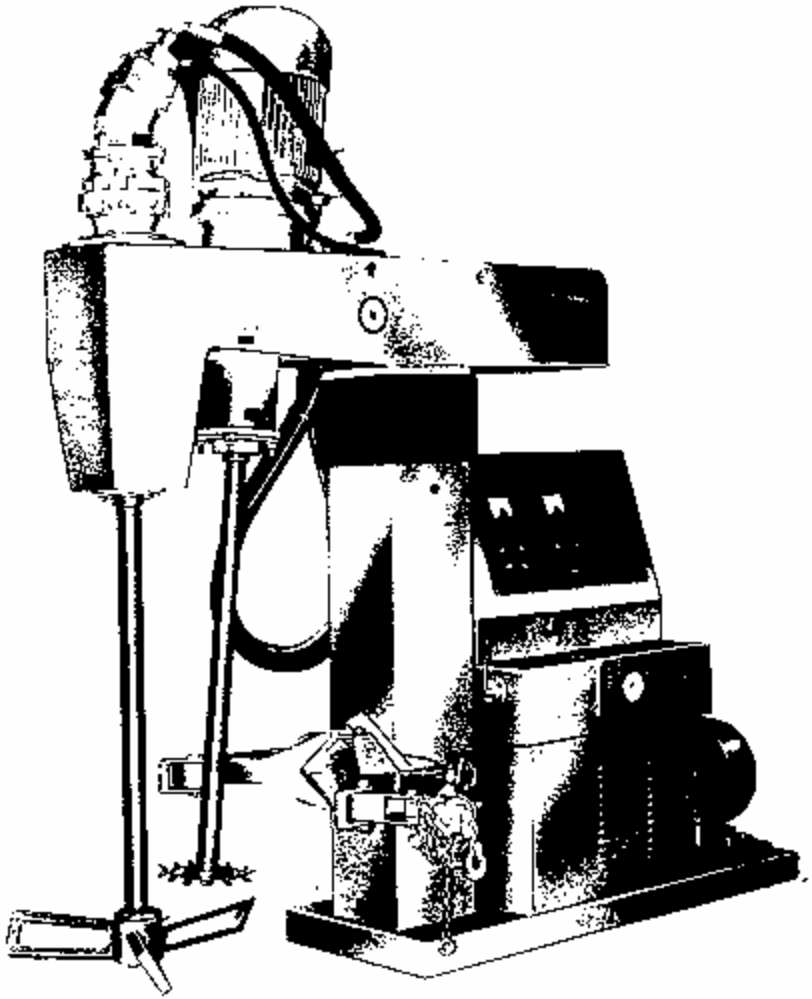
- Compilations of Impeller Numbers
  - Vendors
  - HIM
  - John Smith
  - PostMixing – www.postmixing.com
  - Various papers

MULTIPLE IMPELLERS

- Often multiple impellers are used on one shaft
  - Changing liquid height
  - High aspect ratio
  - Uniformity required near the top – reaction or solids suspension
  - Uniform distribution of energy - fermenters
  - About 80 % of all agitators made by Lightnin have multiple impellers
- Multiple shafts are sometimes used (batch)
  - Material changes with time

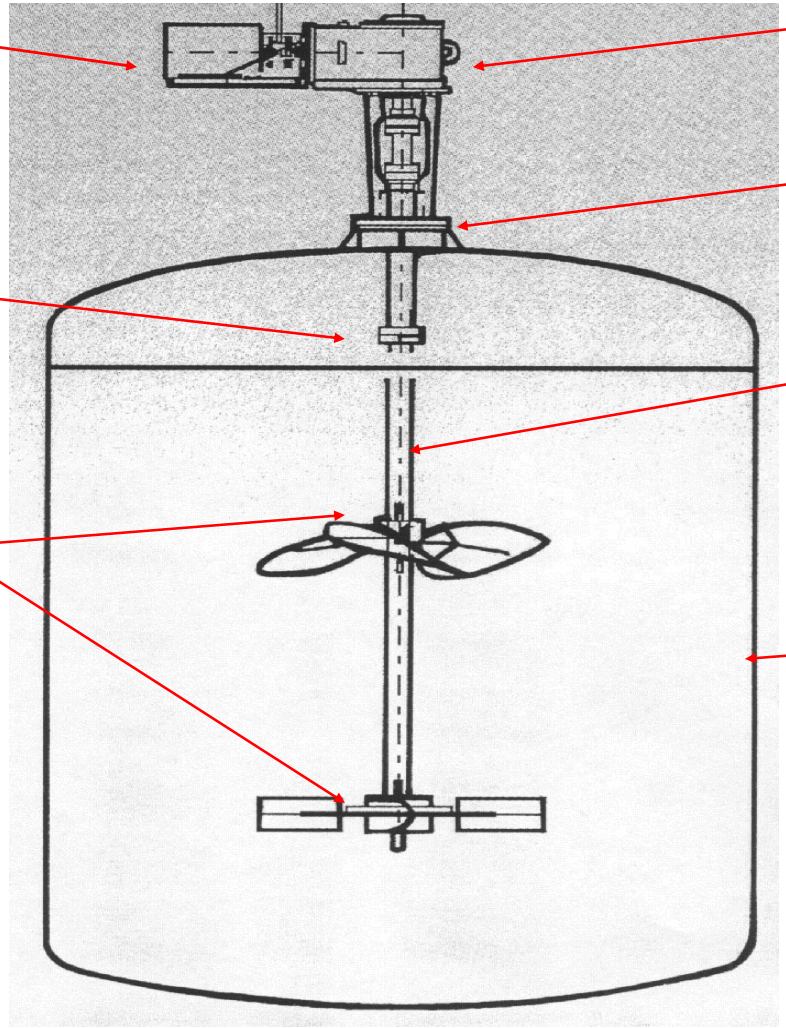
Combination





DUAL BLADE - HIGH AND LOW SPEED DISPERSER

Typical Mixing Equipment



MIXING EQUIPMENT

- MOTORS
  - rotating electrical 1500 to 1800 RPM

- Variable speed
  - old - mechanical
  - new - frequency control on motor
  - constant torque P/N
  - can only go down

MIXING EQUIPMENT

- GEAR BOXES
- reduce speed to more optimum speeds
  - 30 to 100 RPM
- Sturdy clock works
- Built for the use
  - high torques
  - high bending moments

SEALS

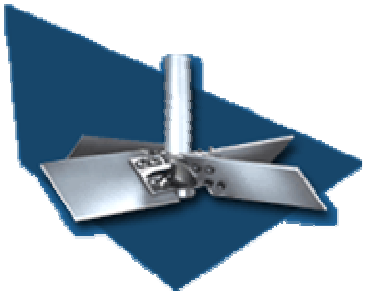
- Keep the contents inside the vessels against pressure through a rotating interface
- Keep the outside from getting in against vaccum
- Rotating and sealing is a challenge
  - High resistance path way
- Packing
- Mechanical seals – single for double
- Can be very expensive – mechanical engineering

MIXING EQUIPMENT

- BAFFLES
- Four vertical
- 1/10 to 1/12 tank diameter
- Off set 1/6 to 1/10 baffle width
- Angled mount
  - small vessel - easy duty
  - Take more power – can cause stagnation

MIXING EQUIPMENT
Flow Patterns

- Radial
  - FBT and Rushtons
- Axial
  - Propellors
  - hydrofoils
  - pitched turbines



AXIAL AND RADIAL FLOW

MIXING EQUIPMENT

- VERY HIGH VISCOSITIES
- Kneaders - batch - horizontal shafts
- Extruders - continuous
  - single screw - pumps
  - twin screws - pumps
  - feeders, melters, pumps, mixers

HIGH VISCOSITY

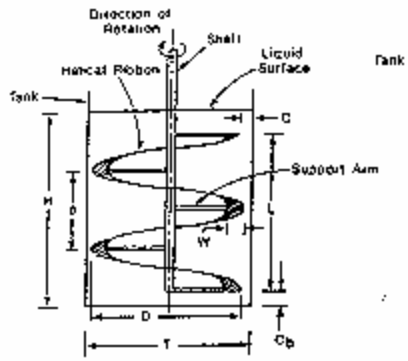
IMPELLERS

* anchor

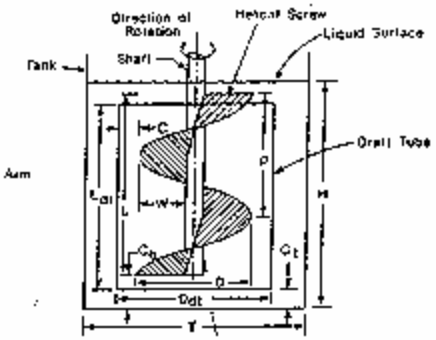
* screw

* single helix

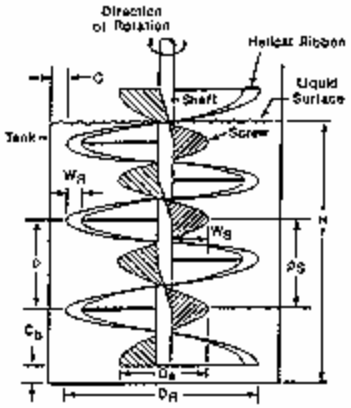
*double helix



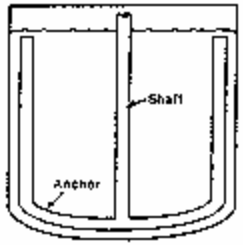
Helical Ribbon Impeller
Showing Geometric Variables
(Helical Ribbon Pumping Down at Wall)



Helical Screw Impeller
in a Draft Tube Showing
Geometric Variables
(Pumping Up in Center)



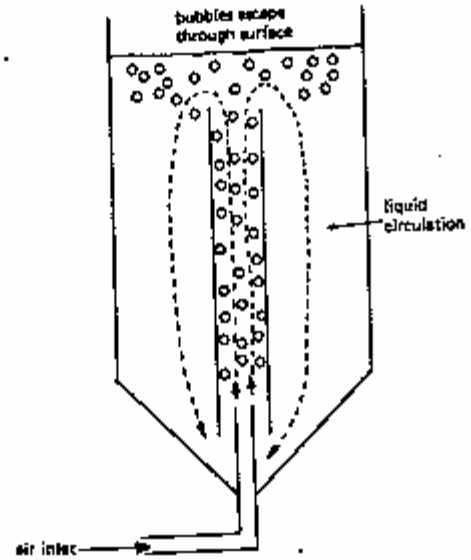
Helical Ribbon Screw Impeller
(Helical Ribbon Pumping Up at Wall
with Screw Pumping Down in Center)



Anchor Impeller

MIXING EQUIPMENT

- Non-mechanical
- TANKS
- jets - eductors
- air sparging
- PIPELINES
- jets
- motionless mixers



TANK MIXING WITH AIR

DRAFT TUBES

- Popular in some continuous crystallizers
- 0.5 TO 0.6 tank diameter
- Work best with fixed liquid level – continuous versus batch
- Give good top to bottom uniformity with only a lower impeller – more regular flow pattern
- Impeller acts as a pump
  - Impeller has a head flow curve
- AIChE Nov 07 meeting – Chemineer presentation – Eric Janz

SPECIAL MIXING EQUIPMENT

- HIGH SPEED DISPERSERS
- HOMOGENIZERS - WHISTLES
- In tanks / vessels and in line
- Colloid Mills
- Agitated Media Mills
  - disperse solids - add a third phase - media

HIGH SPEED DISPERSER

- SAW TOOTH NPO = 0.5

Dynamic In-line Mixers

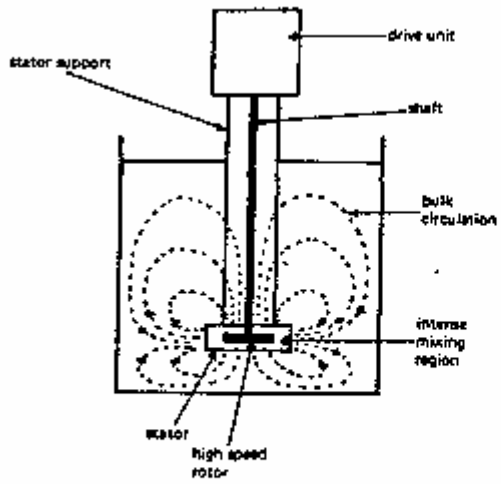
An inefficient pump

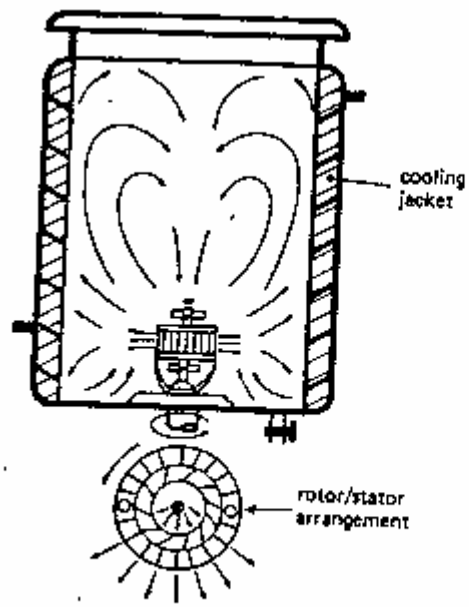
ROTOR STATOR MIXERS

- Many alternate names e.g. colloid mill, dispersator etc.
- Continuous devices or tank type
- Run as once through or on recycle
- Configuration
  - High speed rotor
  - Non-moving stator with perforations
  - Many variations

ROTOR STATOR MIXERS

- Operating parameters
- Tip speed
  - 4000 ft/min or 22 m/s
  - 18000 ft/min or 100 m/s
- Power per unit volume
  - 10 kw/kg


drive unit
stator support
shaft
bulk
circulation
intense
mixing
region
stator
high speed
rotor


cooling
jacket
rotor/stator
arrangement

HIGH PRESSURE HOMOGENIZERS

- Pump fluid up to a high pressure
  - Many atmospheres
- Pump through a nozzle
  - Sometimes with a baffle plate
- Run continuously or batch often with recycle
  - Multiple passes
- Some manufacturers
  - Sonic – sonolator
  - Microfluidics
  - Niro
  - APV Gaulin
  - Premier

MIXING EQUIPMENT

- Multiple Shaft Mixers
- Common in batch service
  - food and cosmetic
  - two or three separate shafts
- Slow speed - anchor - acts as baffle
- Medium speed turbine
- High speed - disperser

GLASS LINED EQUIPMENT

- Particularly popular in pharmaceutical industry
  - As much as 80 percent of reactors
- Glass lining peculiarities
- All entrances through top
- Dip tubes and thermocouples
- Not many openings
- Vertical cantilevered baffles – overhung
- Few if any baffles

GLASS LINED EQUIPMENT

TRANSITIONAL REGIME MIXING

- Relatively new development out of Japan
- Large blade widths and diameters
- Slight angles
- A fair amount of data on Npo and mix time

MAX BLEND

MIXING EQUIPMENT selection

- Desired process result
- Continuous or batch
- Size of volume
  - pipe
  - vessel or tank
- Viscosity, phases, intensity, time

MIXING EQUIPMENT selection

- Many possible mixer selections will give the desired process result
- Secondary considerations determine "best"
  - cost - guidelines
  - retrofit - existing
  - energy - time
  - volume or time

MIXING EQUIPMENT selection

- Most common
- Low to medium viscosity
- Tanks with hydrofoils or turbines
- blending, dispersions, reaction
- batch or continuous
- Avoid special equipment
  - hard to justify

DESIGN VERSUS RATING

- Equations in this course tell how to rate a given design
- Picking a design to rate is the engineering art – experience
- Many designs will work – give the process result
  - Best is relative based on other criteria

COST OF AGITATION

- Investment = torque = power/speed

- Operating cost = power

- Always a balance based on cost of power
- versus value of product/production

GUIDELINES

- Tip Speed
- Power per Unit Volume/Mass
  - turbines 3-4 m/s .2-.6 KW/m3
  - 600 – 700 fpm 1-5 HP/1000 gal
  - HSD 6-27 m/s 10-14 W/KGm
  - 1000 – 5000 fpm
  - Dispersers 20-40 m/s 20-40 W/KGm
  - 4000-8000 fpm 0.2-0.5 HP/gallon
- as scale increases tip speed changes slightly P/V often goes down

UNIT HINT

- ENERGY DISSIPATION
- SI watts/kgm or kwatts/m3 (for water)
- English units Horsepower/1000 gallons
- Turbulence theory suggests
  - Pm - proportional to V^3/ D
    - Where Pm is power per unit mass and V and D are appropriate velocity and dimensions

SCALE UP

- Run small scale experiments to predict large scale performance
- Common question in mixing
- If know enough often can design and do not need scale up
- Will discuss throughout the course
- Will summarize at the end

COMPUTATIONAL FLUID MECHANICS

- A recently developed tool for modeling fluid dynamic situations
- Quite popular in mixing
- Based on solving equations of motion
- Many assumptions – particularly for multiple phase flow
- Will summarize at end.

END M3

M4
MIXING CONCEPTS

Turbulence

LAMINAR AND TURBULENT MIXING

coffee

paint

Turbulence

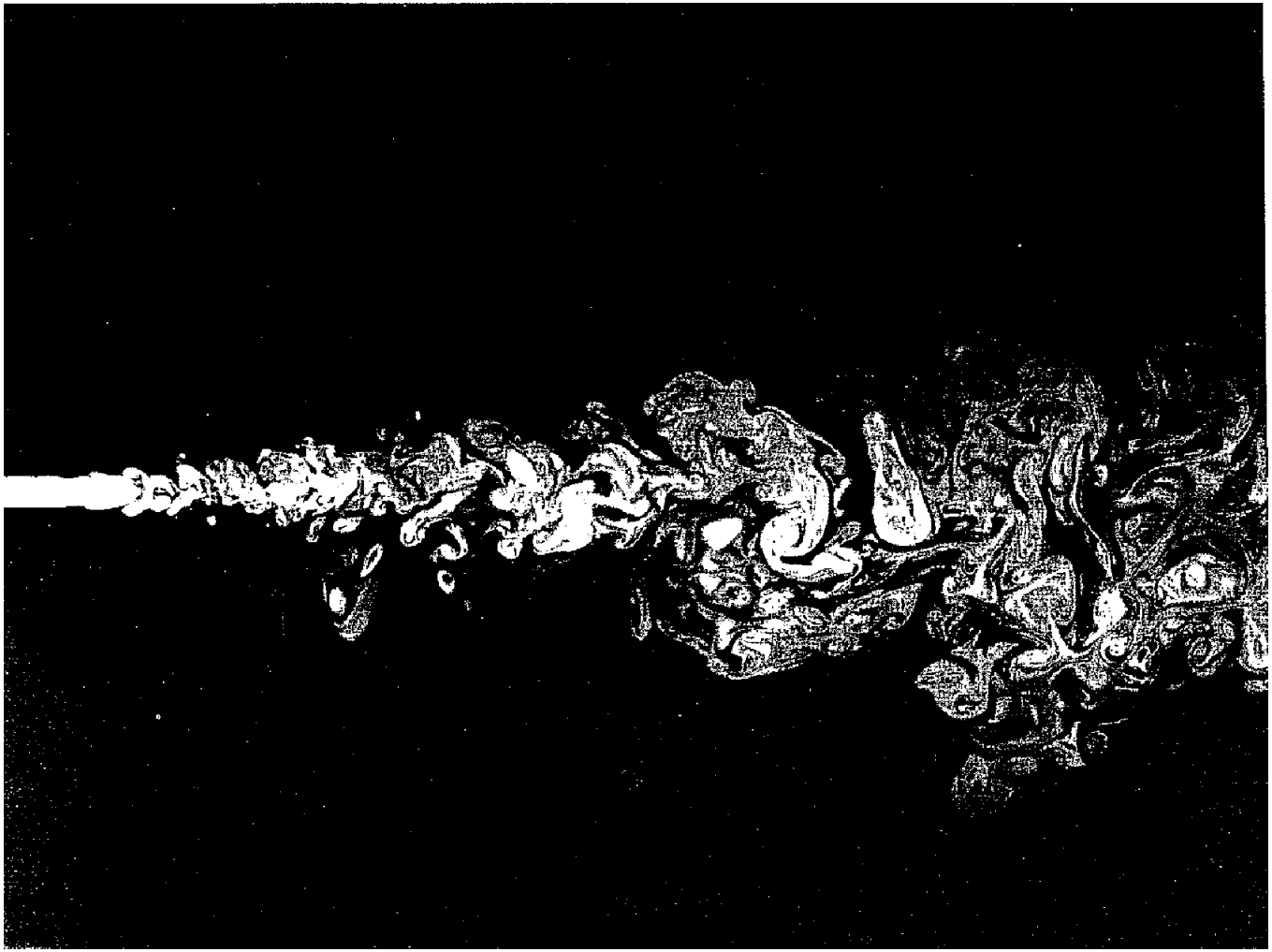
What is turbulence?

1. The state or quality of being turbulent; violent commotion, agitation or disturbance; disorderly character or conduct.

2. Of natural conditions: Stormy or tempestuous state or action.

3. Random fluctuations superimposed on mean velocity in flowing system.

A Turbulent Jet



Example - Reynolds' Experiment

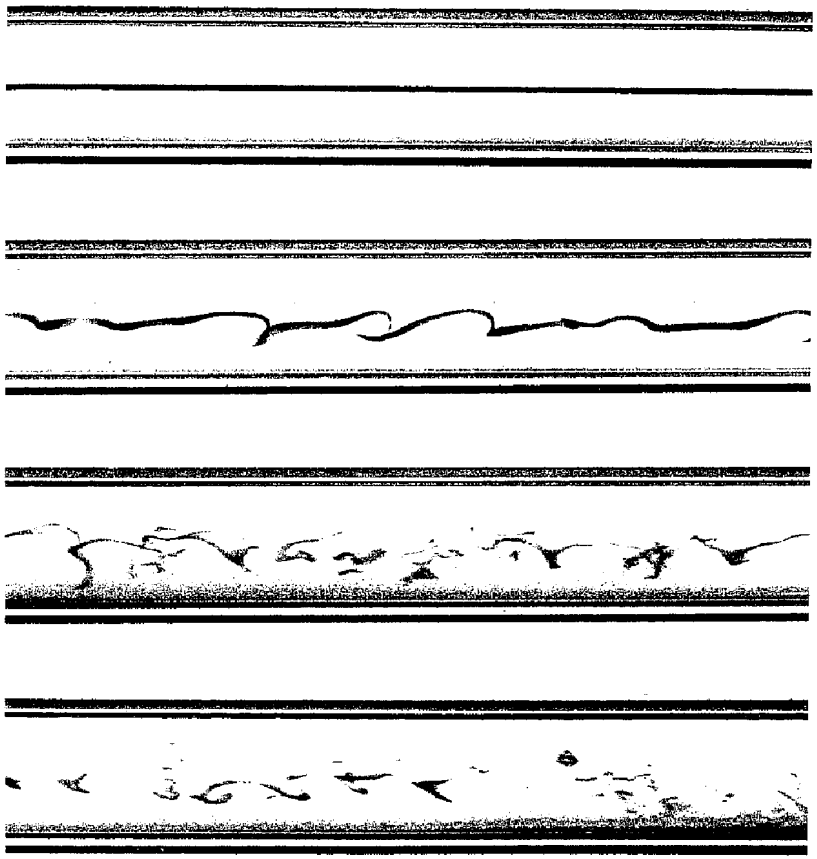
- Classic experiment performed in 1883.

- Set up co-axial horizontal pipes:
  - Colored water flows through central pipe.
  - Clear water through annulus.

- Gradually increase velocity of two streams and observe what happens.

Results

- Low velocity:
  - Central dyed stream flows parallel to pipe walls.
- Increasing velocity:
  - Central core starts to move radially.
  - Breaks-up.
- Higher velocity increases rate of core's dispersion.
- Increases rate of mixing.



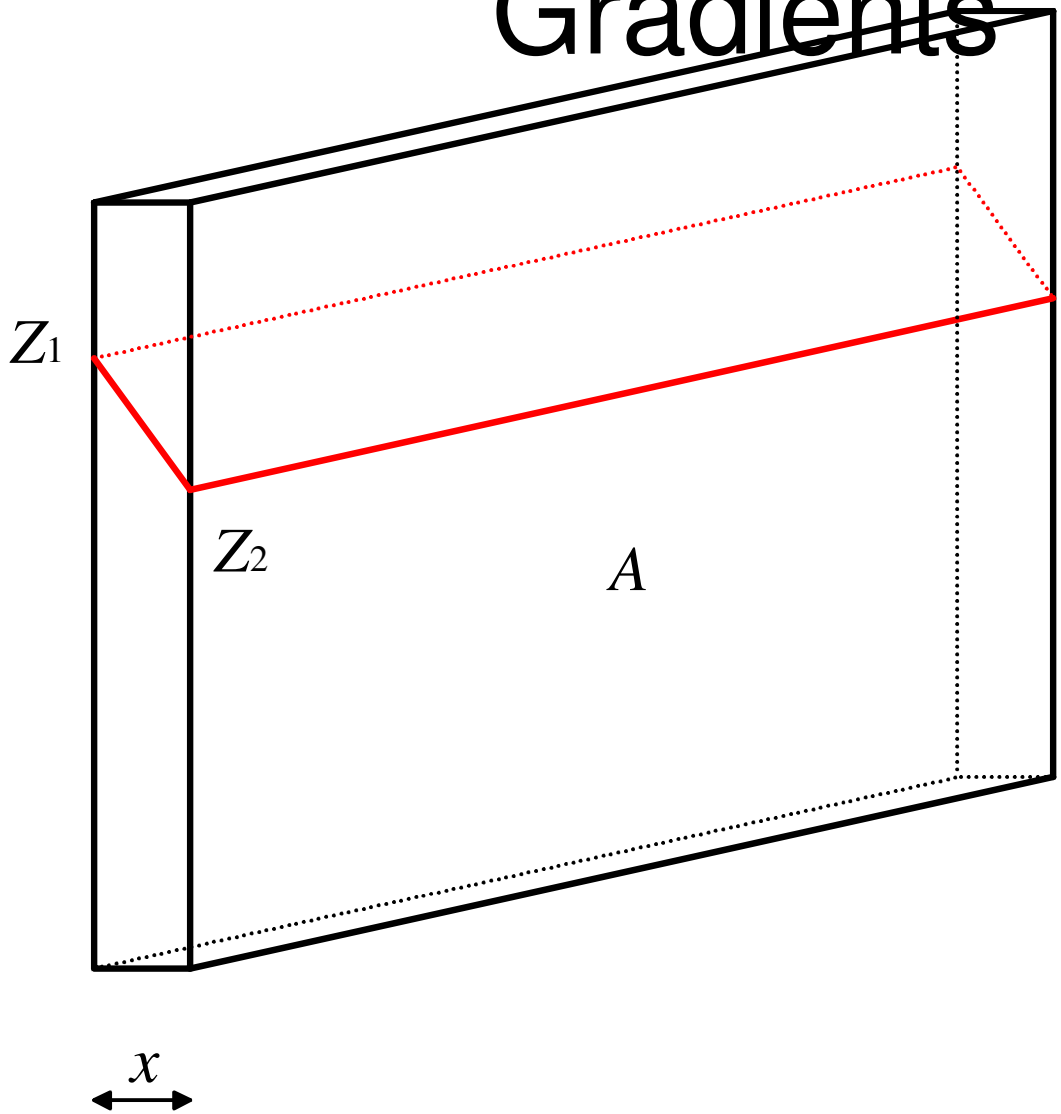
Reynolds Number

- Ratio of inertial forces in flow to viscous stress resisting flow.
- Inertia: $$I = \rho U^2$$
- Viscous stress: $$\tau = \mu \frac{U}{D}$$
- Reynolds No: $$Re = \frac{\rho U^2}{\mu \frac{U}{D}} = \frac{\rho U D}{\mu}$$

What is Happening?

- As velocity of streams increases, inertia dominates viscosity.
- Dyed central stream is mixing with outer clear stream.
- What is “driving force” that promotes mixing?
- A force is generated which transports the dye radially - perpendicular to the direction of flow.
- If no radial movement occurs, mixing can only happen by molecular diffusion. A slow process.
- Something else is diffusing. But what?

Gradients



$$\frac{Flux}{Area} = K\frac{Z_1 - Z_2}{x}$$

Transfer Processes

- Heat transfer: $$\frac{Q/t}{A} = k\frac{(T_2 - T_1)}{x}$$
- Mass transfer: $$\frac{M/t}{A} = D_{AB}\frac{(C_2 - C_1)}{x}$$
- Momentum transfer:

$$\frac{Mo/t}{A} = \mu\frac{(U_2 - U_1)}{x}$$

Momentum Transfer

- Force is rate of change of momentum per unit time.

$$F = \frac{Mo}{t}$$

- Driving force is velocity gradient:
  - Analogous to temperature or concentration gradient.

- Turbulent eddies carry momentum from regions of high to low velocity.

- As momentum is transferred, mass is transferred within the eddies.

$$j_A = \frac{1}{A}\frac{\mathrm{d}N_A}{\mathrm{d}t} = (D_{AB} + D_{EDDY})\frac{\mathrm{d}C_A}{\mathrm{d}x}$$

Eddy Diffusivity

- Eddy Diffusivity quantifies the rate at which momentum is transferred by turbulence.

- It is a property of the flow - not the fluid.

$$D_{EDDY} = u' l_E$$

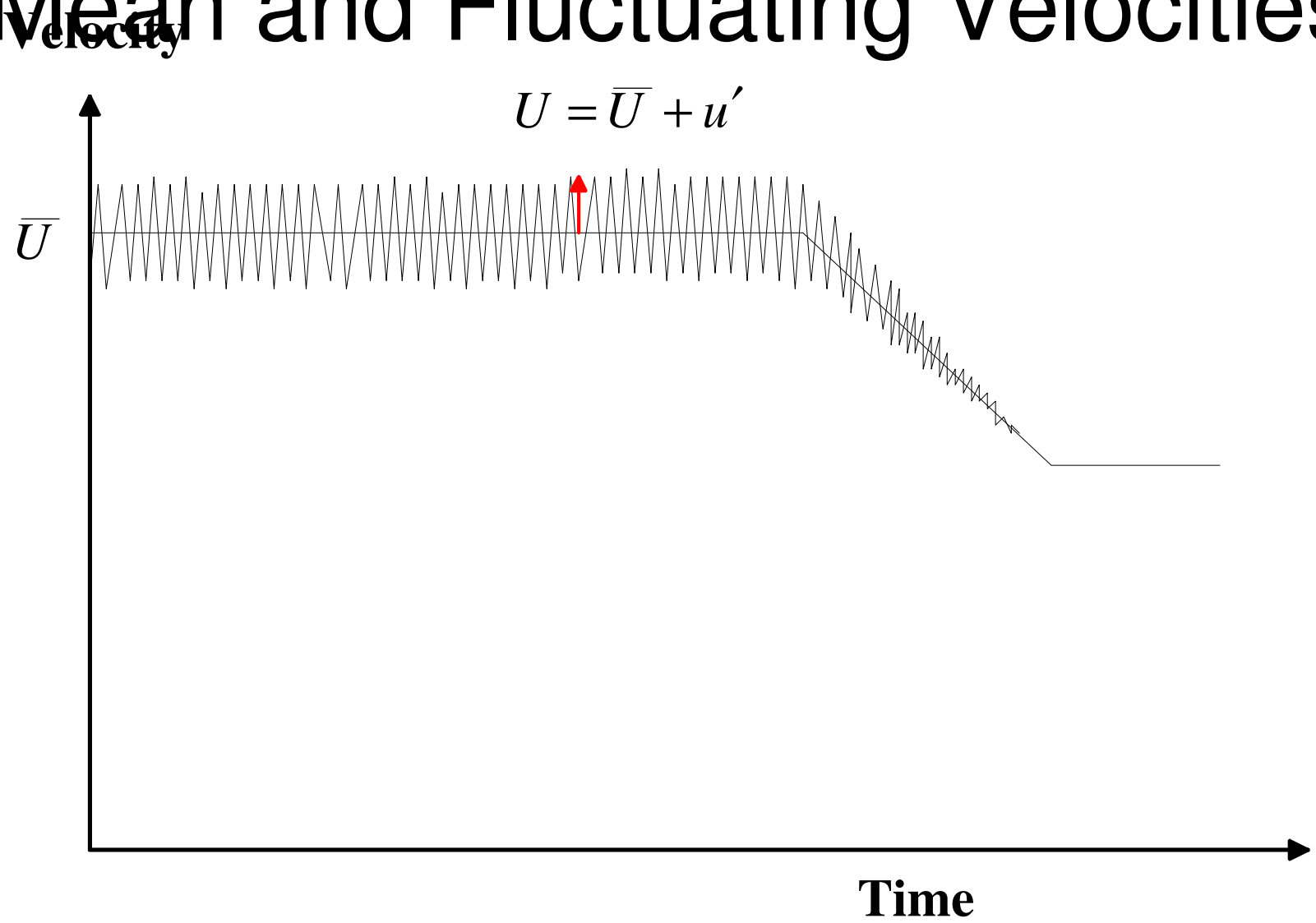
- What is u'?

- It is the fluctuating component of the fluid's velocity.

$$U = \overline{U} + u'$$

- In pipe flow, typically 5 % of mean velocity.

Mean and Fluctuating Velocities



Navier-Stokes Equation

- Laminar flow:
  - Four equations with four unknowns.
  - Can be solved.

- Turbulent flow:
  - Four equations with seven unknowns.
  - Cannot be solved directly.

- Need models to approximate turbulence quantities.

- Area of research and development.

- Computational Fluid Dynamics (CFD).

Example - Shear Stress

- Laminar flow:
  - Contribution due to mean velocities. $$\tau_V = \mu \frac{\mathrm{d}U}{\mathrm{d}x}$$
- Turbulent flow:
  - Contribution due to fluctuating velocities. $$\tau_T = \rho u'^2$$
- Total stress: $$\tau = \tau_V + \tau_T = \mu \frac{\mathrm{d}U}{\mathrm{d}x} + \rho u'^2$$
- Need to know values of fluctuating velocity.
- Need to make approximation ……...

Example - Shear Stress

- One approach (one of many) ……...

- The concept of “eddy viscosity”:

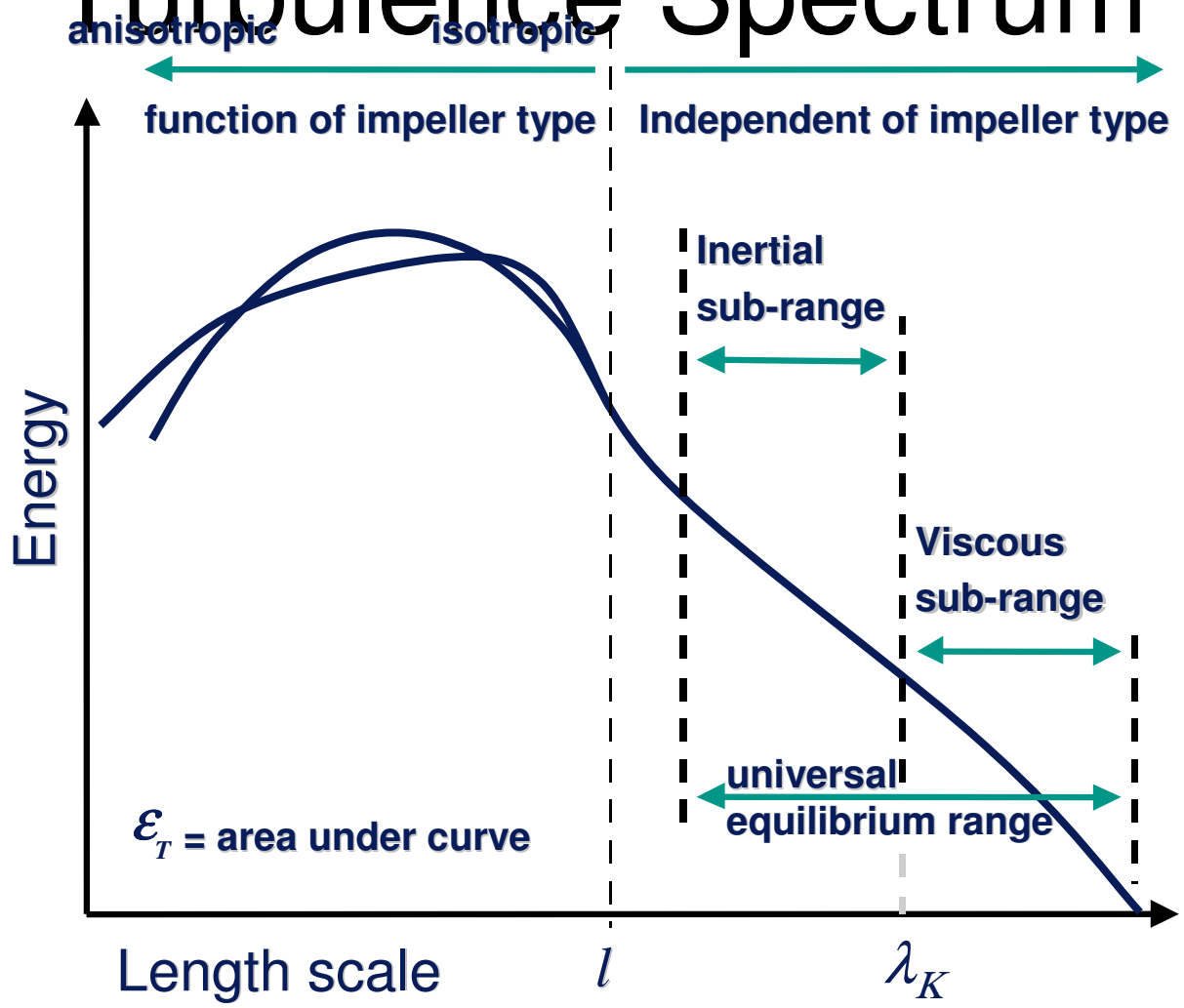
$$\tau = \tau_V + \tau_T = (\mu + \phi)\frac{\mathrm{d}U}{\mathrm{d}x}$$

- Now write total stress in terms of mean velocity gradient.

- ϕ is a property of the flow - not the fluid.

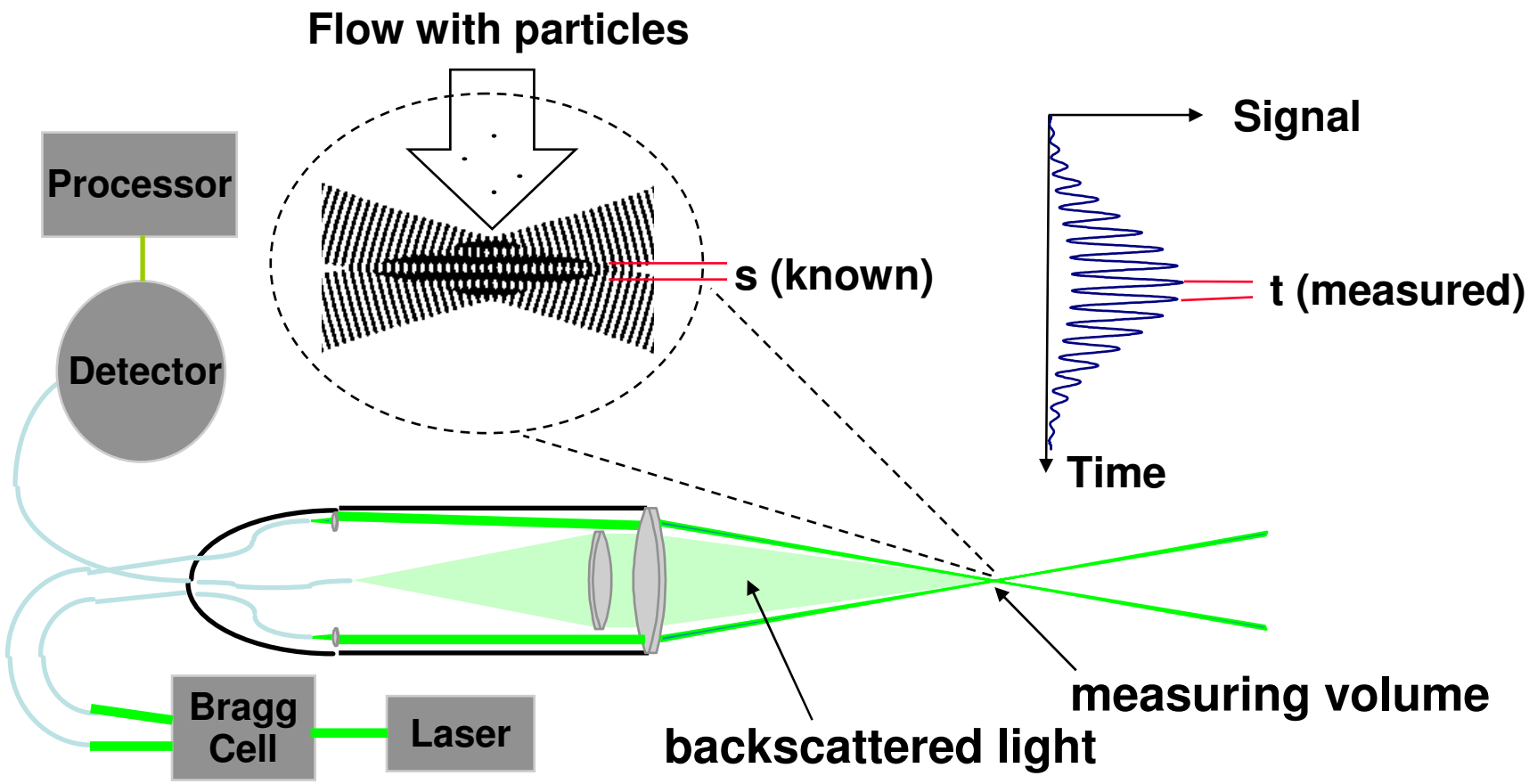
- The turbulent stresses are several orders of magnitude higher than viscous stresses.

Turbulence Spectrum



Measurement of Spectrum - LDA

Velocity = distance/time



Eddy Length Scales

- Largest eddies are determined by size of equipment:
  - Blade diameter.
  - Blade width.
  - Vessel.
- Smallest eddies are at the Kolmogoroff Length Scale.
  - Size determined by fluid viscosity and local energy dissipation rate:

$$\lambda_K = \frac{\nu^3}{\varepsilon}^{1/4}$$

$$Re_{EDDY} = \frac{u_K \lambda_K}{\nu} = 1$$

  - from dimensional analysis!
  - At Kolmogoroff Length Scale mixing occurs by diffusion between eddies.

Kolmogoroff Length Scale

$$Re_{EDDY} = \frac{u_K \lambda_K}{\nu} = 1$$

$$u_K \lambda_K = \nu$$

$$\varepsilon = \frac{u_K^3}{\lambda_K} \qquad \text{or:} \qquad u^3 = \varepsilon \lambda_K$$

$$\varepsilon \lambda_K^4 = \nu^3$$

$$\lambda_K = \left(\frac{\nu^3}{\varepsilon} \right)^{1/4}$$

Energy Containing Eddies

- Most mixing processes take place at a scale between the large and Kolmogorof eddies.

- These are called the “energy containing eddies”:

$$u' \sim (\varepsilon l_E)^{1/3}$$

- ε is the local energy dissipation rate:
  - Energy / time mass $\rightarrow$ power input per unit mass.

- ε varies with position in an agitated vessel:
  - High near impeller - low near walls and liquid surface.

- Use mean or local depending on process.

Blending Miscible Liquids

- Expect blending rate to be proportional to eddy diffusivity?

$$\theta \sim \frac{T^2}{D_{EDDY}} \sim \frac{T^2}{u' l_E}$$

$$\theta \sim \frac{T^2}{\varepsilon^{1/3} l_E^{4/3}}$$

- Macro-scale process so, $l_E \sim D$:

$$\theta \sim \frac{T^2}{\varepsilon^{1/3} D^{4/3}} \sim \varepsilon^{-1/3} \left(\frac{T}{D} \right)^{4/3} T^{2/3}$$

- Supported by experimental results:
  - Implications for scale-up?
  - Different exponent on T / D ratio.

Drop or Bubble Formation

- Inertial forces due to turbulent fluctuations of flow are balanced by viscous forces and interfacial tension:

$$\rho_C (u')^2 \sim \mu_D \frac{u'}{d} + \frac{\sigma}{d}$$

- If dispersed phase viscosity is low:

$$(u')^2 \sim \frac{\sigma}{\rho_C d}$$

$$(\varepsilon l_E)^{2/3} \sim \frac{\sigma}{\rho_C d}$$

- Drops will be affected by eddies of the same size:
  - Convected by larger eddies.
  - Smaller eddies will have no effect.

Drop or Bubble Formation

- Substitute droplet size for eddy length scale:

$$(\varepsilon d)^{2/3} \sim \frac{\sigma}{\rho_C d}$$

$$d^{5/3} \sim \frac{\sigma}{\varepsilon^{2/3}\rho_C}$$

$$d \sim \varepsilon^{-2/5}\left(\frac{\sigma}{\rho_C}\right)^{3/5}$$

- This relationship has been found experimentally in stirred tanks, motionless mixers and high-speed dispersers:
  - Implications for scale-up?

Future Discussions

- Turbulence determines rates of many mixing processes.
- Experimental results and correlations show important factors in determining mixing rates (process results).
- We will always attempt to explain results from a fundamental physical basis:
  - Look for functionality.
  - Need to do experiments to find constants of proportionality.
- If such an explanation is possible, we have much higher degree of understanding and confidence:
  - Especially when looking at design and scale-up of equipment.

A NOTE OF PHYSICAL PROPERTIES

- Fluid and solids physical properties
- Density – liquids and gases – solids crystal or chemical density
- Viscosity – the resistance to deformation – to be discussed in detail
- Thermal – heat capacity and thermal conductivity
- Surface – surface and interfacial tensions and others

MIXING AND GEOMETRY

- Mixing equipment is characterized by complex geometry and many geometric variables

- Certainly compared to previously studied geometries
  - Pipes, blunt bodies etc.

- Fluid dynamics in complex geometries

- Correlations are often very geometric dependent

Impellers

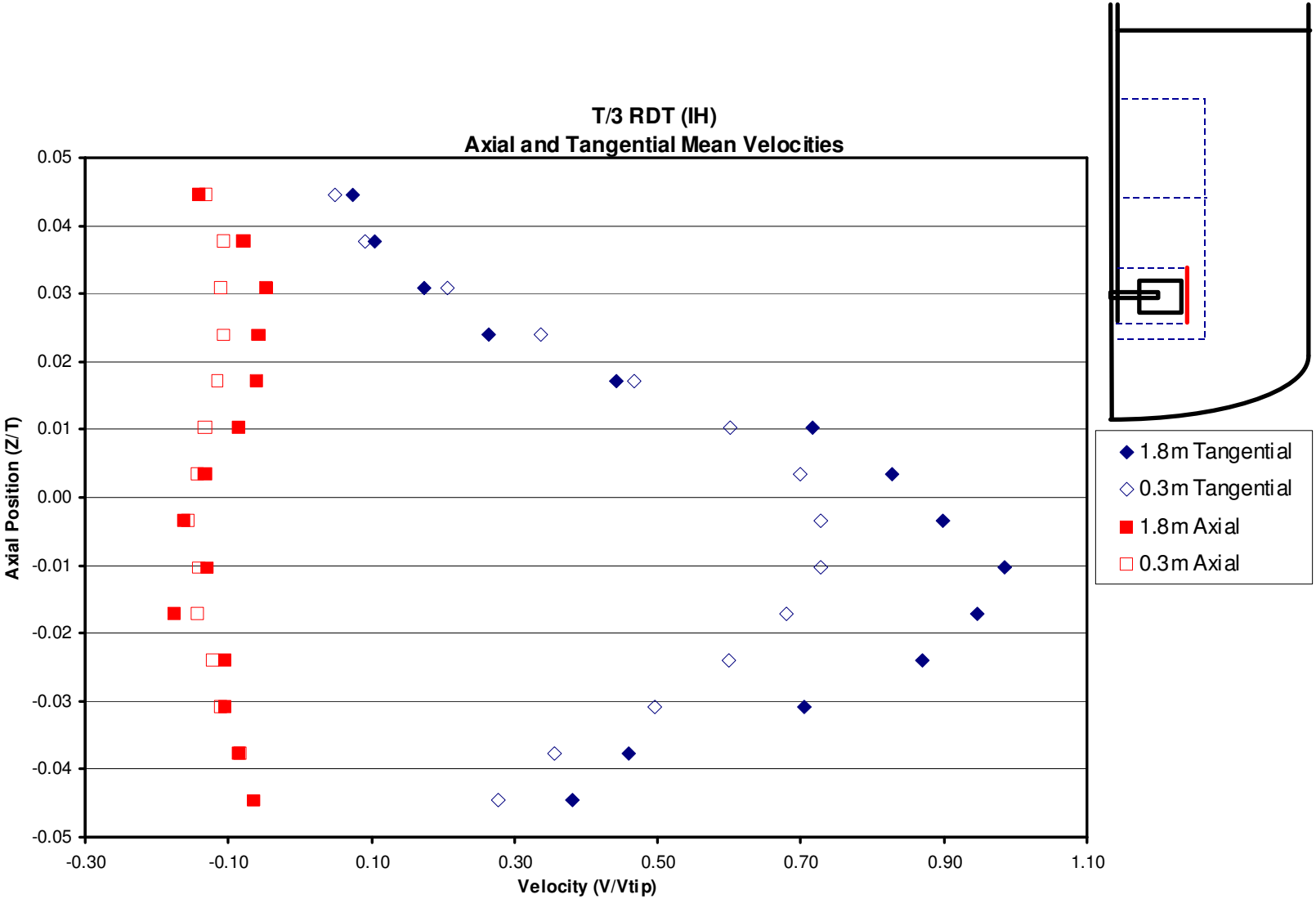
- An agitator's impeller is a high flow - low head pump.
- Many of the rules that apply design of agitators are analogous to those used for pump design and pipe flow.
- Relationship between power, flow and head.

$$Pa = Q\Delta H$$

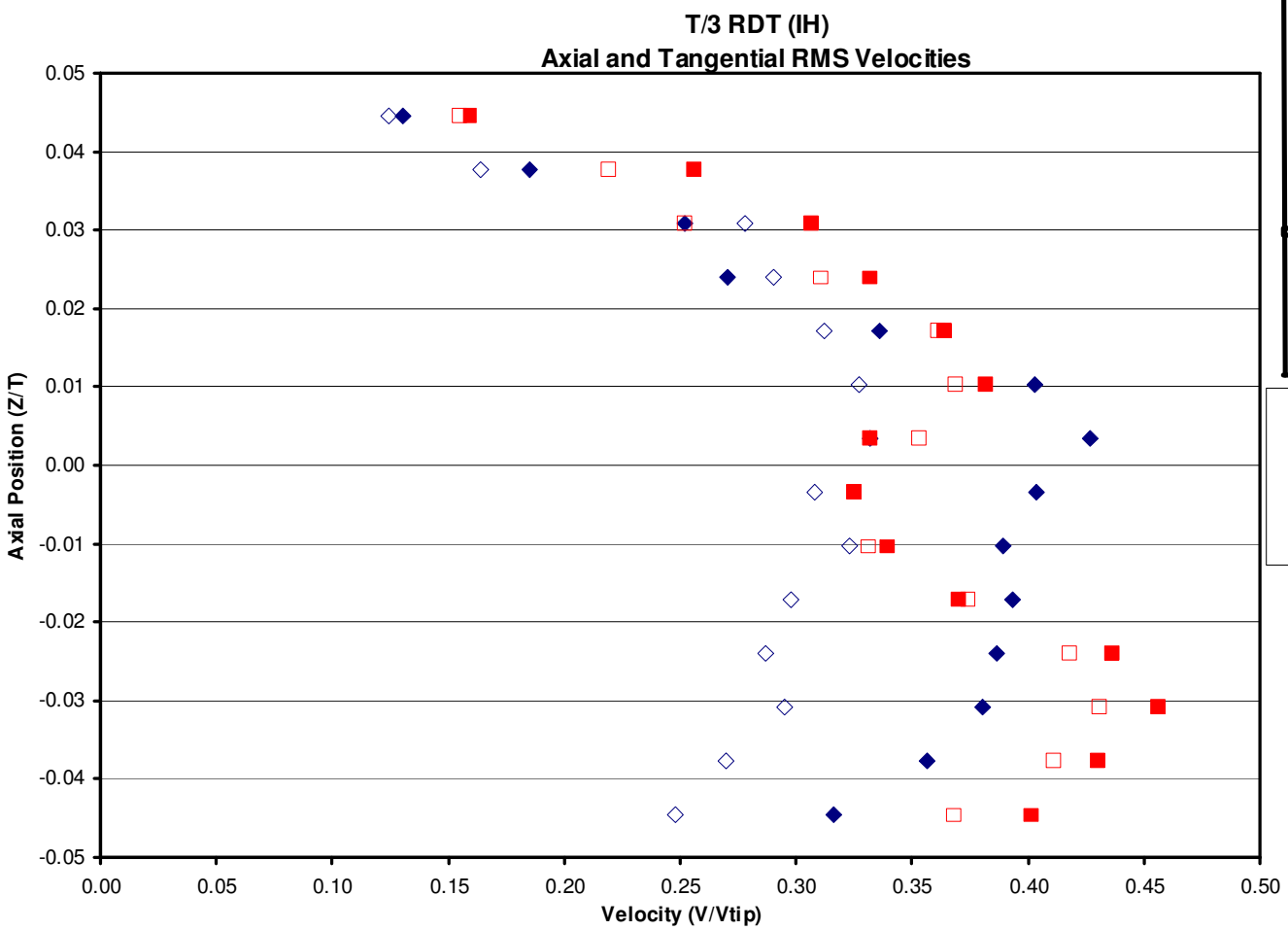
Flow

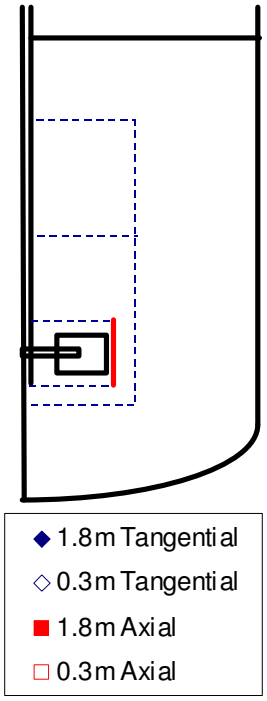
- How is flow measured?
- Laser-Doppler Anemometry.
- Define Flow number, *Fl*: $$Fl = \frac{Q}{ND^3}$$
- *Q* is measured.
- *N* is known.
- *D* is known.

Rushton Turbine - Mean

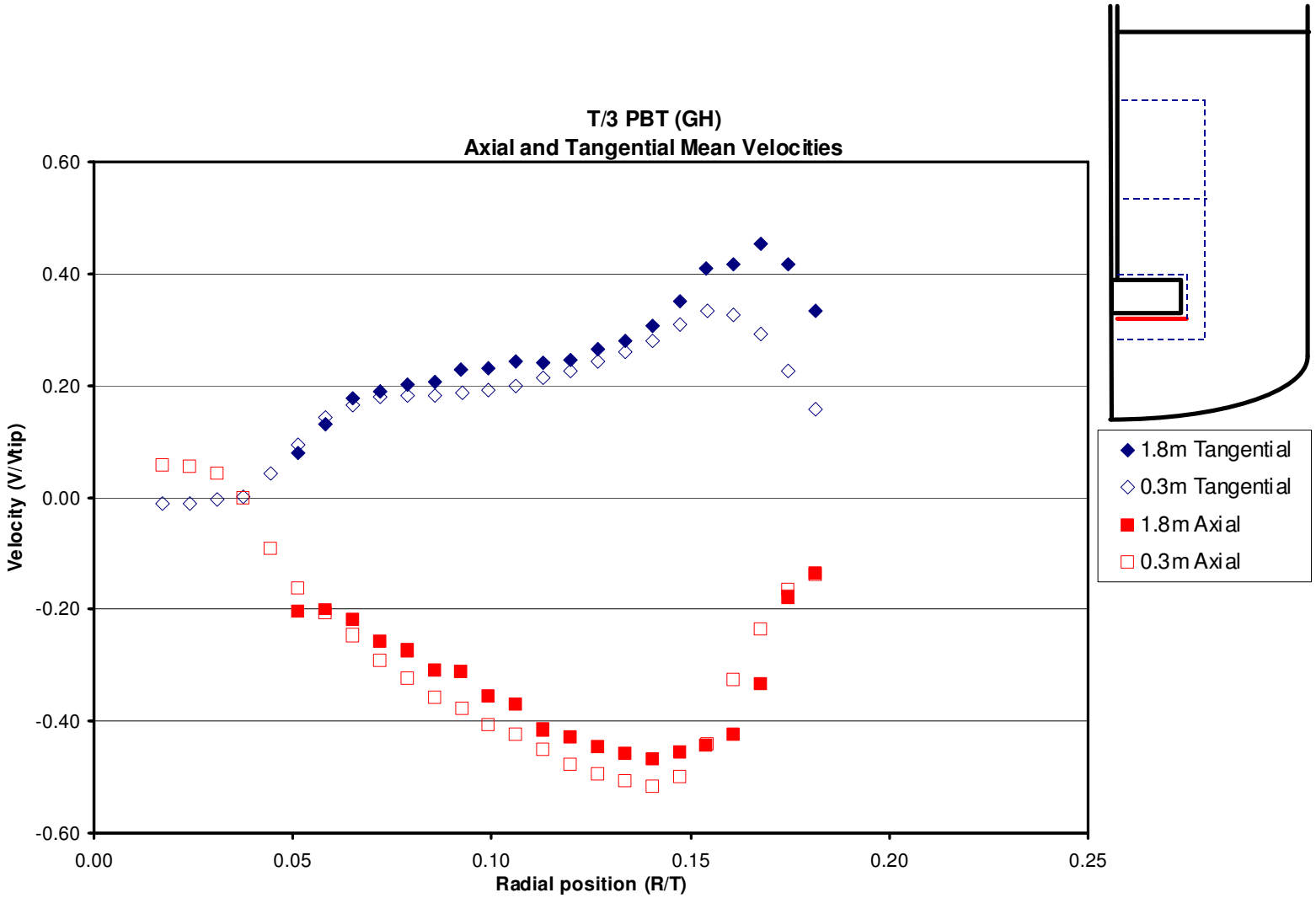


Rushton Turbine - Fluctuating

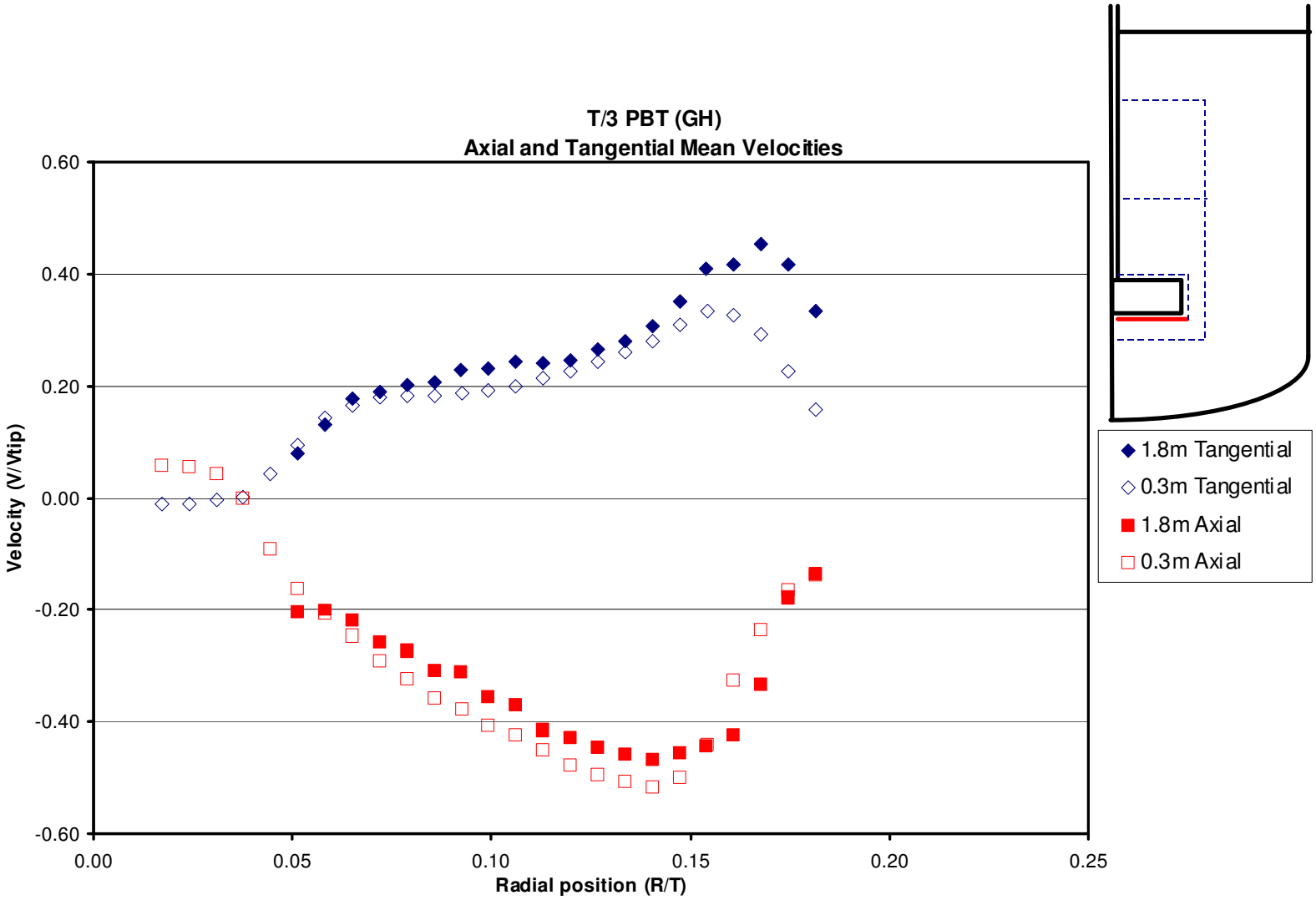




Pitched Blade Turbine - Mean



Pitched Blade Turbine -



Analysis

- Velocity measured at each position in discharge.
- Integrate velocities around discharge area.

$$Q = \int U \mathrm{d}A$$

- What are limits of discharge area?
- Leads to problems when comparing results from different labs.
- Small differences.

Primary Flow

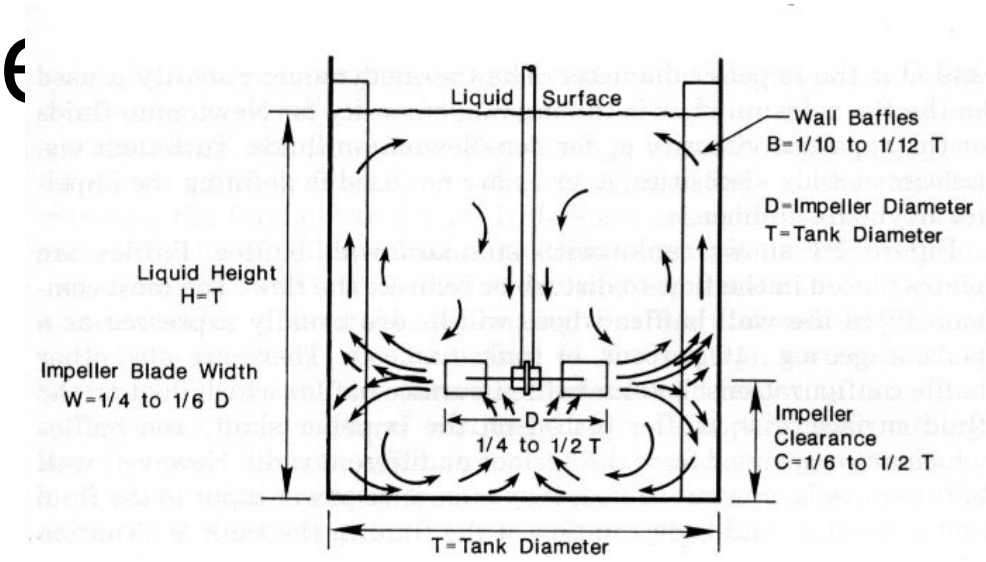
- Measure “primary” flow with LDA.

- This flow entrains surrounding fluid:
  - Secondary flow.
  - More on this when we talk about jet mixers.

- Entrained flow depends on primary flow and equipment geometry.

Discharge - Radial Flow Impe

- Discharge towards wall of tank.
- Draw fluid into impeller from above and below.
- Discharge area is vertical cylinder.
- Radial flow impellers:
  - Flat blade turbine.
  - Rushton turbine.
  - Smith turbine.
  - Bakker turbine.

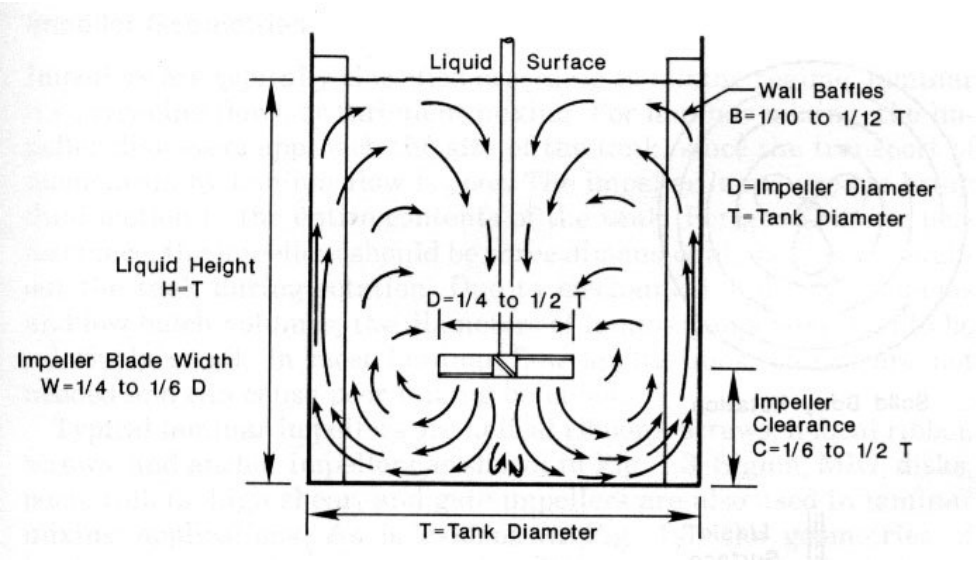


Discharge - Axial Flow Impellers

- Discharge towards base of tank or liquid surface.

- Draw fluid into impeller from above or below.

- Discharge area is horizontal circle.



- Axial flow impellers:
  - Pitched blade turbine - blade angle 30 - 60 degrees, 45 most common
  - Hydrofoils.
  - Marine propellers.

Typical Flow Numbers

Impeller Type	Flow Number, *Fl*
Radial (4 blades)	0.6
Radial (6 blades)	0.7 – 0.85
Axial (4 blades)	0.8
Axial (6 blades)	0.9
Propeller	0.5
A310 Hydrofoil	0.56

Prediction of Flow Number

- Correlation of flow number as a function of power number.

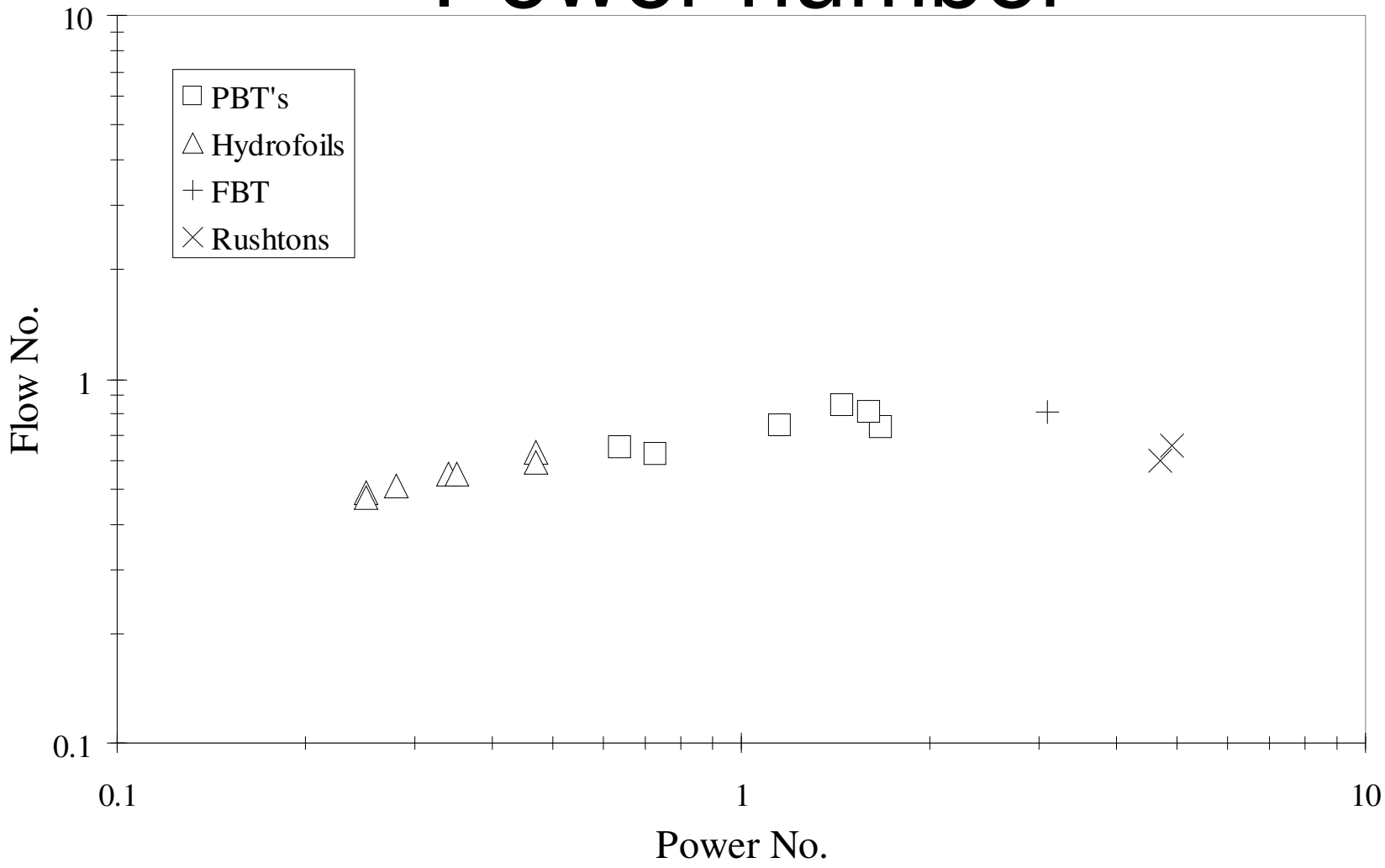
$$Fl = 0.77 Po^{1/3}$$

- Works for axial flow impellers:
  - Hydrofoils.
  - Pitched blade turbines.

- Does not work for radial flow impellers.

- <u>NOTE:</u> Only holds for turbulent regime.

Plot of Flow number versus Power number



Power

- Need to calculate power because:
  - Important for process results.
  - Determines size of equipment.
  - Operating cost.
  - Mechanistic / Theory e.g: power input per unit mass.

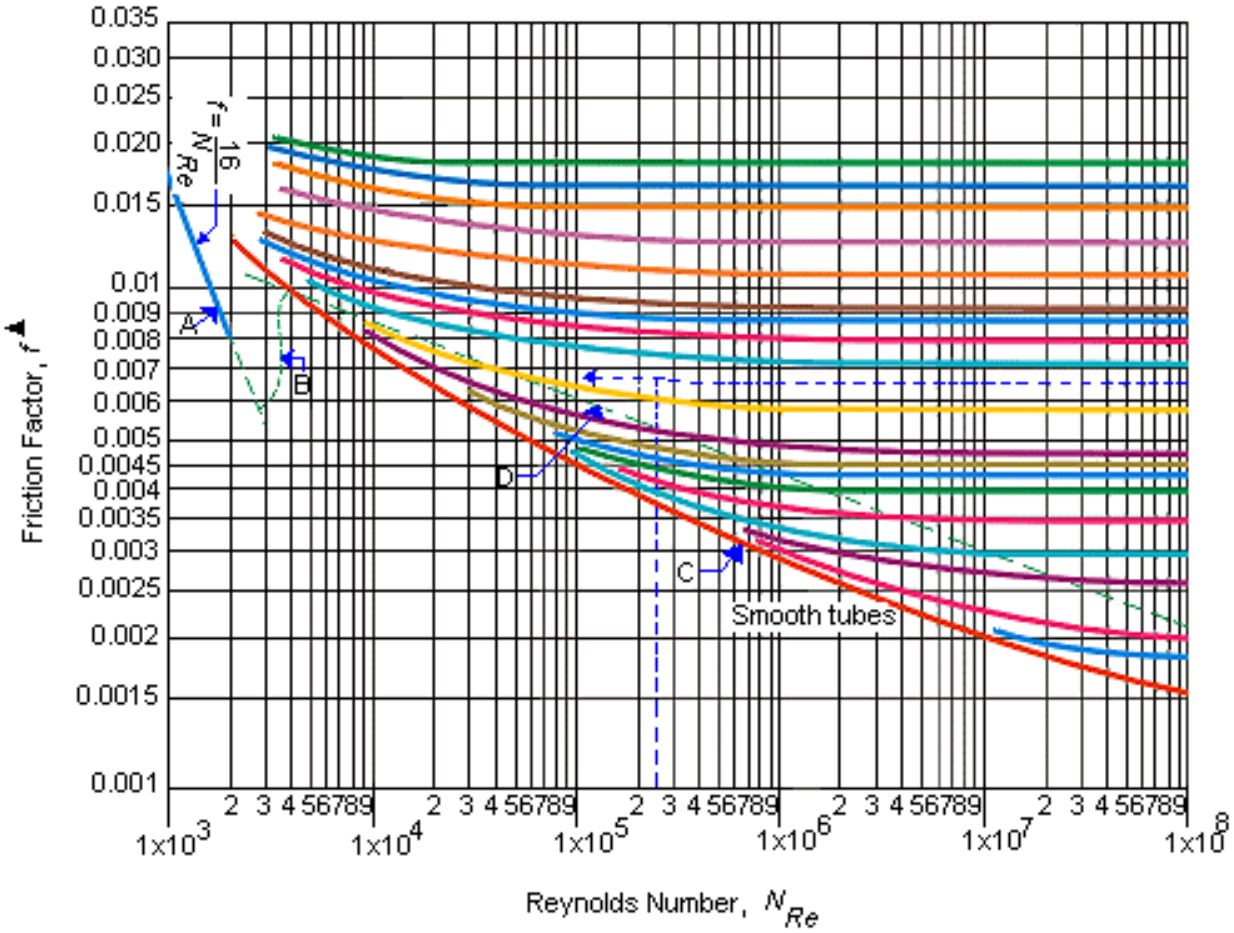
- Dimensionless group: Power number.

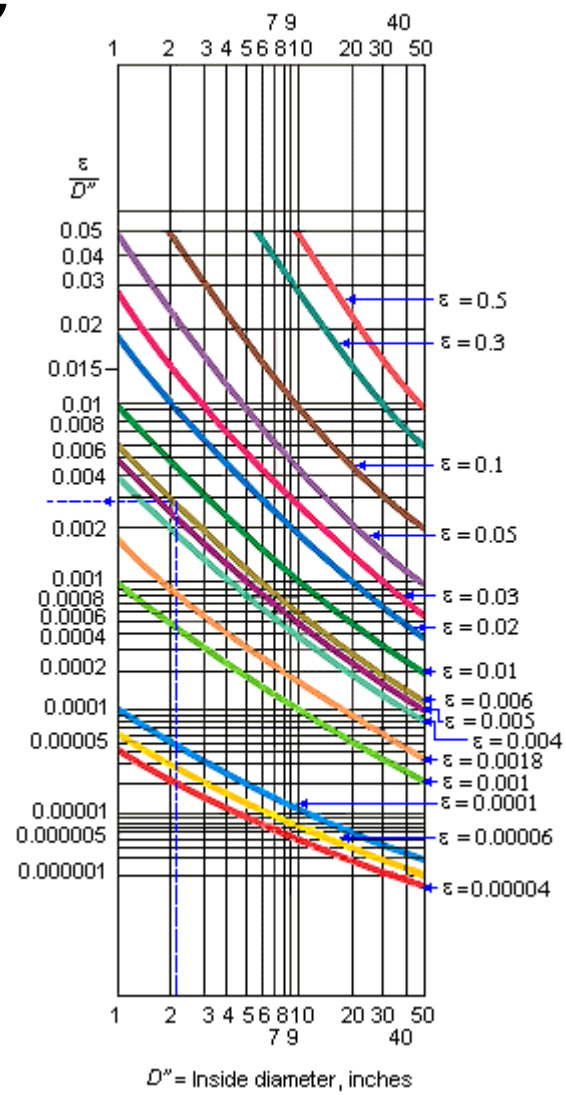
$$Po = \frac{P}{\rho N^3 D^5}$$

- Power number is a drag factor:
  - Analogous to friction factor in pipe flow.

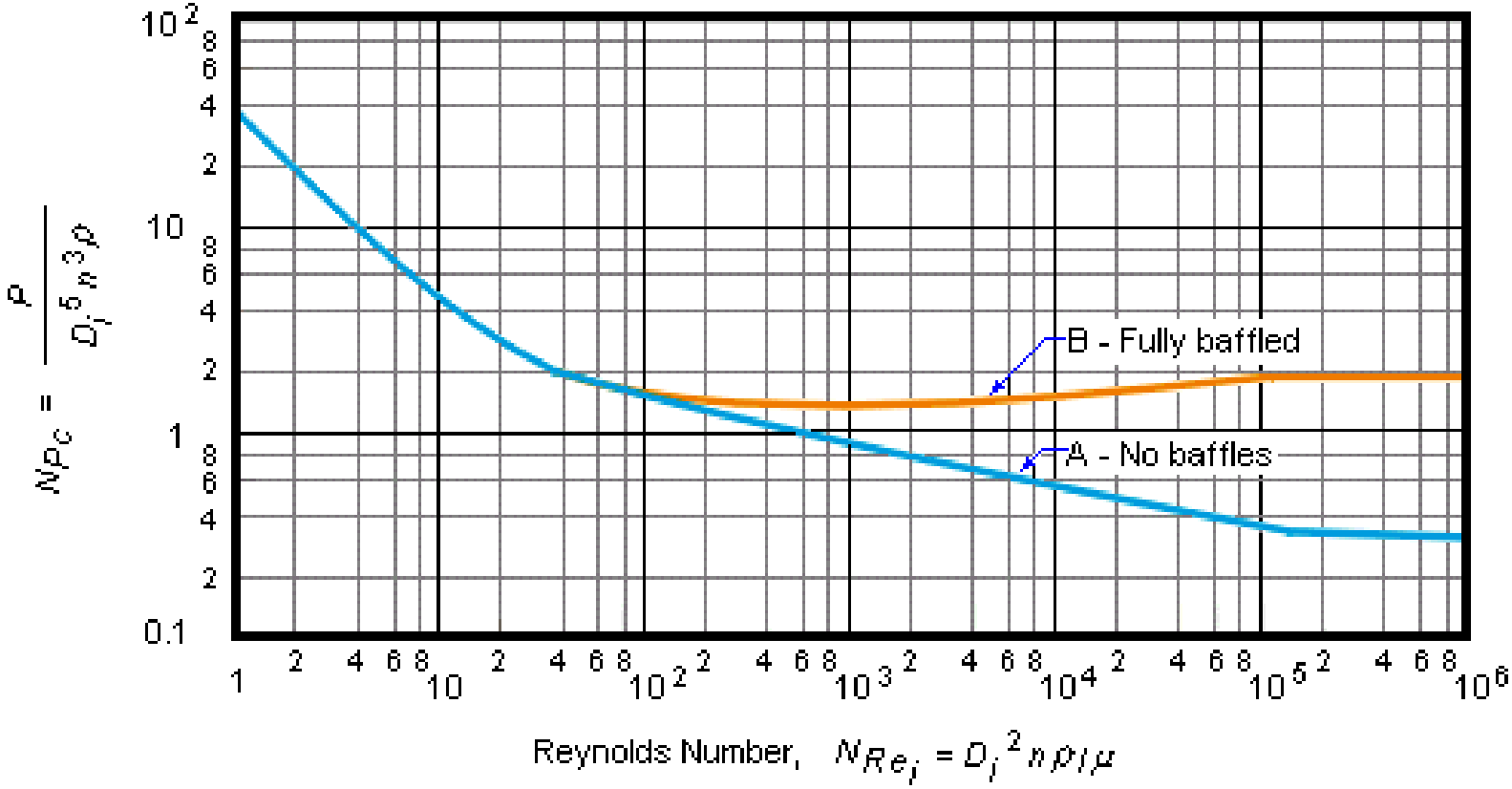
- Power number is a function of impeller geometry.

f versus Re for Pipe Flow





Po versus Re for Standard Flat Paddles



Power Numbers under Turbulent Conditions
Shuie & Wong, Can. J. Chem. Engg., 62 (1984)

Impeller Type	Blade Angle	Number of Blades	D_w/D	P_o
FBT	90	6	0.2	5.0
CBT	90	6	0.269	4.0
CBT	90	4	0.154	4.6
PBT	45	4	0.231	1.74
PBT	45	2	0.231	1.2
Propeller	--	3	--	0.67
FBT	90	4	0.20	3.0
PBT	45	4	0.20	1.0

FBT-Flat Blade Turbine, CBT-Curved Blade Turbine, PBT-Pitched Blade Turbine

Power Numbers under Laminar & Turbulent Conditions
Prof. J.M. Smith, Univ. Surrey

$$Po = \frac{A}{Re} + B + \frac{C}{1000 + Re}$$

Impeller Type	Blade Angle	Number of Blades	A	B	C
FBT	90	6	67	3.2	1.8
CBT	90	6	67	2.6	2.2
PBT	45	4	49	1.5	0.3
PBT	60	4	50	4.0	1.0
Propeller	--	3	47	0.9	0.3
FBT	90	4	50	4.0	1.0

FBT-Flat Blade Turbine, CBT-Curved Blade Turbine, PBT-Pitched Blade Turbine

Turbulent and Laminar Relationships

- Turbulent regime:

$$P = Po\rho N^3 D^5$$

- Power number is constant (in a baffled vessel).
  - Value is dependent on impeller geometry.

- Laminar regime: $$Po = \frac{K_P}{Re}$$

$$P = \frac{K_P}{Re}\rho N^3 D^5 = K_P \mu N^2 D^3$$

- Power is inversely proportional to *Re*.
  - Value of K_P is dependent on impeller geometry.

Torque

- Forces:
  - Impeller exerts forces on fluid.
  - Fluid exerts equal and opposite force on impeller blades.

- Torque is the “twist” force acting on the agitator shaft.

$$Tq = \frac{P}{2\pi N} = \frac{Po\rho N^3 D^5}{2\pi N}$$

$$Tq = \frac{Po\rho N^2 D^5}{2\pi}$$

- Measure of the size of equipment:
  - Shaft diameter.
  - Blade thickness.
  - Gear box size.

Mixing Tank Equipment

- Tanks/Vessels

- Right Cylinder most common
  - Height-to-diameter ratio - 0.8 to 1.5
  - Dished bottom and flat bottom
  - Taller vessels are used in some applications.

- Top Entering Shafts
  - Rotational, vertical

- See http://www.mixing.net/ for links to Mixing Equipment Vendors

Mixing Tank Baffles

- Mixing Tank without baffles
  - Predominantly rotational flow (solid-body rotation)
  - No interchange between top and bottom

- Vertical Baffles
  - Turn rotational component into vertical component
  - Increase top to bottom flow
  - Always specify baffles for turbulent flow

- Alternatives to Baffles (for small vessels)
  - Angle mount shaft
  - Off-center vertical mount shaft
  - Side-entering shaft

Standard Baffle Configuration

- Full Baffling
  - 4 Vertical Baffles at full length of straight side of tank
  - Baffle Width, $B_w = T / 12$
  - Offset from Wall = $T / 72$ (or $B_w / 6$)

- Partial Baffling Alternatives
  - 1/2 Height, 1/2 Width, 2 Baffles
  - Results in some surface vortex

- *Po* (Baffled) is greater than *Po* (Unbaffled) by 20-50%

MULTIPLE IMPELLERS AND SHAFTS

- About 80% of all agitators have more than one impeller
  - Uniformity of process result over whole tank
  - All impellers same speed
  - Tall tanks
- Assume additive
  - Calculate individual powers
  - Good for power when impellers are about one impeller diameter apart
  - Other wise some what less
- Mix time - assume reciprocals mix times are additive
  - Take away height correction

- Multiple shafts – nature of flow changes with time
  - Batch
  - Different speeds – anchor can act as baffle
  - Only recently studied – pretty independent

Power, Flow and Head

- Impeller generates flow and head.

- Tip Speed = πND
  - 2 - 6 m / s
  - Defines maximum velocity.

$$P = Q\Delta H$$

$$\Delta H = \frac{P}{Q} = \frac{Po\rho N^3 D^5}{FlND^3}$$

$$\Delta H = \frac{Po}{Fl}\rho N^2 D^2$$

- For axial flow impellers:

$$\Delta H = 1.30\rho(Po^{1/3}ND)^2$$

Example Calculations

- Pitched blade turbine:
  - Diameter: 1 m
  - Operating speed: 84 RPM
  - Power number: 1.74
  - Fluid: Water

- Power:

$$P = Po\rho N^3 D^5$$

$$P = 1.74 \times 1000 \times (84/60)^3 \times 1.0^5$$

$$\boldsymbol{P = 4775\ \ \mathbf{W}\ \ (\mathbf{or\ 6.40\ HP})}$$

- Flow:

$$Q = FlND^3 = 0.77 Po^{1/3} ND^3$$

$$Q = 0.77 \times 1.74 \times (84/60) \times 1.0^3$$

$$\boldsymbol{Q = 1.297\ \ \mathbf{m^3/s}\ \ (\mathbf{or\ 20553\ GPM})}$$

Example Calculations

$$Tq = \frac{Po\rho N^2 D^5}{2\pi}$$

$$Tq = \frac{1.74 \times 1000 \times (84/60)^2 \times 1.0^5}{2\pi}$$

- Torque:

$$\boldsymbol{Tq = 542.8\ \ \mathrm{Nm}}$$

$$\Delta H = 1.30\rho(Po^{1/3} ND)^2$$

- Head:

$$\Delta H = 1.30 \times 1000 \times (1.74^{1/3} \times (84/60) \times 1.0)^2$$

$$\boldsymbol{\Delta H = 3686\ \ \mathrm{Pa}\ \ (\mathrm{or}\ 0.376\ \mathrm{m})}$$

Characteristic Velocities

- Tip Speed = πND
- Thrust Velocity: Flow / Pumping Area:
- Axial Impeller:

$$U_{Th} = \frac{Q}{A_I} = \frac{FlND^3}{(\pi/4)D^2}$$

- Radial Impeller:

$$U_{Th} = \frac{Q}{A_I} = \frac{FlND^3}{\pi Dw}$$

M5
MIXING CONCEPTS II

Blending Liquids

BLENDING LIQUIDS

- Low viscosity – water like
- In turbulent flow
- Usually not controlling
- Most important when fast chemical reactions are involved
  - Will not be discussed

ChemScale Method

- Need method to rate motion in tanks.
- Bulk Fluid Velocity: Flow / Vessel Area.
- Use Bulk Fluid Velocity as criterion for describing motion:
  - Combines Tank Size and Impeller Effect.
- Little turbulence is required with homogeneous liquids:
  - Bulk Flow or Pumping is preferred.
- Standardize:
  - 45° Axial-4 Impeller (45° Pitched Blade Turbine).
  - Off-bottom Clearance, *C*.

ChemScale Method

- Rate Bulk Velocity on 1-10 Scale
  - Low: ChSc = 1: 6 ft / min
  - Medium: ChSc = 5: 30 ft / min
  - High: ChSc =10: 60 ft / min

- Correct for *Re* effect:
  - Increase viscosity reduces pumping ability of impeller.
  - Crude method but often good enough.

$$BFV = \frac{Q}{A_T} = \frac{FlND^3}{(\pi/4)T^2}$$

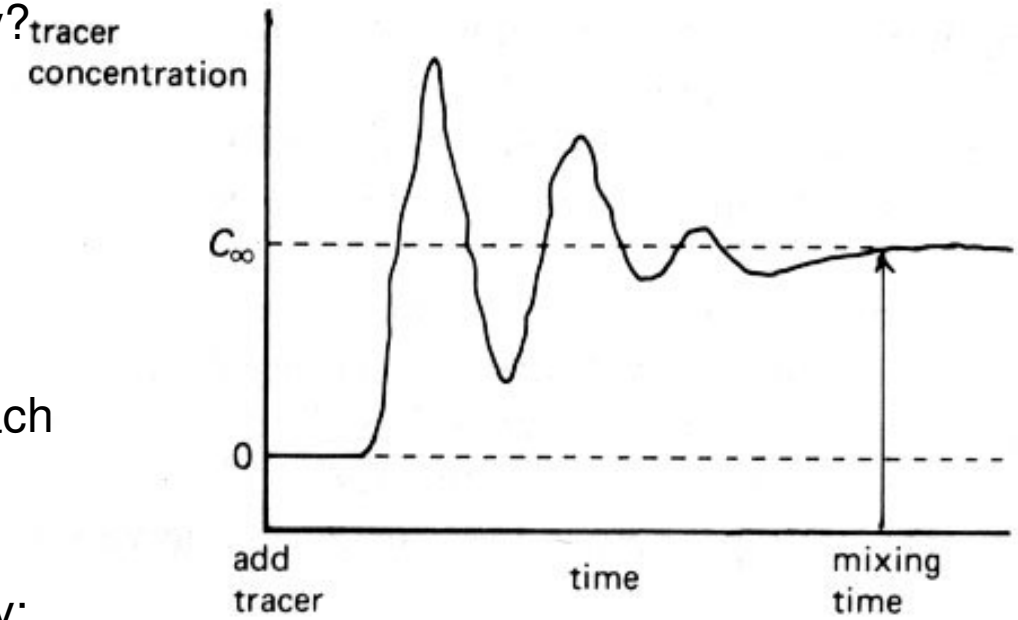
$$BFV \propto FlND\left(\frac{D}{T}\right)^2$$

BLEND TIME

- How long does it take to become uniform after a change?
- Sampling
- Batch blending
- Etc.

Blend Time

- How fast to get to Homogeneity?
- Measurements - Batch Stirred Tank:
  - Color Change - somewhat arbitrary
  - Conductivity or pH - approach to steady state
- Approach to Average Uniformity:
  - 95% approach (or 5 % of steady state).
  - Extrapolation along exponential decay curve.



$$\Delta c' = \exp(-k\theta)$$

Blending Mechanism

- Concentration fluctuations decay exponentially.
- 95% homogeneity when $\Delta c' = 0.05$:

$$\theta_{95} = -\frac{\ln(\Delta c')}{k}$$

$$\Delta c' = 0.05$$

$$\theta_{95} = -\frac{\ln(0.05)}{k} = \frac{3}{k}$$

- If time to certain degree of homogeneity is known, time to different degree can be calculated:
  - *k* can be calculated.
  - Used to calculate blend time to different degree of homogeneity.

Blend Time in Turbulent Regime

- Correlation for estimation of blend time in agitated vessels:
  - $0.33 < D / T < 0.50$.
  - $C / T = 0.33$.
  - $0.50 < H / T \leq 1.00$.

$$\theta_{95} = \frac{5.40}{Po^{1/3} N} \left(\frac{T}{D}\right)^{1.5} \left(\frac{H}{D}\right)^{0.5}$$

- In turbulent regime (in baffled vessel), *Po* is constant.

- For a given impeller / vessel geometry:

$$N\theta_{95} = C$$

Impeller Efficiency

- Power:

$$\theta_{95} = \frac{5.40}{Po^{1/3} N} \left(\frac{T}{D} \right)^2$$

$$\theta_{95} \propto \bar{\varepsilon}^{-1/3} \left(\frac{T}{D} \right)^{1/3} T^{2/3}$$

- Conclusions (at the same power input per unit mass):
  - Different impellers with same D / T will give same blend time.
  - Scaling-up increases blend time by scale factor raised to 2/3 power.
  - Compare with theoretical turbulence model.

- Torque:
  - Impeller with low power number must run at higher speed.
  - Higher speed $\rightarrow$ lower torque.
  - Hydrofoil impellers need same power but lower torque to achieve blend time.

FOURIER NUMBER

- A relation between mix time and physical properties
- Moderate use
- $Fo = \mu * \theta / \rho / T^2$

OTHER EFFECTS

- Physical property differences between what is added and bulk
- Density effects
  - Not significant

- Viscosity effects
  - Can be significant if add at quite region
  - Can increase time – acts like dissolving

Transitional Regime - Experimental

- Dimensionless Blend Time:
  - Constant of proportionality dependent on *Po*, *D* / *T*.

$$N\theta \propto Re^{-1}$$

- Power Number:

$$Po = K$$

- Experiments show three dimensionless groups in transitional regime:

$$Po^{1/3} Re = \frac{186}{\sqrt{Fo}}$$

$$N\theta_{95} = \frac{186^2}{Po^{2/3} Re} \left(\frac{T}{D} \right)^2$$

$$N\theta_{95} \propto Re^{-1}$$

Expand Dimensionless Groups

$$\theta_{95} = \frac{186^2 \mu}{Po^{2/3} \rho N^2 D^2} \left(\frac{T}{D} \right)^2$$

- Blend time is proportional to fluid viscosity.
- Re-arrange to express in terms of power input, etc.

$$\theta \propto \left(\frac{1}{\bar{\varepsilon}} \right)^{2/3} \left(\frac{T}{D} \right)^{2/3} \frac{\mu}{\rho} T^{-2/3}$$

- Blend time reduces on scale-up?
- Implications for researchers working in lab?

Boundary between Regimes

- Solve two correlations:

$$Po^{1/3}Re = \frac{5.40}{Fo} \qquad Po^{1/3}Re = \frac{186}{\sqrt{Fo}}$$

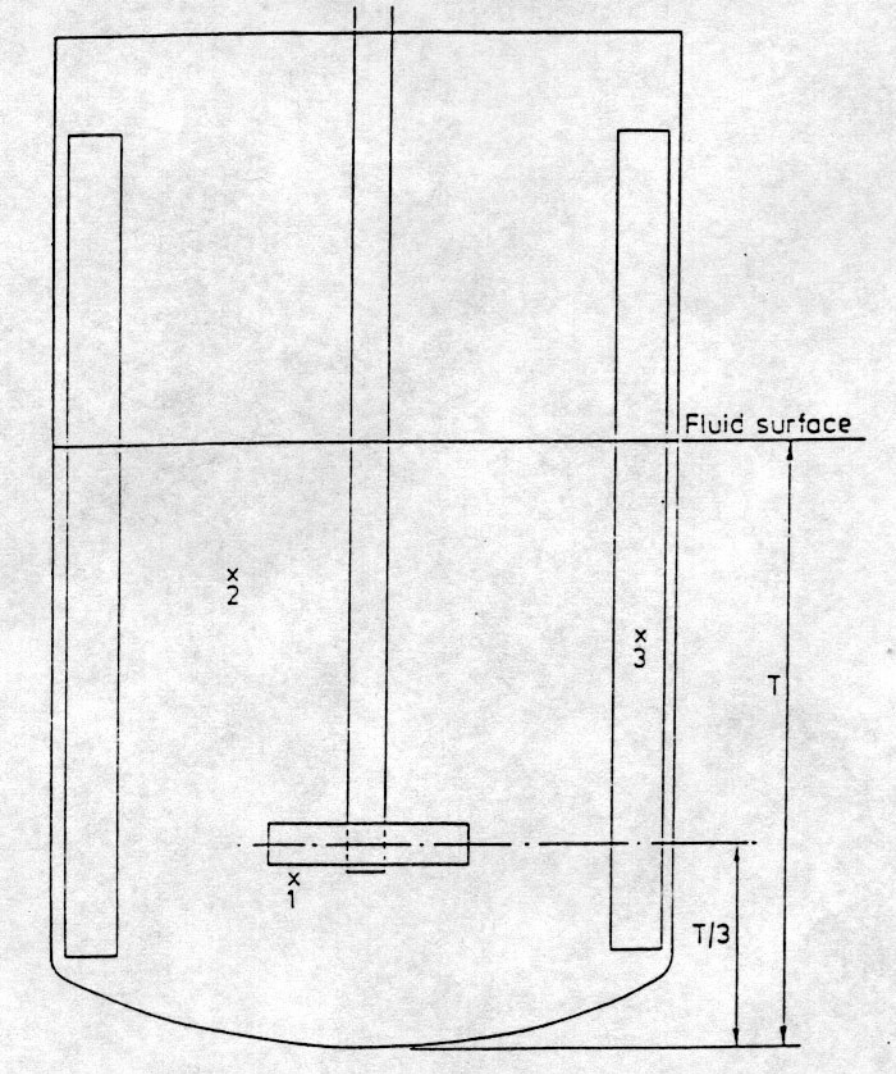
- At boundary of two regimes:

$$\frac{1}{Fo} = 1186 \qquad Po^{1/3}Re = 6404$$

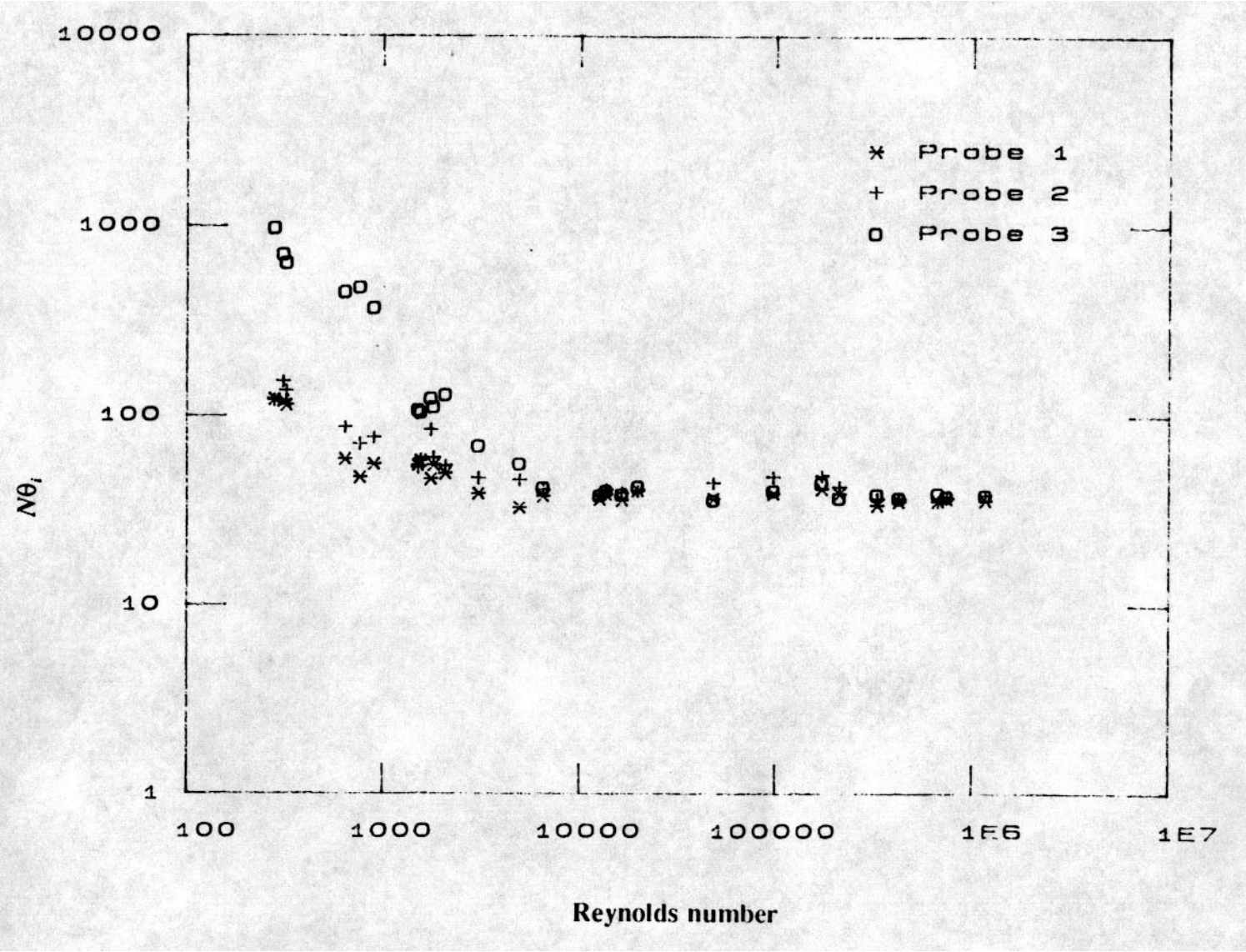
- Values are independent of impeller type.

Blend Time versus Position

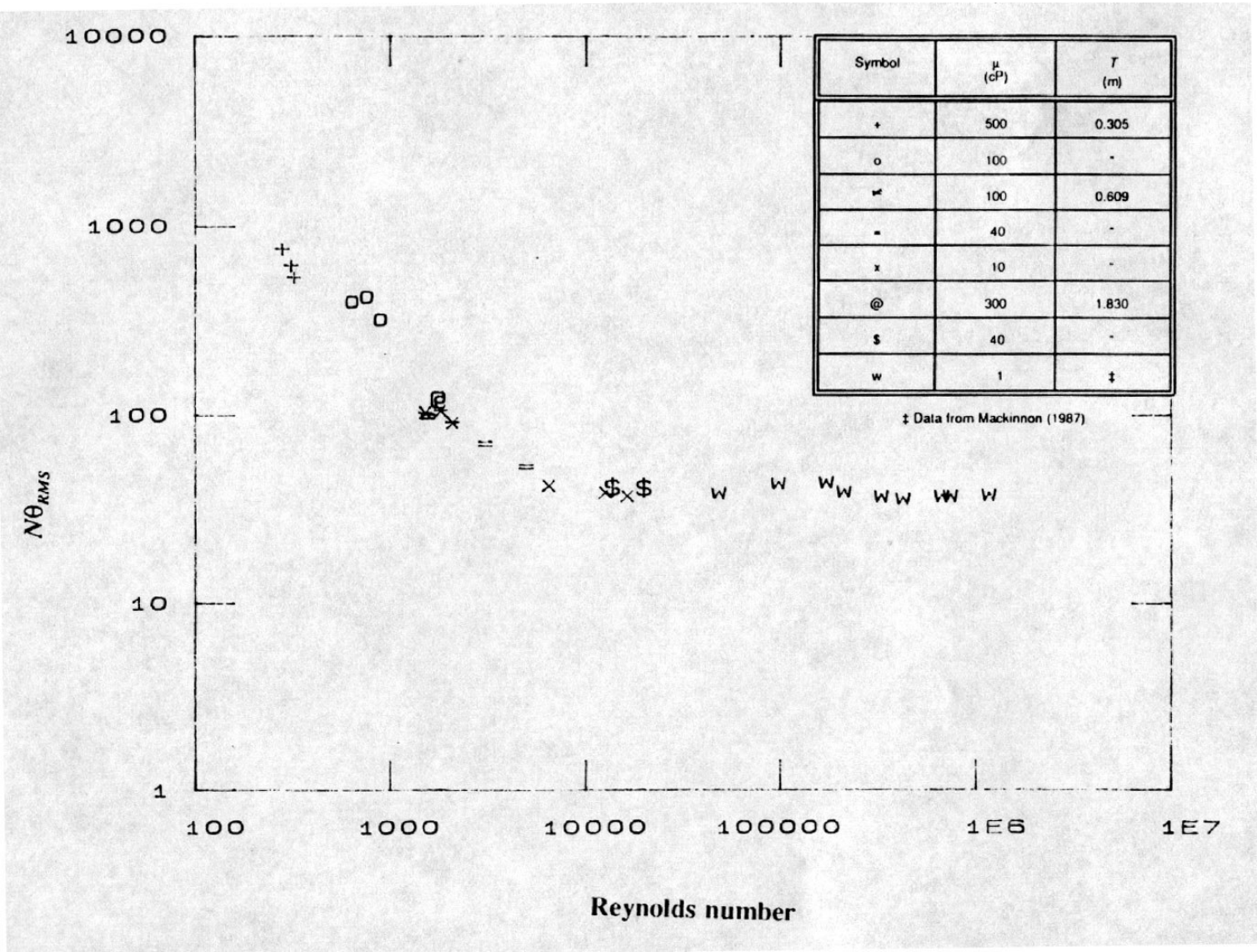
- Experimental set-up:
- Three probes in regions of different mixing intensity:
  - 1 - Under Impeller
  - 2 - Between Shaft and Wall
  - 3 - Behind Baffle



Results for Pitched Blade



Results for Pitched Blade



Symbol	μ (cP)	T (m)
+	500	0.305
o	100	-
⊠	100	0.609
=	40	-
x	10	-
@	300	1.830
$	40	-
w	1	‡

Conclusions

- In Turbulent Regime:

$$N\theta_1 = N\theta_2 = N\theta_3$$

- In Transitional Regime:

$$N\theta_1 < N\theta_2 < N\theta_3$$

  – Degree of deviation increases as Reynolds number decreases.
  – Blend Time for whole vessel is weighted by longest individual time.

- Impeller region is turbulent (Blend time and Power No.).

- Viscosity damps intensity of turbulence away from impeller.

Which Regime?

- Need to know if impeller operates in turbulent or transitional regime:
  - Which correlation to use for blend time?
- Designing new vessel and agitator.
- Rating existing vessel and agitator for new duty.
- How far can one go with turbine impellers?

Laminar Regime

- Boundary between transitional and laminar regimes:

$$Re < 200: \qquad N\theta \propto Re^{-10}$$

$$Re < 50: \qquad Po \propto Re^{-1}$$

- Blending performance becomes laminar before Power No. starts to rise.
  - Blend time for vessel governed by slowest mixing region.
  - Slow near wall, behind baffles, at surface.
  - Turbulence still being generated near impeller.

- May be possible to use turbine at lower Reynolds numbers:
  - Depends on process requirements (e.g. cycle time).

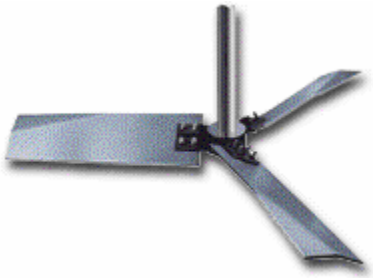
Impeller Selection

$D / T \approx 0.95$

Increasing μ

Increasing *Re*

$0.3 < D / T < 0.5$



Impeller Selection for Laminar Blending

- Blending vessel contents relies on convection of fluid from impeller zone to wall and back again.
- As viscosity rises, pumping capability of turbines reduces.
- Need “positive-displacement” impeller.
- Close-clearance - $0.90 < D / T < 0.98$.
- Anchors and Helical Ribbons.
- Use Helical Ribbons.

Helical Ribbon Power Consumption

- Equation for predicting power consumption:

$$P = Po\rho N^3 D^5$$

- In the laminar regime:

$$Po = \frac{K_P}{Re} = \frac{K_P \mu}{\rho N D^2}$$

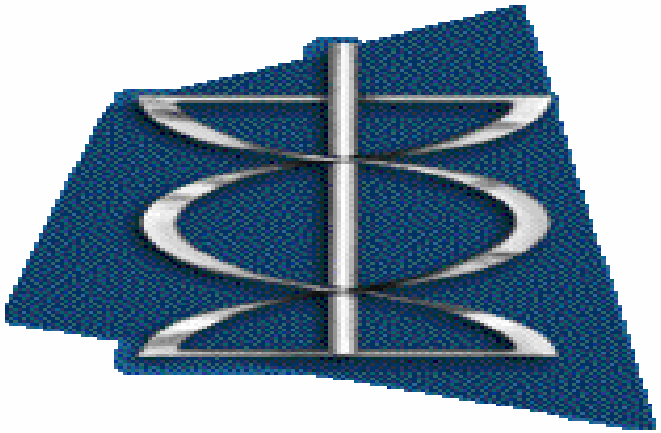
- Combining:

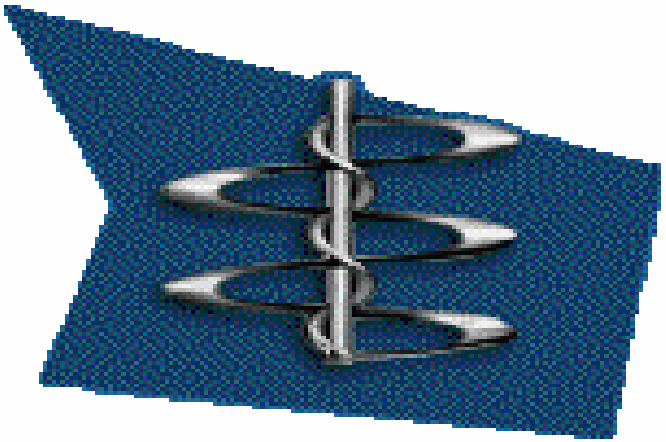
$$P = K_P \mu N^2 D^3$$

- For turbine impellers: $50 < K_P < 70$.
- For Anchors: $K_P = 225$.
- For Helical Ribbons: $K_P = 350$.

Helical Ribbons

- Single or Dual Flight.
- May have Central Auger.
- Ribbon width - 8 - 10 % of diameter.
- Pitch = Height of one turn.
- Clearance = Gap between impeller and vessel wall.

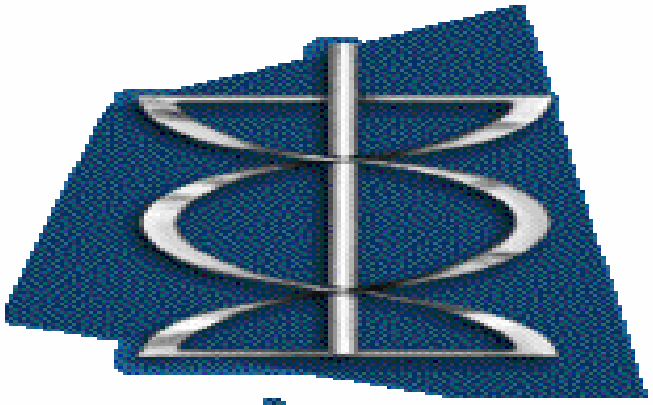




Anchors versus Ribbons

- Helical Ribbon:
  - Produce 3-D flow pattern.
  - Good mixing.
  - Higher power.

- Anchors:
  - Produce strong tangential flow.
  - Little axial flow.
  - Poor overall mixing.



Blend Time

- For Rieger's eight helical ribbons:

$$N\theta = 896 \times 10^3 K_P^{-1.69}$$

- No fundamental physical basis.

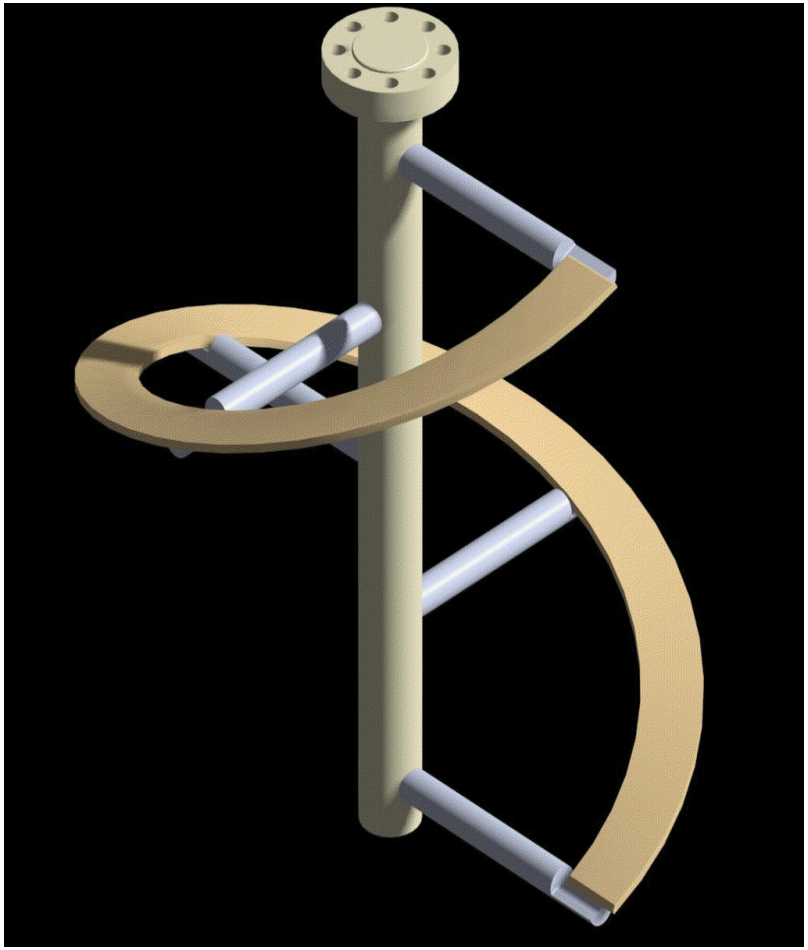
- Design new agitator:
  - Blend time is known.
  - Vessel size is known → impeller diameter → impeller geometry.
  - Calculate K_P.
  - Calculate $N\theta$.
  - Calculate N → check standard speed → iterate if necessary.
  - Calculate power draw → next standard motor power.

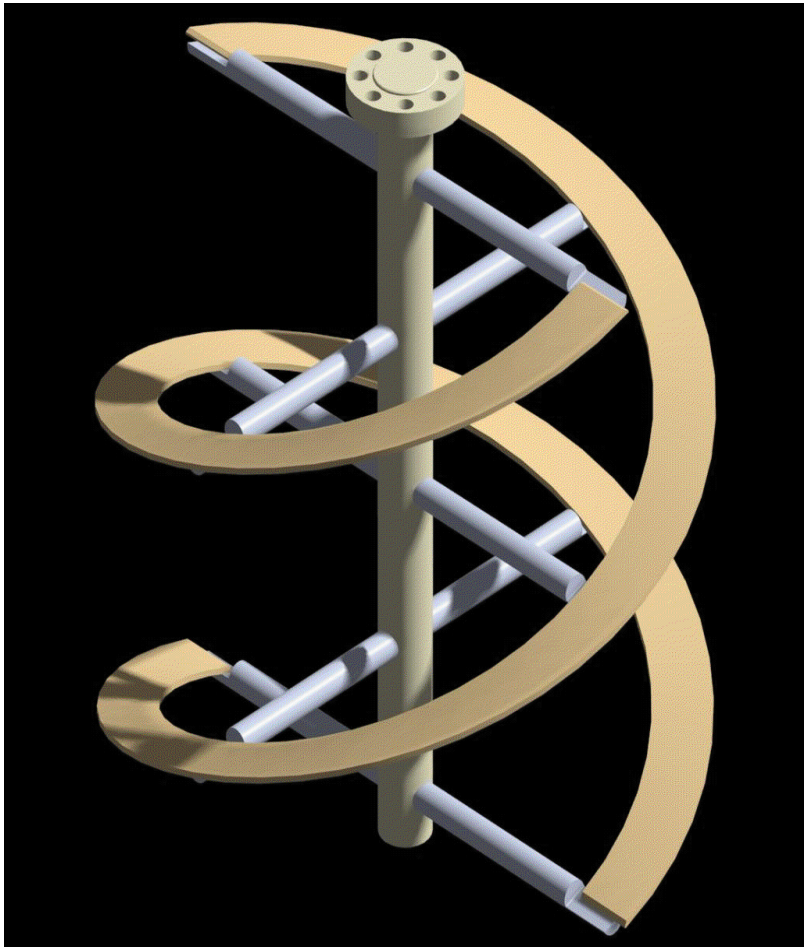
- Rate existing agitator?

Ribbon Nomenclature

- h is impeller height.
- D is impeller diameter.
- n_b is number of ribbons.
- p is impeller pitch.
- c is clearance from wall.

Which Impeller?





Ribbon Selection

- Dual ribbon:
  - Balanced forces on shaft.
  - Higher $K_P \rightarrow$ lower $N\theta$.

- Does lower $N\theta$ result in lower power?

- Shaft diameter may be determined by construction:
  - Must be large enough to have arms welded on safely.

Alternates to Helical Ribbons

- Many exist
- Cheaper to make not better
- May be sensitive to non Newtonian effects

Problem

- Correlations for blend time and power consumption exist for Newtonian fluids.
  - Transitional regime: Blend time proportional to viscosity.
  - Laminar regime: Power proportional to viscosity.

- Many viscous fluids used in industry are non-Newtonian.

- How should rules relating to Newtonian fluids be modified for non-Newtonian ones?

NON NEWTONIAN BLENDING

- To be discussed later

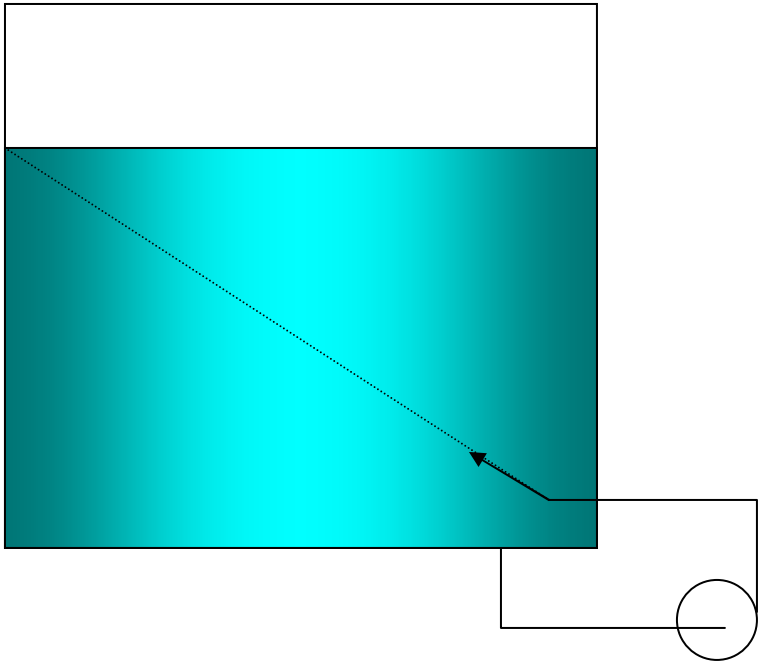
BLENDING WITH JETS

Turbulent jets

MIXING WITH JETS LIQUID

- Jets for tank Mixing
- Maximize path length
- Involve whole tank
- Single jet near bottom
  - no swirl
- Aimed at upper surface a t 2/3 to full diameter
- Remove liquid from near injection point

JET MIXING OF TANKS



MIXING WITH JETS LIQUID

- Mix time correlation

- Mix time to 95 % = 7.0*T^1.5*H^.5 /Vj / Dj

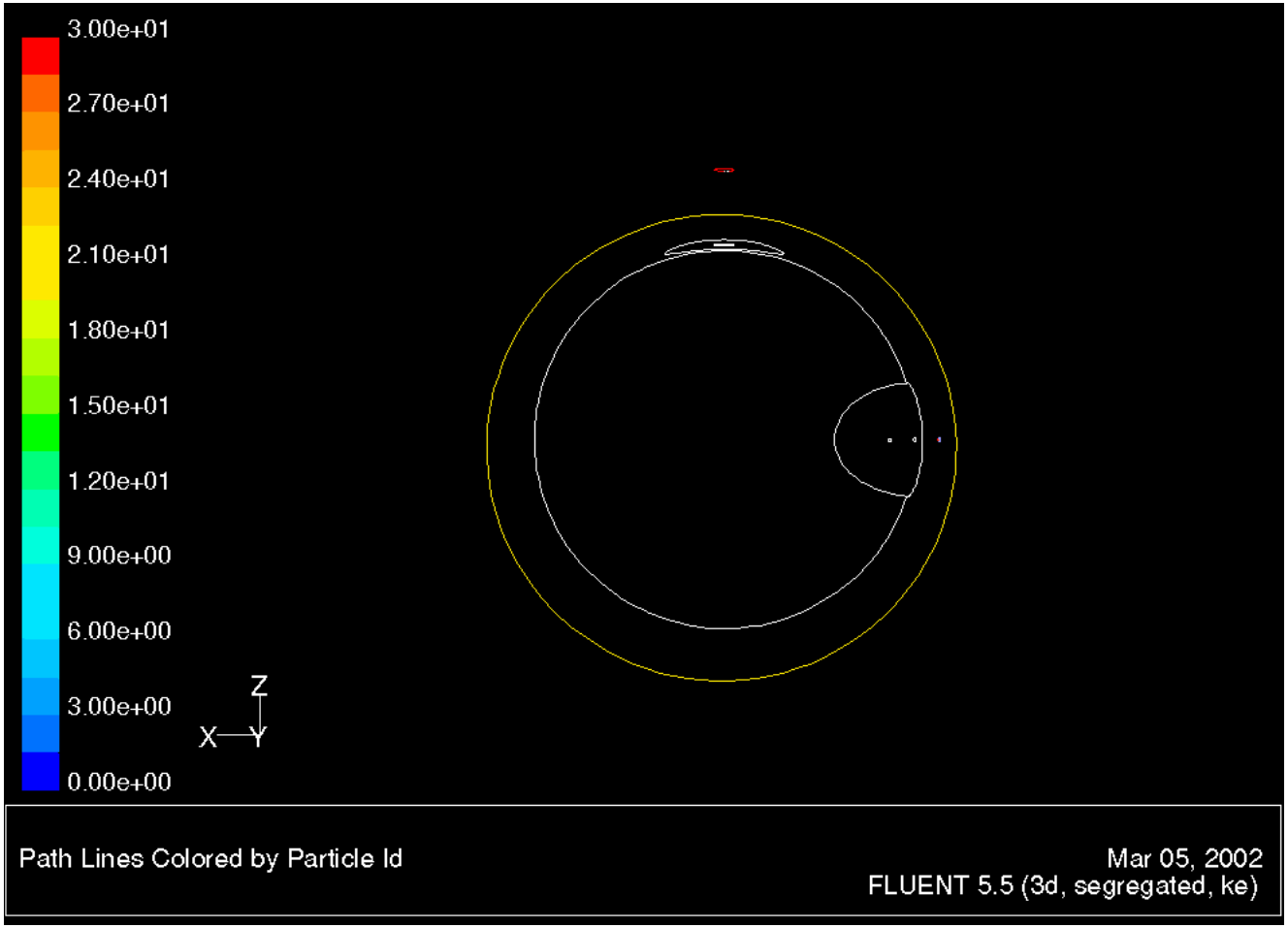
MIX TIME CORRELATION

- Grenville and Tilton
- Use path length
  - Mix time = 3*Z^2/(Vj*Dj)
- For Vj*Dj/Z> .0132 m/sec
- See Handbook of Industrial Mixing 2003 for more details

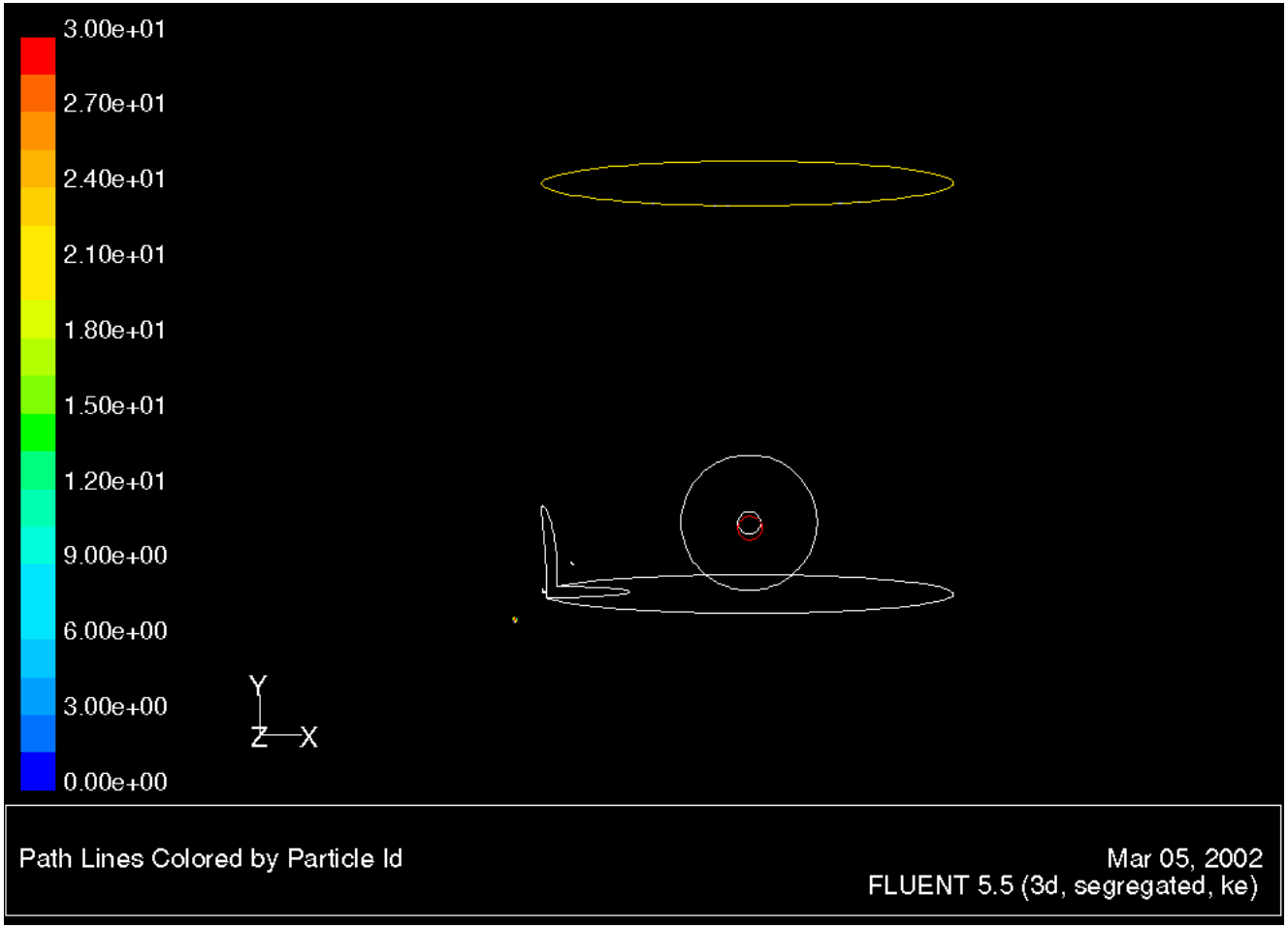
HINTS

- Start with Vj = 10 m/sec – a reasonable velocity
- Expect mix time of the order of minutes to hours roughly proportional to tank diameter rather than the seconds found with mechanical mixer
- Extension into transitional, laminar and non Newtonian flow not easily achieved. Stay above REj=10,000

JET MIXING
CFD – Chris Wolf - Fluent



JET MIXING


3.00e+01
2.70e+01
2.40e+01
2.10e+01
1.80e+01
1.50e+01
1.20e+01
9.00e+00
6.00e+00
3.00e+00
0.00e+00
Y
Z
X
Path Lines Colored by Particle Id
Mar 05, 2002
FLUENT 5.5 (3d, segregated, ke)

HOW FAR CAN THIS BE PUSHED

- Limit of jet action
- See Tilton and Grenville in HIM
- Suggest that jet effectiveness ends when turbulence drops below a certain value.

NOMENCLATURE

- H – liquid height
- Z, z – jet path length
- T – tank diameter or temperature
- Dj – jet diameter
- Q – flow – water, l – liquid, g- gas
- Qj – flow out of jet
- Vj – velocity at jet
- Pm – power per unit mass
- RE – Reynolds Number at jet

END SECTION M5

M6
LIQUID BLENDING
YIELD STRESS MATERIALS

Yield Stress Fluids

- Important class of fluids for industry.

- Fluid must experience certain shear stress before it will move:

$$\tau = \tau_Y + \mu_\infty \dot{\gamma} \qquad \mu_A = \frac{\tau_Y + \mu_\infty \dot{\gamma}}{\dot{\gamma}}$$

$$\tau = \tau_Y + K\dot{\gamma}^n \qquad \mu_A = \frac{\tau_Y + K\dot{\gamma}^n}{\dot{\gamma}}$$

- Typical yield stress is 10 - 50 Pa:
  - At low shear rates, apparent viscosity is very high.

Shear Stress - Shear Rate Models

- These models are simply “fits” of data to a model.
- No physical basis.
- A yield stress fluid may be considered a shear-thinning fluid with very low *n*.
- A shear-thinning fluid may be considered a yield stress fluid with a low yield stress.
- Need to design mixers that work and yield stress model allows us to do this.

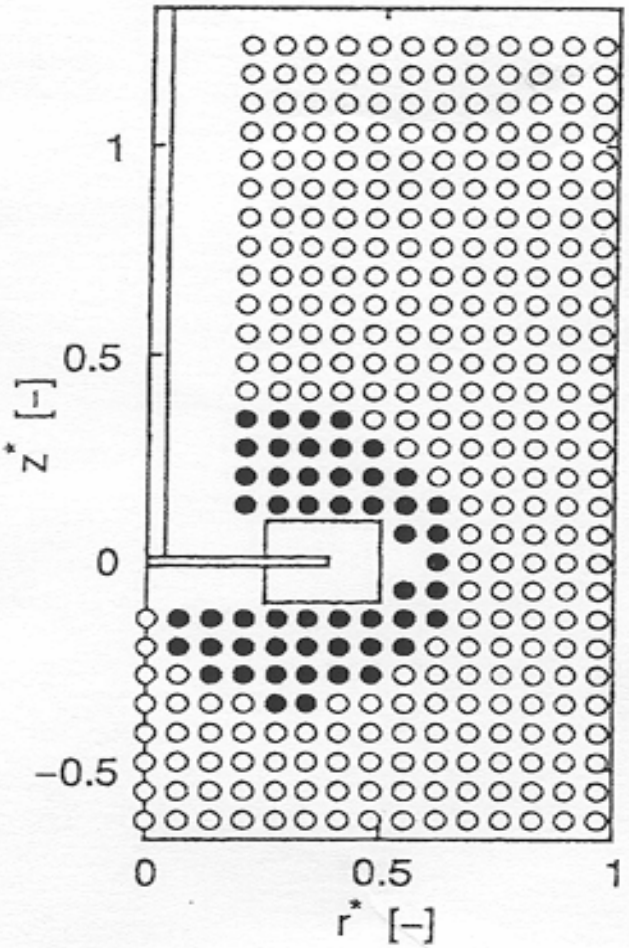
Observations

- A “cavern” of moving fluid is created around the impeller.
- A the boundary, the shear stress generated by the impeller is equal to the fluid’s yield stress.
- As impeller speed increases, cavern size increases.
- Until cavern reaches wall:

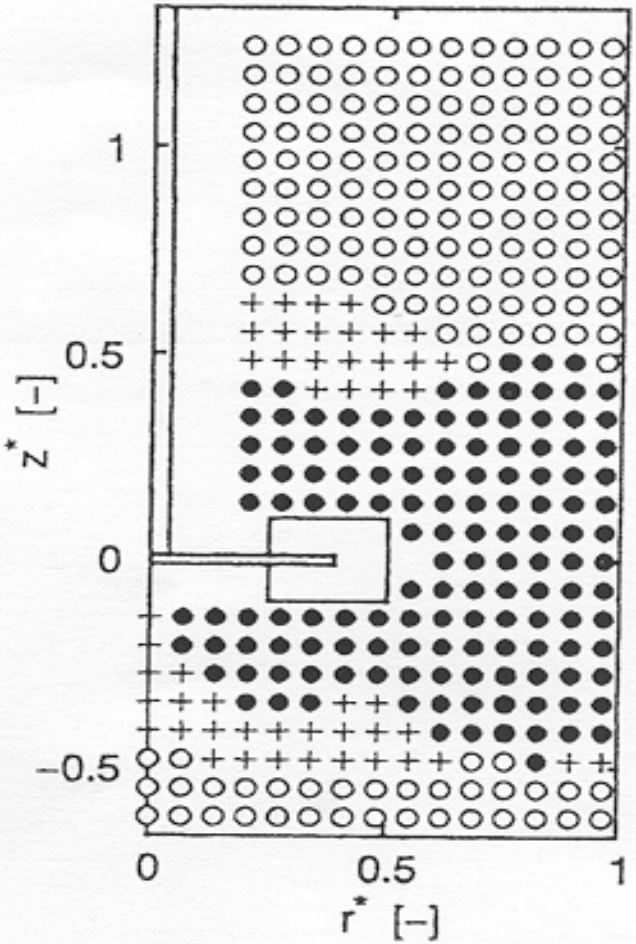
$$H_C = xD_C$$

- Once cavern reaches wall, cavern grows axially.

Hirata & Aoshima, ChERD,



(a) Re=28 (1.1rps)



(b) Re=183 (3.4rps)

Streak Photography - Nienow

Impeller Blade

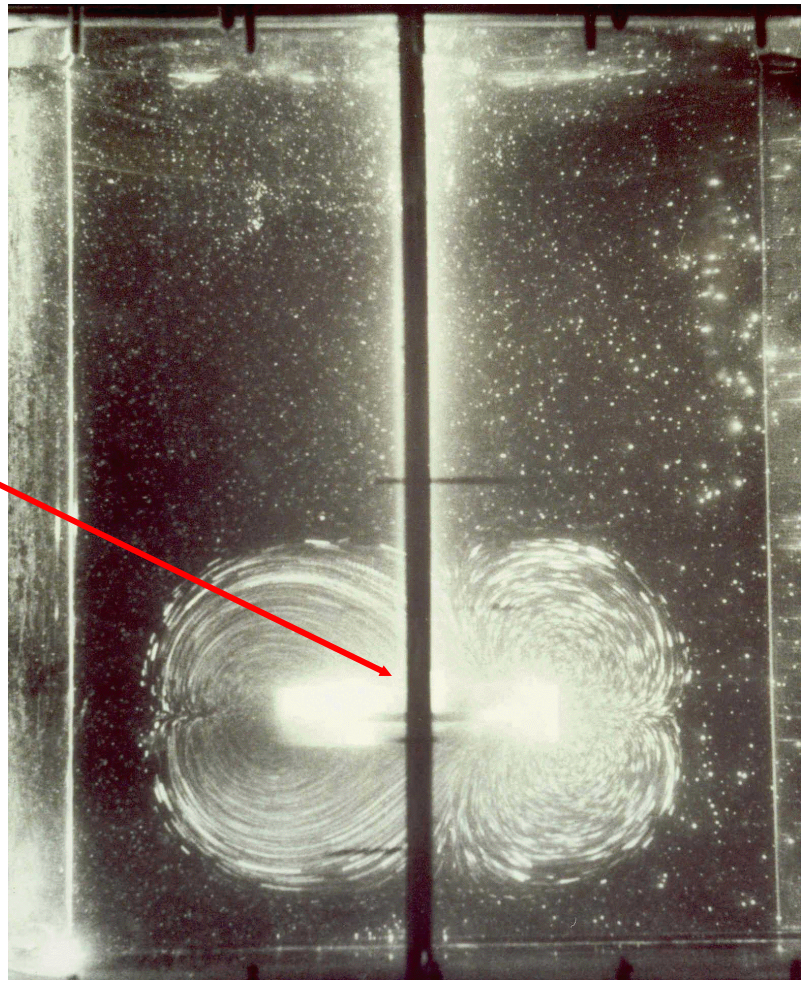
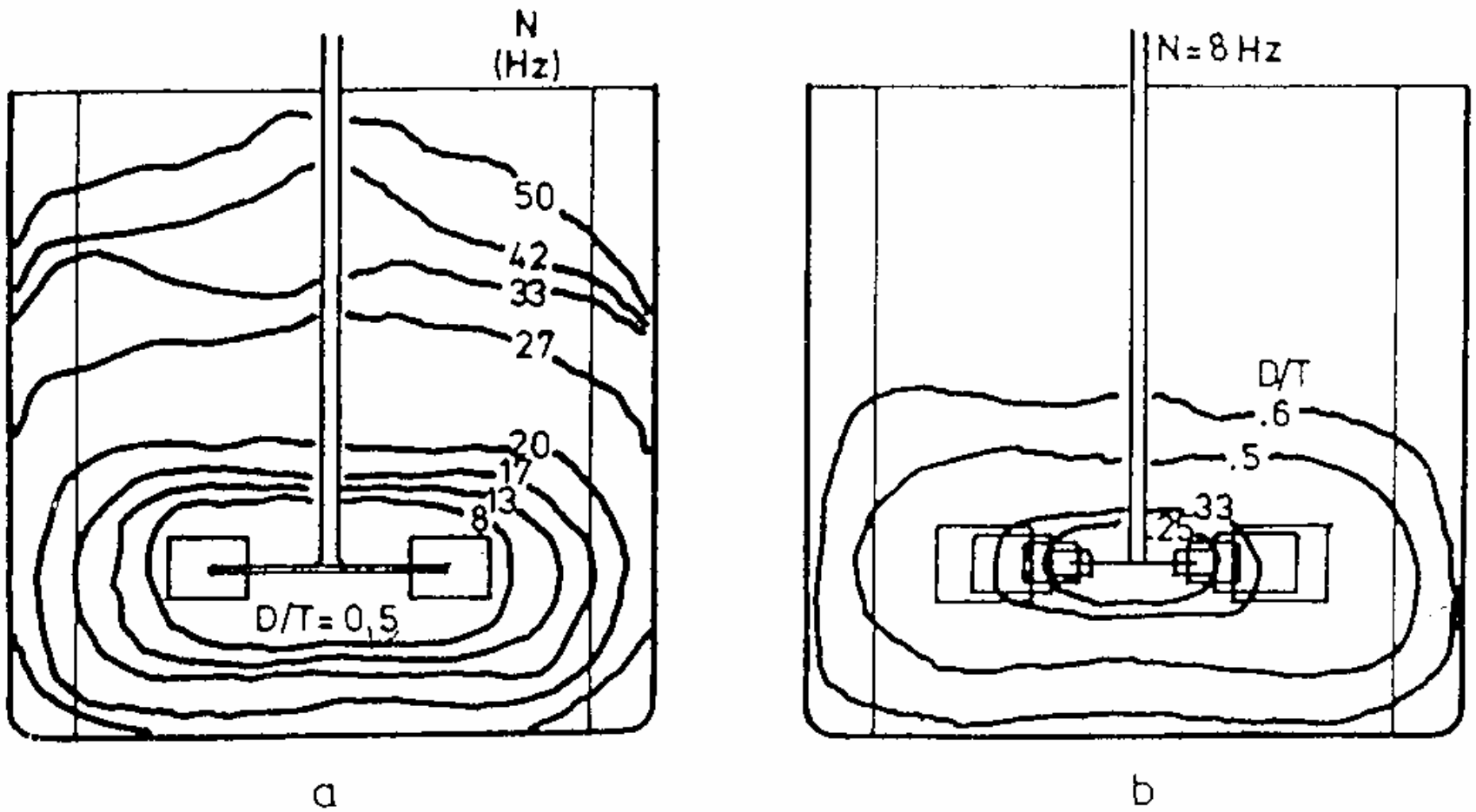


Figure 7. The effect of (a) impeller speed
(b) impeller diameter on cavern size.



Cavern Diameter

- Prediction of Cavern Diameter:

$$\left(\frac{D_C}{D}\right)^3 = \frac{1.32}{\pi^2} PoRe_Y$$

$$Re_Y = \frac{\rho N^2 D^2}{\tau_Y}$$

- Minimum speed when cavern reaches vessel wall:

$$D_C = T$$

$$N_C = \sqrt{\frac{\pi^2}{1.32}\left(\frac{T}{D}\right)^3 \frac{\tau_Y}{Po\rho D^2}}$$

Growth of Cavern

- Once cavern reaches vessel wall, it grows axially:

$$\frac{H_c}{T} = x\frac{N}{N_c}$$

- Need to compare power and speed of single impeller design versus multiple impellers (intersecting caverns).

- Value of *x* dependent on impeller type:
  - Rushton turbine: $x = 0.40$
  - Paddle: $x = 0.45$
  - Pitched blade turbine: $x = 0.55$
  - Propeller: $x = 0.75$

Shear-thinning vs. Yield stress Correlations

- For yield stress fluids, when cavern reaches wall:

$$\left(\frac{T}{D}\right)^3 \propto \frac{Po\rho N^2 D^2}{\tau_Y}$$

$$\frac{Po\rho N^2 D^5}{T^3} \propto \tau_Y \rightarrow \frac{Tq}{T^3}$$

- For shear-thinning fluids:

$$\tau_W \propto \frac{Tq}{T^3}$$

YIELD STRESS

- Correlations work amazing well
- Often used for design and prediction
- Scale up is simple even without rheology

CAVERNS AND JETS

- Momentum sets reach of jet
- Work by Battelle on PJMs for WTP Bechtel
- Show caverns and stagnant zones

MOBILIZATION

- Radius of cleaning increases with time
- Is this a time dependent rheology effect?
- Is this an erosion effect?
- Kinetic are usually so fast in mixing they are neglected.
- Time dependent rheology (thixotrophy) requires very complicated models and is studied by only a small group

M7
SOLIDS LIQUID MIXING

OFF BOTTOM AND
DISTRIBUTION AND ATTRITION

SOLID-LIQUID MIXING

- **Solid suspension**
- **Solid distribution**
- **Dual impeller configurations**
- **Jet solid suspension**
- **Attrition - breakage**

PROCESS RESULTS

- Off bottom suspension
- Distribution vertically (and horizontally)

- Secondary
- Size reduction – break up of agglomerates
- Draw down of floating solids
  - Wetting
  - Low solids density
  - Low bulk density – dispersion

- NOTE: dispersion often has a double meaning
  - Dispersion over height and break up or dispersion of primary particles

CAUTION

- There are two kinds of slurry.

- <u>Slow Settling:</u> Pseudo-homogeneous.
  Treat as non-Newtonian.
  High concentration.
  Small particles.

- <u>Fast Settling:</u> Rate > 1 m / min.
  Two phases (solid and liquid).
  Low concentration.
  Large particles.

- Need to observe sample.

Solid liquid mixing

- Most common mixing application

- The problem with solids in processes
  - 90 % of “fluid only” plants achieve project goals
  - 60 % of solid handling plants achieve project goals
  - ~ 3 months start up time for a “fluid only”;
  - 9- 18 months for a solids handling plant

Background

- In DuPont, 80% of products are particulate solids or have solids handling steps in their process.

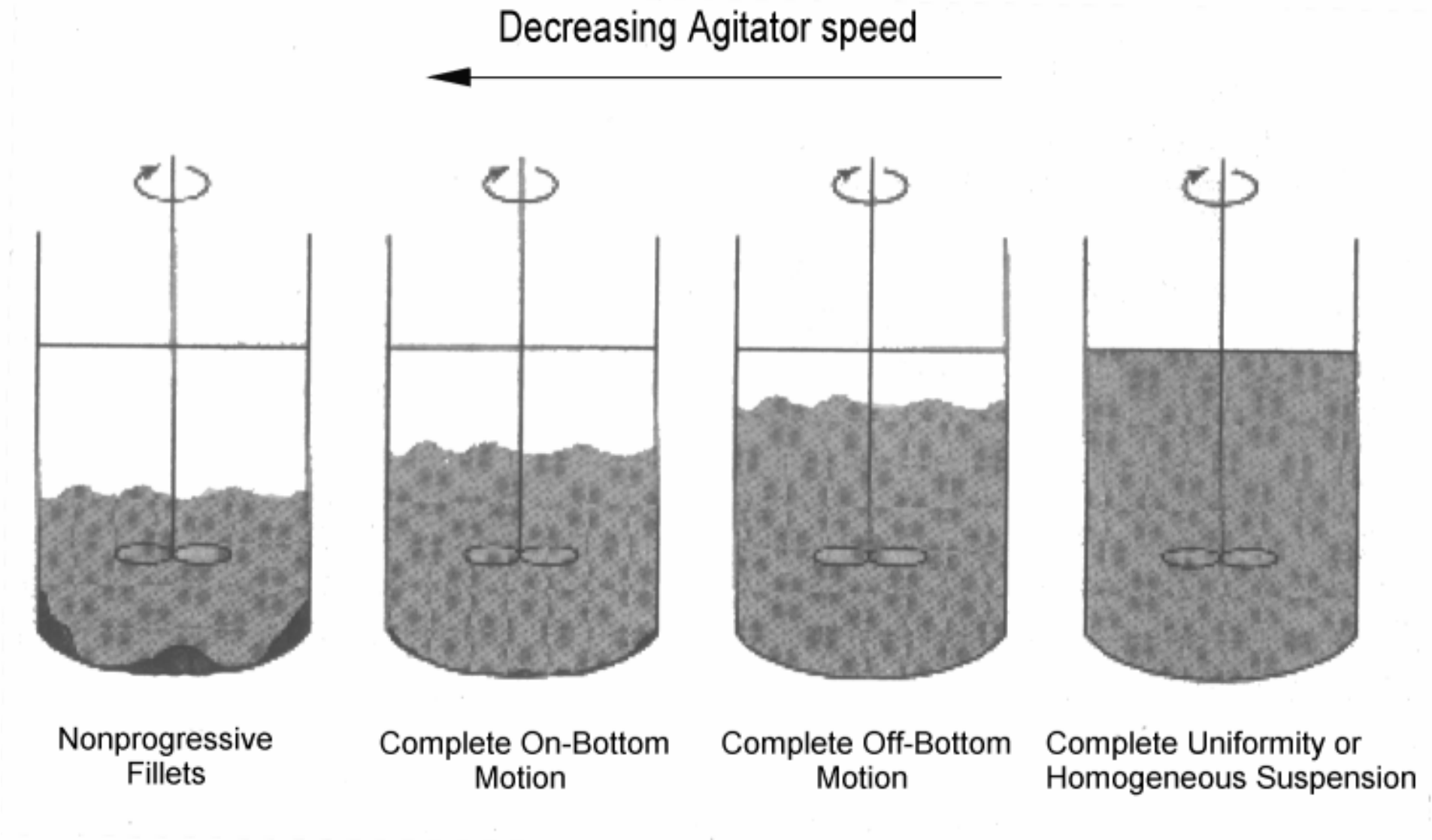
- Plants which have solids handling steps are most likely to experience problems on start-up.

- Examples of problems:
  - Slurry transport in pipelines – plugging when particles settle out:
    - Insufficient velocity.
    - Poorly designed piping - elbows, valves etc.
  - Particle attrition in equipment - separation problems.
  - Poor mass transfer rate:
    - Settled particles in reactors (e.g. catalyst).
  - Etc. etc.

SOLID LIQUID MIXING

- Lots of good data
- Hard to correlate
- Correlations not readily obvious
- Three process results
  - Off bottom
  - Mass transfer
  - Vertical distribution

2. Solid suspension



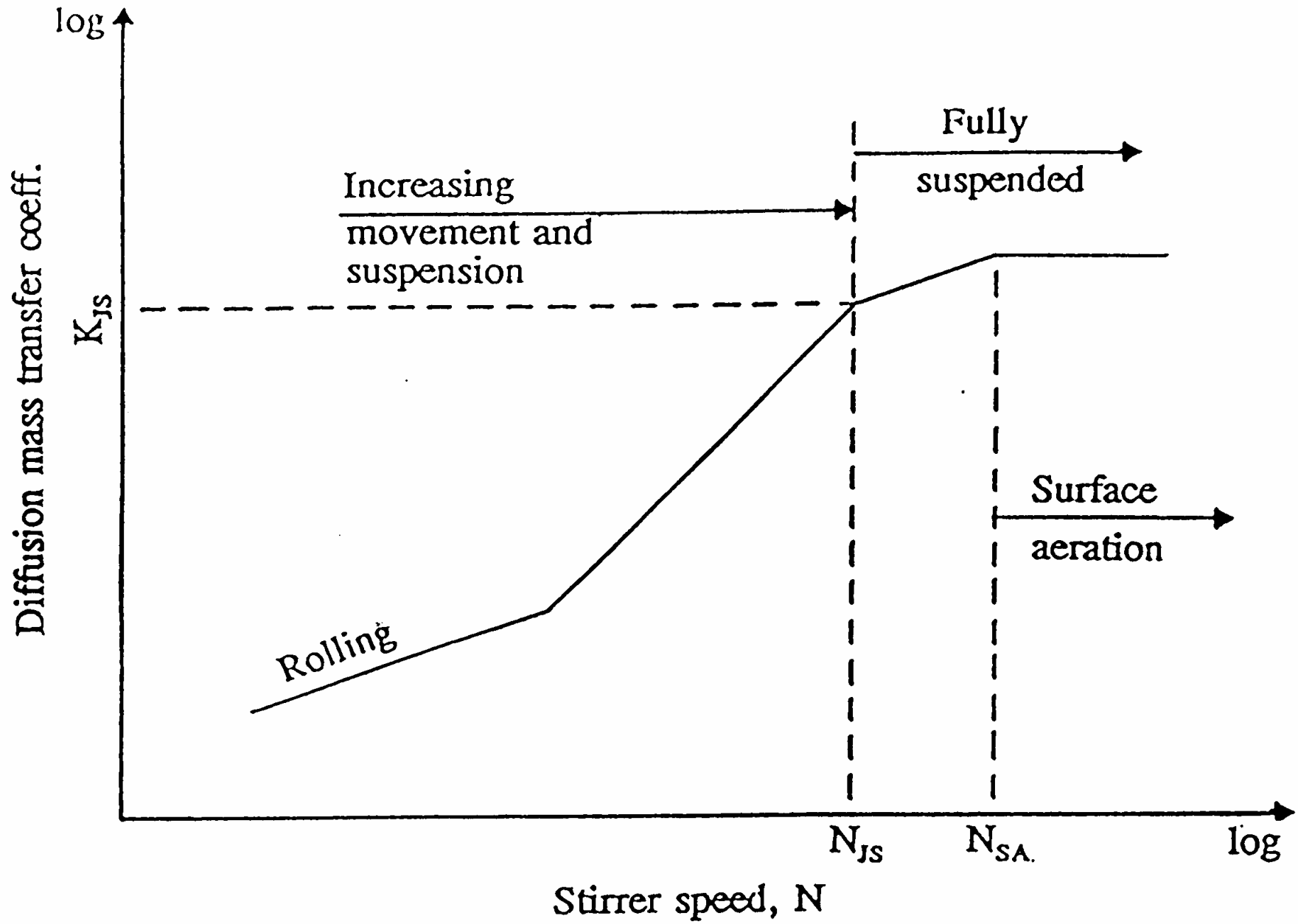


Figure 2. Solid-liquid mass transfer coefficient over a wide range of stirrer speeds.

Solids Suspension and Mass Transfer

- A great deal of work has been done in this area (see Davies, Nienow, Armenante & others).

- Data are correlated by Sherwood No.

$$Sh = \frac{k_L d}{D_{AB}} = 2.0 + xRe^y Sc^{1/3}$$

- Values of *x* and *y* appear to be dependent on particle size.

- Need to compare particle size with eddy length scale in defining Reynolds No.

Settling Rate

- Laminar - Stoke's Regime:

$$U_{TO} = \frac{(\rho_S - \rho_L) g d^2}{18\mu}$$

$$\frac{U_{TO}^2}{(\Delta\rho / \rho_L) g d} = \frac{1}{18} \frac{\rho_L U_{TO} d}{\mu}$$

$$Fr_G = \frac{Re_P}{18}$$

Settling Rate

- Turbulent - Newton's Regime:

$$U_{TO} = 1.74\left(\frac{(\rho_S - \rho_L)gd}{\rho_L}\right)^{1/2}$$

$$\frac{U_{TO}^2}{(\Delta\rho/\rho_L)gd} = 1.74^2$$

$$Fr_G = 3.0$$

Settling Rate

- Transitional - Allen's Regime:

$$U_{TO} = 1.04\left(g\,\frac{(\rho_S - \rho_L)}{\rho_L} \right)^{0.72} \frac{d^{1.18}}{\nu^{0.45}}$$

$$\frac{U_{TO}^{1.44}}{((\Delta\rho/\rho_L)gd)^{0.72}} = 1.04\,\frac{U_{TO}^{0.44} d^{0.46}}{\nu^{0.45}}$$

$$Fr_G = 1.04 Re_P^{0.3125}$$

SOLID SUSPENSION

- What is needed?
- OFF BOTTOM
- Dissolving and many chemical reactions
  - Off bottom to maximize surface area
- Some storage and delivery
- UNIFORM DISTRIBUTION
- Sampling
- Crystallization and precipitation
- Feeding

2.1 Mechanistic models for solid suspension

- **Turbulence model**

  Suspension due to energy transfer from turbulent eddies of size similar to that of d_p .

- **Fluid velocity model**

  Suspension due to average velocity and hydrodynamic forces (lift and drag) acting upon the particles.

Which is correct?

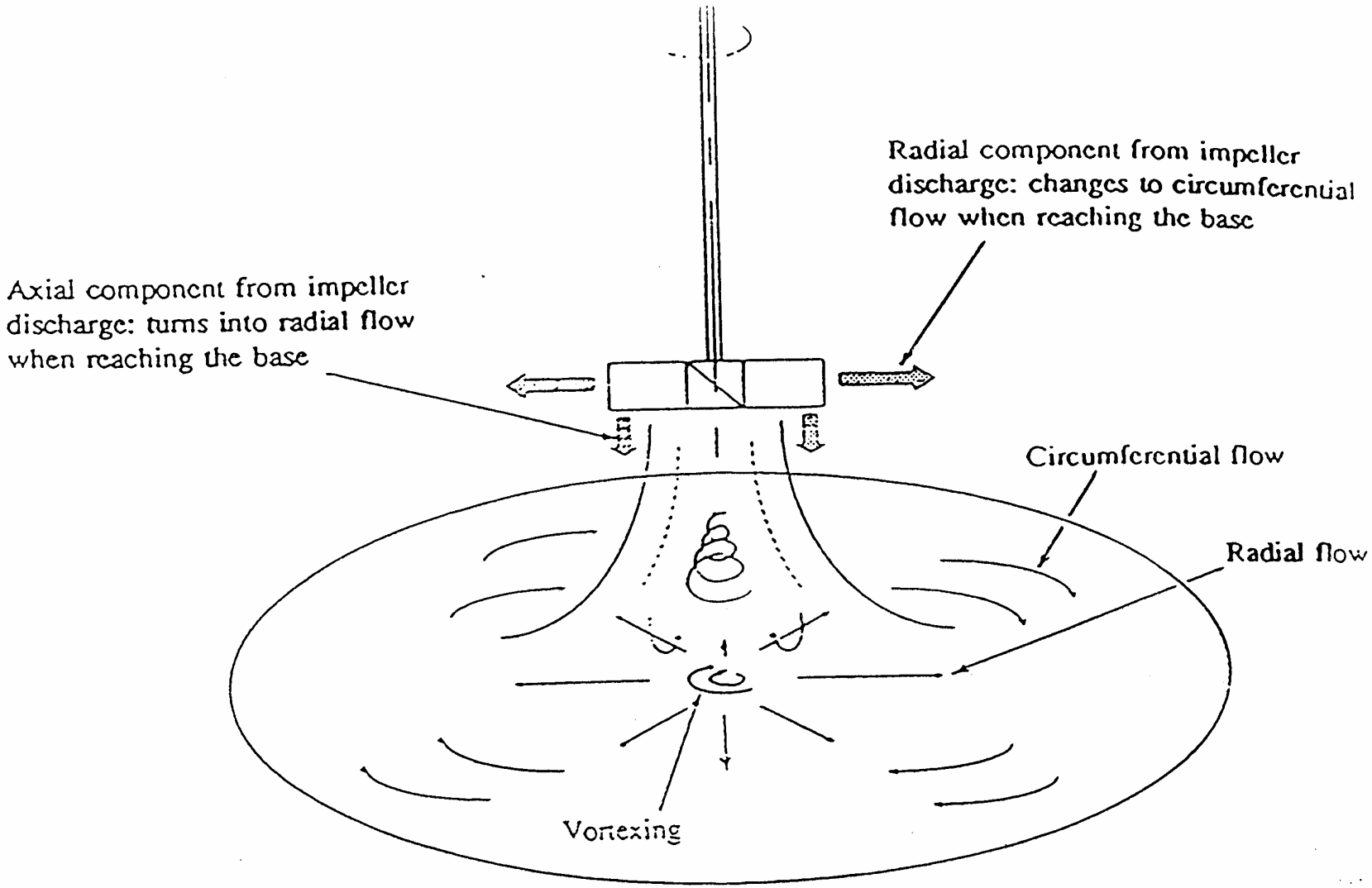


Figure 4. Relationship between the flow patterns discharged by an impeller and those acting on particles on the vessel base.

2.1 Models for solid suspension (ct.)

Neither of the two classes of models alone represent the exact physical conditions.

Both convection and turbulence act on settled particles.

2.2 Semi-empirical correlation by Zwietering

$$N_{JS} = s\,\nu^{0.1}\left(\frac{g\Delta\rho}{\rho_L}\right)^{0.45} X^{0.13}\, dp^{0.2}\, D^{-0.85}$$

No solids remain at the vessel base for more than 1-to-2 seconds

Extensive data sets can be correlated

Some theoretical justification

Limitations and applicability

- **“s” values have to be known**
  - **Characteristic of geometry only - maybe**

- **not applicable outside the experimental conditions**

- **applies to free and hindered settling conditions (i.e. low to moderate X), X is weight of solids divided by weight of liquid times 100 - UGH**

  but may not be valid or relevant in significantly non-Newtonian systems (i.e. at high X)

Table 1. Geometrical constant, s, values for three different impeller types in a flat base vessel.

s values at C/T =		**1/3**	**1/4**	**1/6**	**1/8**
RDT	D = T/2	4.72	4.2	3.67	3.41
	D = T/3	8.37	7.43	6.50	6.03
PBT4-45	D = T/2	4.97	4.74	4.52	4.40
	D = T/3	6.95	6.64	6.32	6.16
HE-3	D = T/2	7.14	6.82	6.50	6.35
	D = T/3	10.41	9.95	9.48	9.25

Table 2. Operating conditions over which Zwietering's correlation is obtained

Parameter	Range covered
$\Delta\rho$(kg/m^3)	560 – 1800
d_p (μm)	140 – 520
X(%)	0.5 – 20
T(m)	0.1 – 0.6
D/T	1/6 – 1/2
n_b	4
C/T	1/20 – 1/2
Impel. type	4
Vessel base shape	Flat, dish & conical
μ_L(mPa s)	0.3 - 9

Z CORRELATION

- Since has been extended to many more geometries and sizes and densities

- Not perfect but conservative and nothing better

Main Findings

- **Impeller Geometry**
  - **Axial flow impellers (e.g. hydrofoils) pumping downwards are the most; radial flow (e.g. 6DT) and saw tooth impellers are the least energy efficient for off-bottom suspension.**
  - **Low C generally more energy efficient, except at extremes (e.g. D/T>0.5 C/T<1/6)**

Findings

- **Tank Geometry**
  - **Flat base tanks and dished**
  - **not recommended: cone**

  - **Multiple impellers for processes where H varies**

2.3 Scale-Up Criteria for Suspension Speed

- **Geometrical similarity (Figure 5)**

$$\frac{D_1}{T_1} = \frac{D_2}{T_2}; \frac{W_1}{D_1} = \frac{W_2}{D_2}; \frac{H_1}{T_1} = \frac{H_2}{T_2}; etc.$$

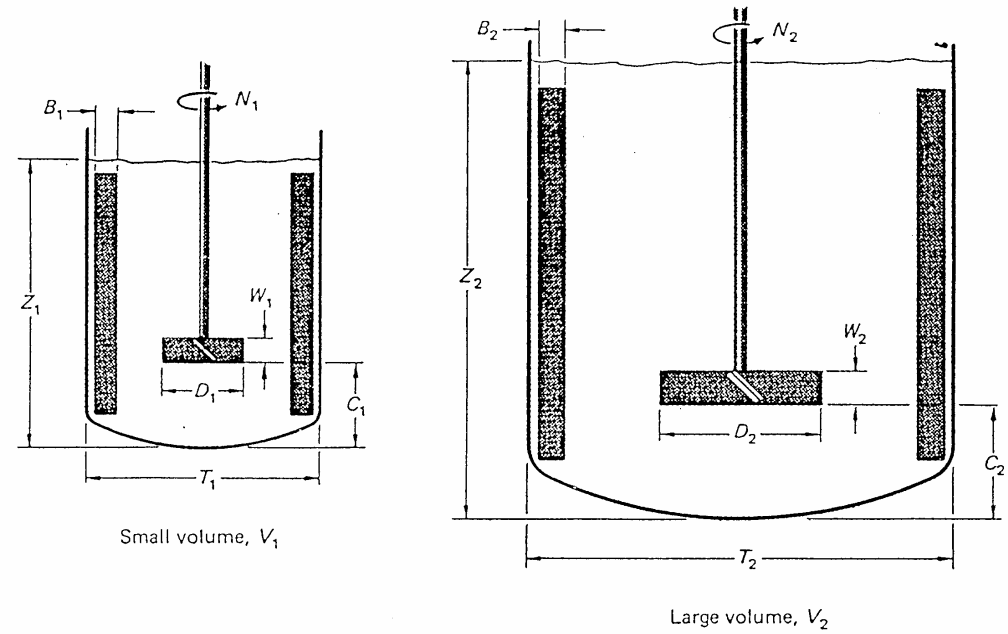


Figure 5. Geometrical similarity as an important scaling criteria

2.3 Scale up (ct'ed)

- **Zwietering's Correlation**

$$N_{JS} \propto D^{-0.85}$$

N_{JS} decreases as scale increases, however, there is some disagreement between workers on the exact value of the exponent on *D*.

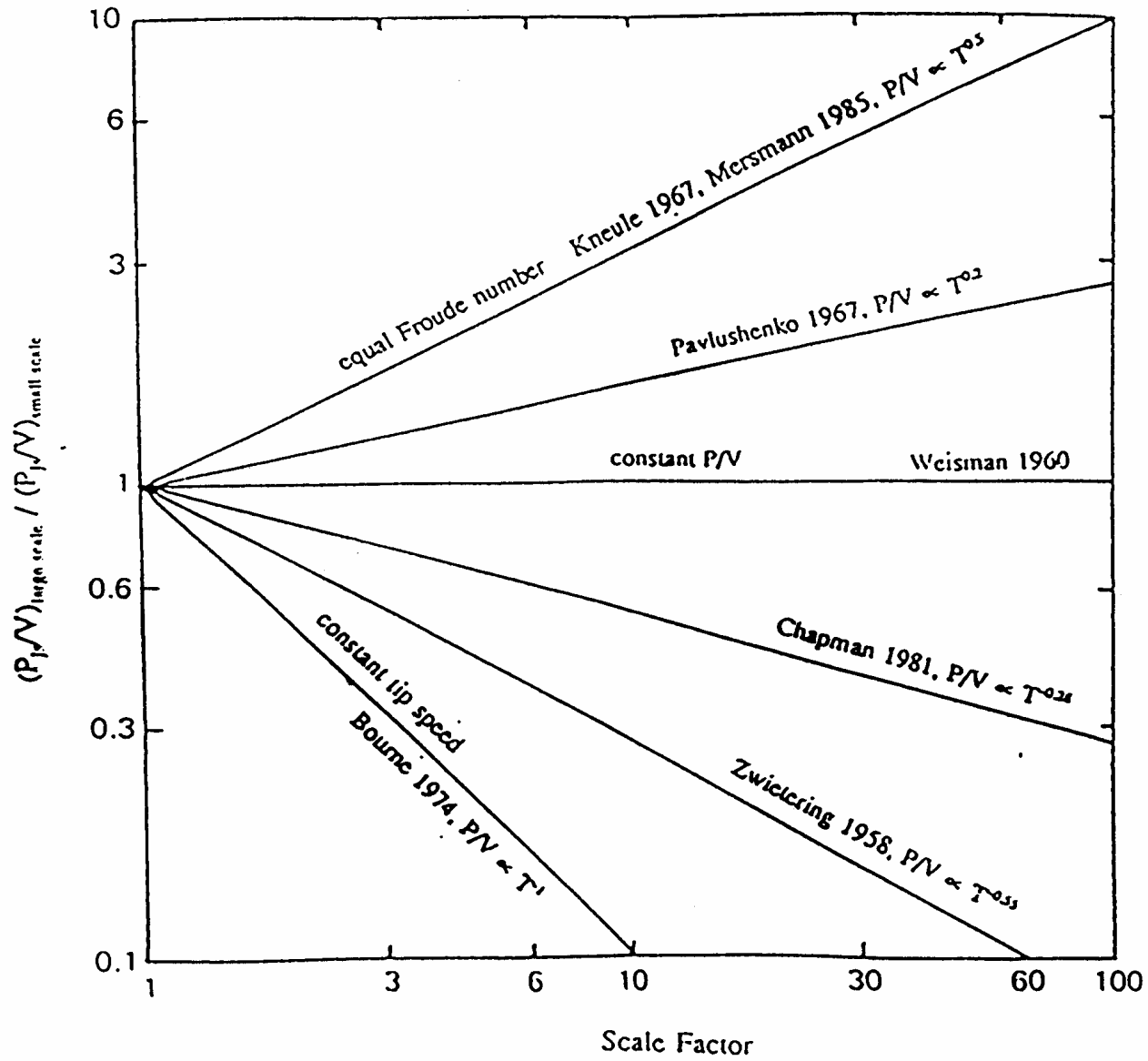


Figure 3. Scale-up rules for solid suspension as published in literature.

SETTLING RATE AND Z

- Zwietering suggests

- Njs proportional to density difference to 0.45 and particle size to 0.2
- Settling rate is density difference times particle size square or to the first
- Density is much more important
- Most data in transitional settling rate

SETTLING RATE AND Z

- At extremes – very large and very small particles – Z breaks down
- Is there another group missing? –
  - Galileo or Archimedes Number
  - Separates settling regions
- Settling rate is in quiet liquid
  - What is the effect of settling in turbulent field?
- Stay tuned

SUMMARY

- Zwietering is for mainly sand of several 100s microns in water – good simulant
- Use Zwietering directly or from scale down experiments
- Note – Zwietering is NOT settling rate
  - Density dominates not particle size

3. SOLID DISTRIBUTION - Introduction

Off-bottom suspension $\Rightarrow$ mass transfer

Certain degree of homogeneity required for other processes

The mechanisms considered for distribution: <u>convection</u> and <u>turbulence</u>. Convection distributes particles by drag force, the turbulence eddies act to distribute at a smaller scale by fluctuating drag forces acting in all directions.

3.2 Measurement and assesment

- Different experimental techniques: conductivity, sampling, slurry height,…

- Mixture quality assessed in terms of:
  - slurry height,
  - Relative Standard Deviation,
  - concentration profile

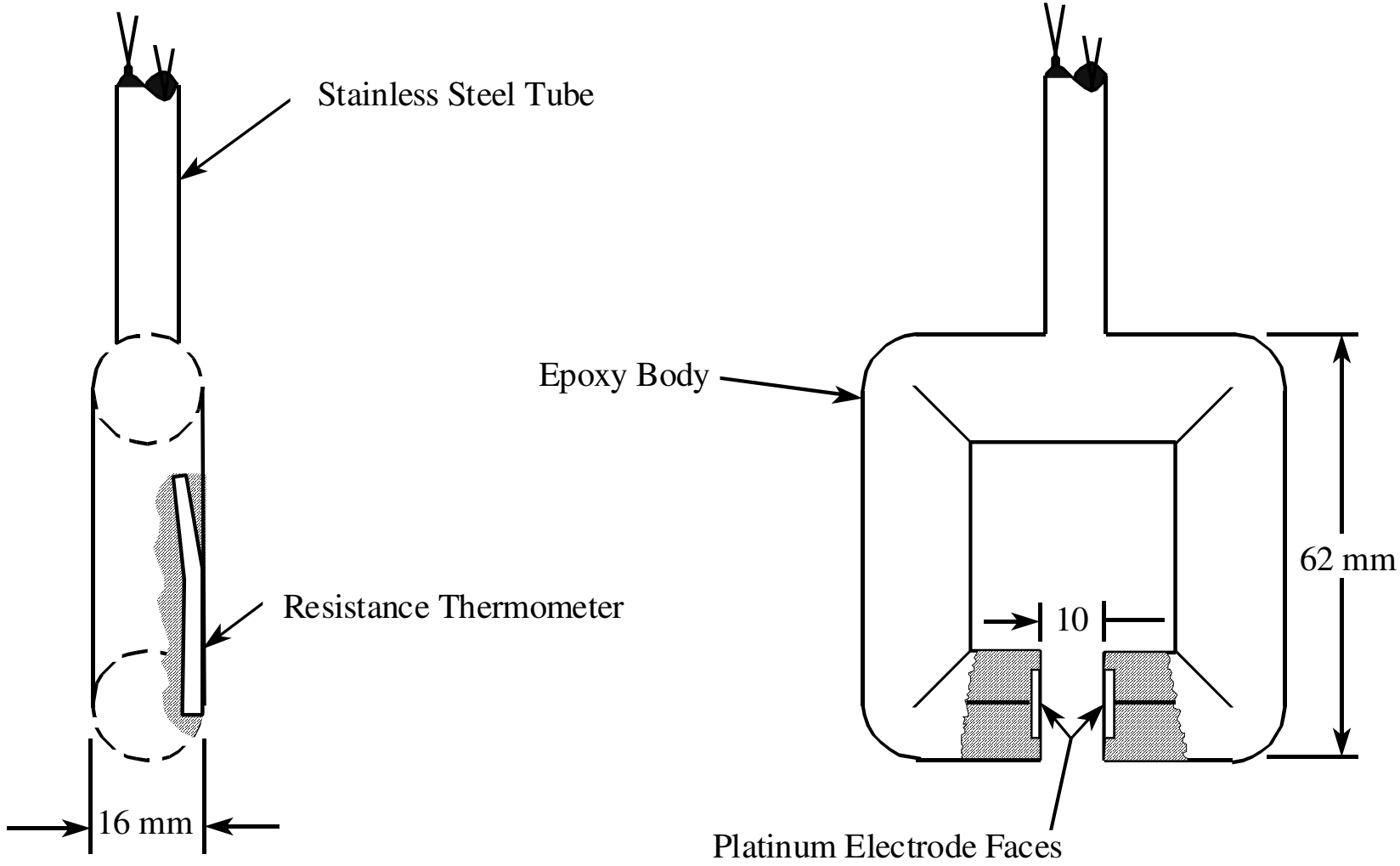
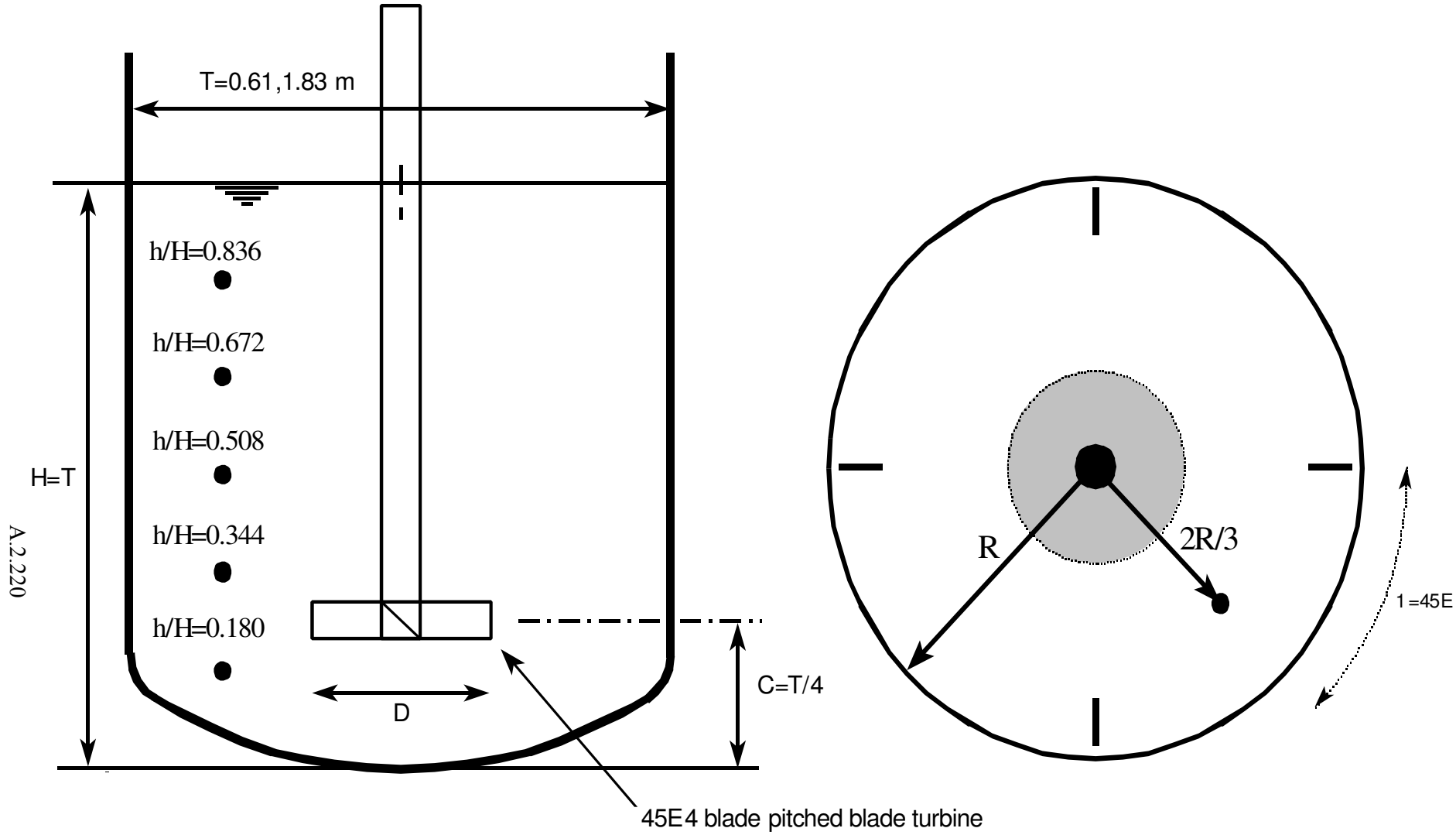


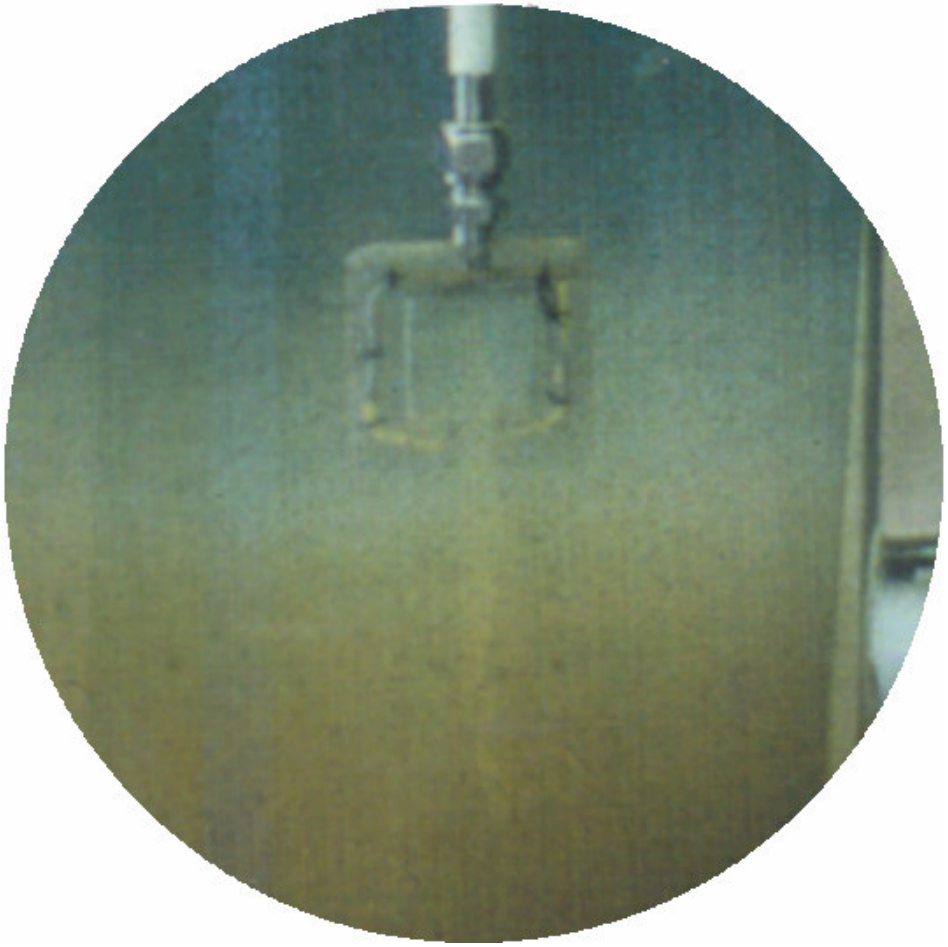
Figure 6. Conductivity probe used at BHR Group to measure local solids concentration



A.2.220

Figure 7. Measurement positions in experimental studies at BHR Group (Taylor, 2000)

beam



Relative Standard Deviation (RSD)

$$RSD = \frac{1}{C_m}\left[\frac{1}{n-1}\sum_{1}^{n}\left(C_{ij} - C_m\right)^2\right]^{\frac{1}{2}}$$

C_m: Calculated mean bulk solid concentration

C_{ij} : Measured local solid concentration

n : Number of samples

RSD = 0 for perfect homogeneity

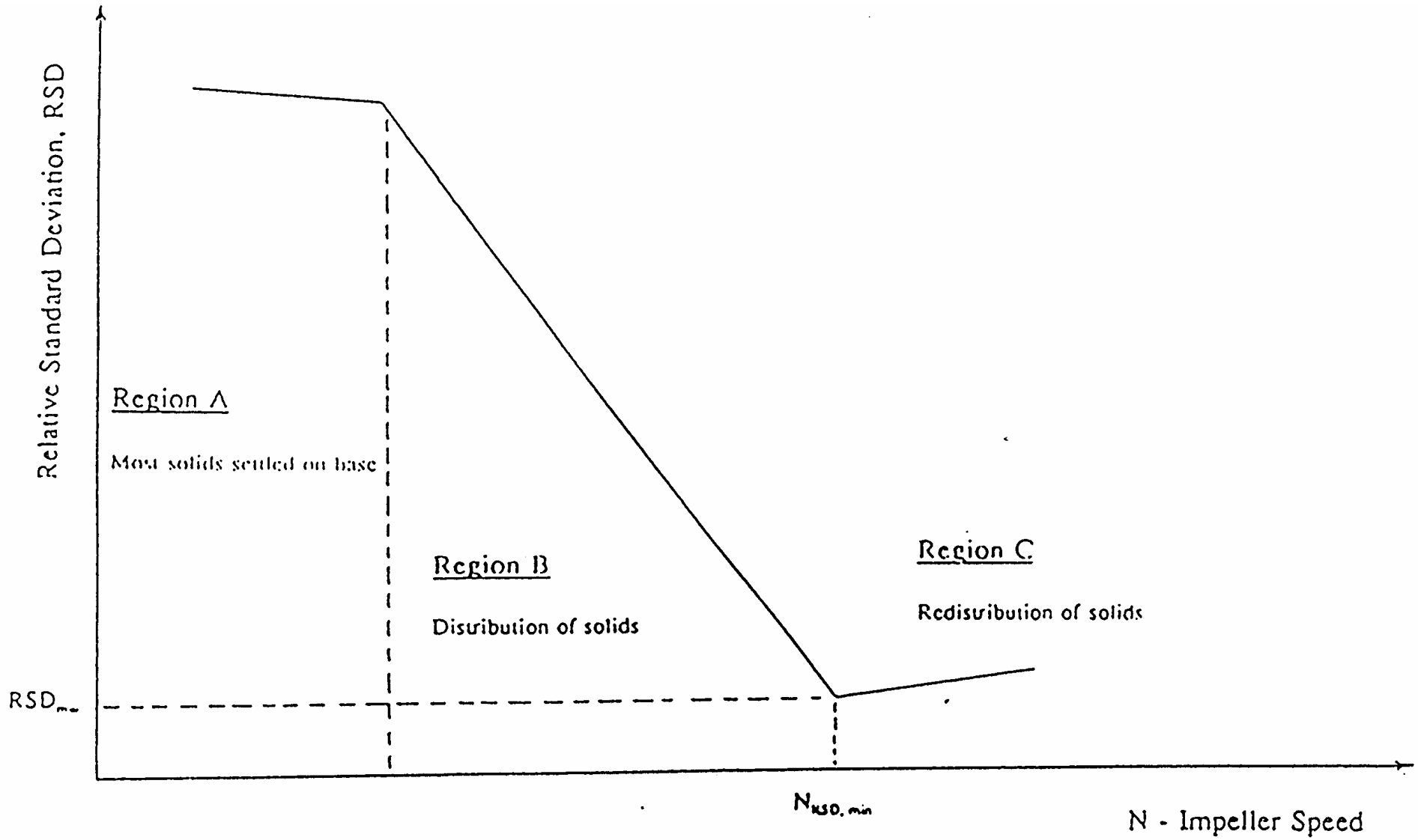


Figure 9. A typical solid distribution curve.

3.2. Measurement and assessment (ct'ed)

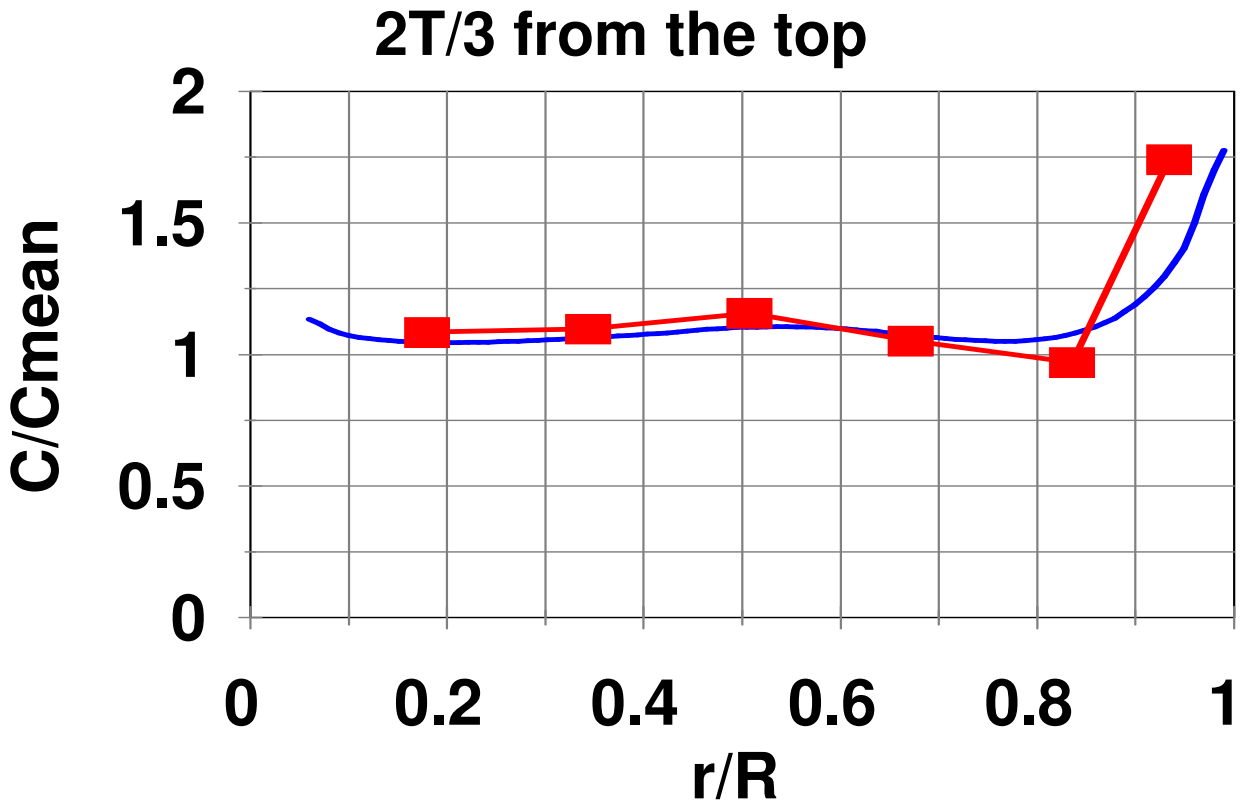
- RSD a single value assigned to the whole mixture

  but does not provide information on how solids are distributed

- Concentration profiles required for a detailed assessment and

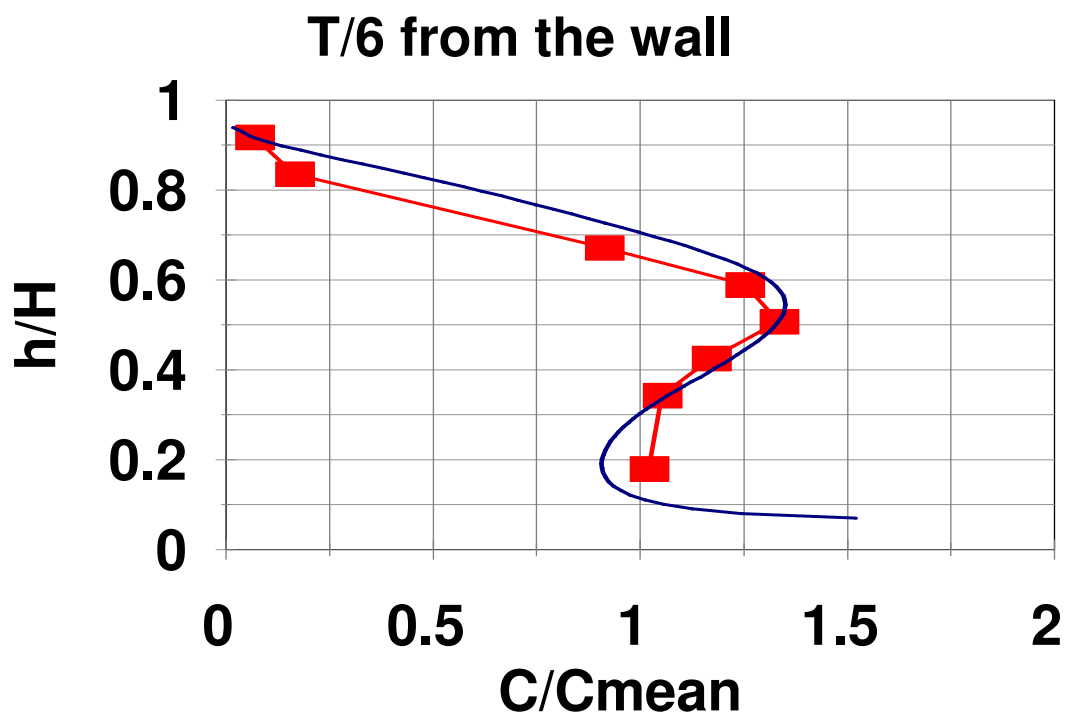
  and to correctly position the inlet and outlet in a CSTR

3.3 Major points related to solids



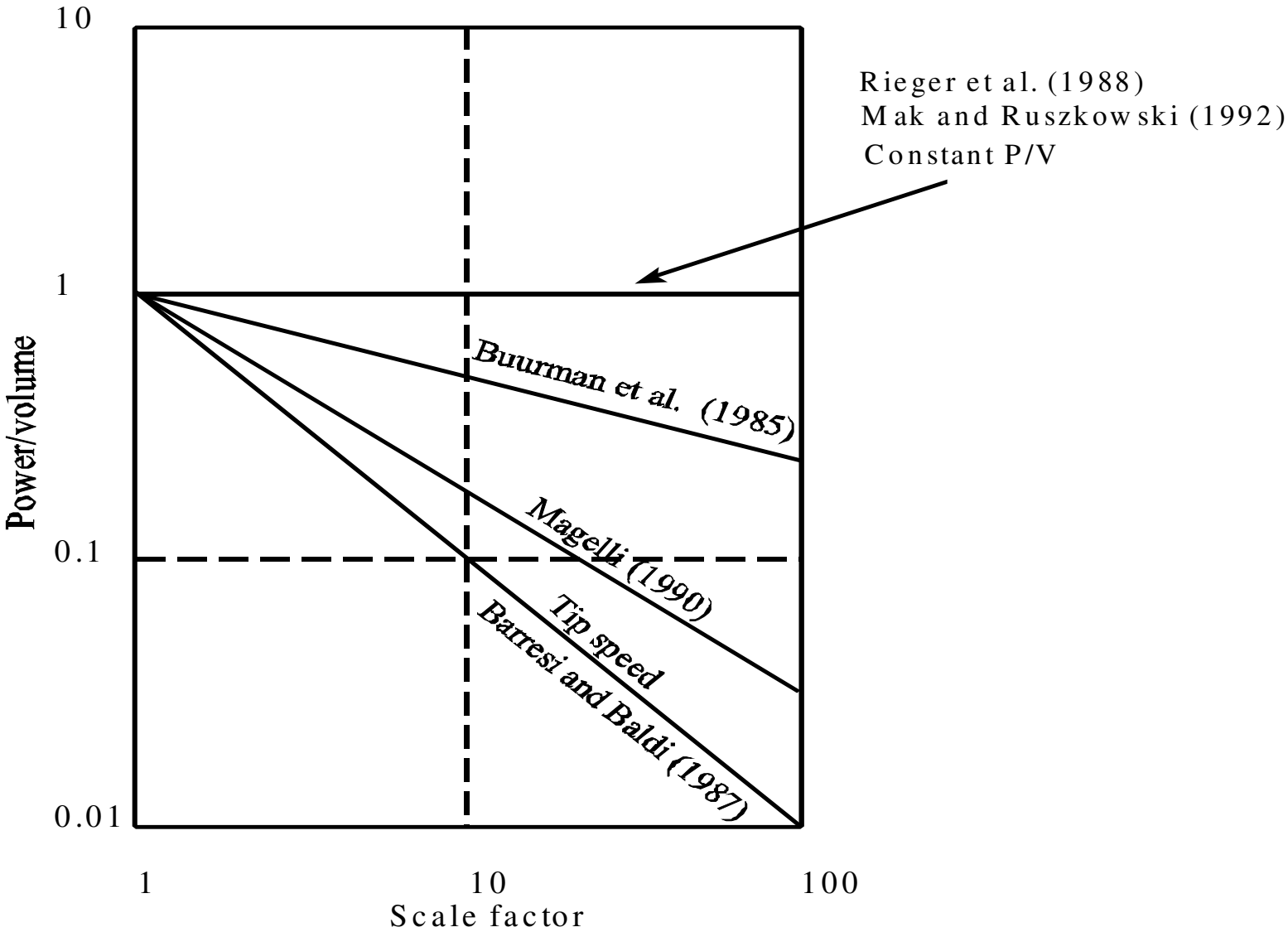
Practically uniform radial concentration profiles

3.3 Major points on solids



- **"Belly plot" on the axial traverse**
- **Position and value of max. depends on *N, C/T, D/T*, particle properties,...**

3.3 Major points on solids



4. DUAL IMPELLER CONFIGURATIONS

- Commonly used in ‘tall’ tanks
- No advantage for suspension: P_{js} higher than in a single impeller configuration.
- Dual impellers markedly improve distribution quality

5. SOLID SUSPENSION USING A JET

- alternative to mechanical agitation
- good off bottom suspension but poor top-to-bottom distribution
- limited access above the vessel, there may not be enough headroom to install a gear box and motor
- portable/flexible

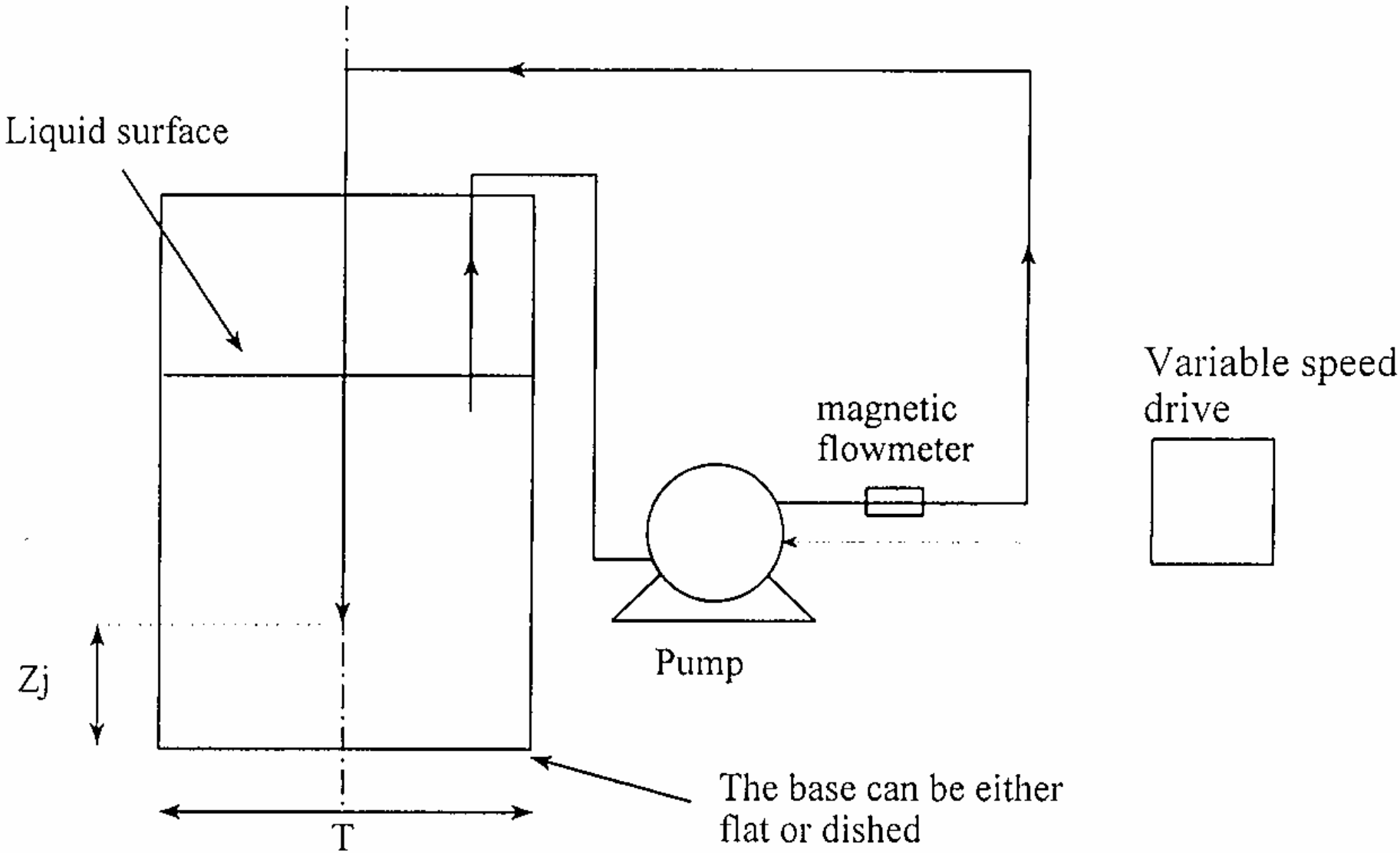


Figure 16. A typical arrangement of a jet mixer for solid suspension.

Advantages of using jets

- mechanical constraints on agitator size in very large vessel
- energy efficiency for solid suspension in comparison to agitation tanks depends on the specific design
- easier to install in wide, shallow vessels: agitator system requires extra supports

Advantages of using jets

- No moving parts in vessel : jet motion induced by an external pump (reduced maintenance)

- low maintenance cost: jet mixing system is very simple and pump is remote from the tank

- low capital cost: pump may already be present for tank drainage.

PULSE JETS

- Not steady
- Multiple jets in one tank
- Being studied for waste work
  - Parsons/SWPF, Battelle/WTP
- Off bottom similar to steady if short off time or slow settling
- Distribution strongly affected

Effect of Physical Properties

- The effects of changes in settling rate from changes in physical properties can be predicted:

$$d \uparrow \Rightarrow U_{TO} \uparrow$$

$$\frac{\Delta\rho}{\rho_L} \uparrow \Rightarrow U_{TO} \uparrow$$

$$\mu \uparrow \Rightarrow U_{TO} \downarrow$$

- Effect of concentration:
  - Increasing concentration slows settling velocity.

Mechanistic Approach

- Solids suspension is not governed by balance between settling velocity and mean flow.
- Role of turbulent eddies?
- Pick up and re-suspend particles from the vessel base.
- Baldi (*Chem Eng Sci.* 1978) looked at the gravitational force acting on a particle and the fluid force acting on it due to turbulence.

$$(\rho_S - \rho_L) g d^3 = K \rho_L (u')^2 l_E^2$$

Mechanistic Approach

- The size of the eddies influencing the motion of the particles will be the size of the particles

$$l_E \approx d \qquad\qquad u' \propto \sqrt{gd(S-1)}$$

- The fluctuating velocity can be related to the local energy dissipation rate:

$$u' \propto (\varepsilon l_E)^{1/3} \rightarrow (\varepsilon d)^{1/3}$$

- The local EDR is proportional to the average power input per unit mass:

$$\varepsilon \propto PoN_{JS}^3 D^2 \left(\frac{D}{T}\right)^3$$

- N_{JS} is the impeller speed required to just suspend the particles.

Mechanistic Approach

- Re-arranging:

$$Po^{1/3} N_{JS} D^{2/3} \left(\frac{D}{T}\right) d^{1/3} \propto \sqrt{gd(S-1)}$$

$$N_{JS} = s \frac{d^{1/6} (g(S-1))^{1/2}}{Po^{1/3} D^{2/3}} \left(\frac{T}{D}\right)$$

- *s* is a dimensionless constant. Its value must be determined experimentally.

- Note: weak effect of particle size and no effect of viscosity.

Zweitering - Experimental

- Work done in late 50's.
- Measured speed required to just suspend particles.
- No particle stationary on vessel base for longer than 1 - 2 seconds.

$$N_{JS} = s\frac{\nu^{0.1} d^{0.2} X^{0.13} (g(S-1))^{0.45}}{D^{0.85}}$$

$$X = 100 \times \frac{M_S}{M_L}$$

- Correlation confirmed in vessels up to 9 ft. diameter.
- Very similar functionality to mechanistic form.

Values of “s”

Impeller	*D / T*	*c / T*	*Po*	*s*
Hydrofoil	0.33	0.25	0.30	9.3
Hydrofoil	0.50		0.30	7.0
PBT – 45	0.33		1.73	4.6
PBT – 45	0.50		1.53	6.1
FBT – 90	0.33		3.19	4.4

- Hydrofoils need to operate at higher speeds than PBT’s.
- Which impeller type is most energy efficient for solids suspension?

Scale-Up

- Very confident about Zweitering's correlation:
  - Good set of data.
  - Taken at large scale.

- What happens to power requirements on scale-up?

$$\frac{N_L}{N_S} = \left(\frac{D_S}{D_L}\right)^{0.85} \qquad \frac{\varepsilon_L}{\varepsilon_S} = \frac{N_L^3 D_L^2}{N_S^3 D_S^2}$$

$$\frac{\varepsilon_L}{\varepsilon_S} = \left(\frac{D_S}{D_L}\right)^{3\times 0.85-2} = \left(\frac{D_S}{D_L}\right)^{0.55}$$

- Power input per unit mass decreases on scale-up!

Buurman -5th Europ Conf. On Mixing

- Buurman worked for Shell in the Netherlands.
- Studied particle distribution in vessels up to 63 m^3:
  - Solids suspended to 90 % of liquid depth.
- For $H = T$, $D = 0.4\ T$ and $L = 0.9\ T$:

$$\frac{\rho N^2 D^2}{g \Delta\rho d}\left(\frac{d}{D}\right)^{0.45} \geq 20$$

- For fixed geometry and physical properties:

$$N^2 D^{1.55} = K \qquad\qquad N^3 D^{2.33} = K'$$

- Almost constant power input per unit mass.

Mak / FMP Consortium

- FMP Consortium has published early work on solids distribution.
- Measured distribution in vessels of 2, 6 and 9 feet diameter.
- Used conductivity technique:
  - Conductivity proportional to slurry concentration in probe volume.
  - Calibrated in fluidized bed.
- Measured concentration at different axial positions.
- Best work in field.

Data Analysis

- Defined: Relative Standard Deviation of Concentration:

$$RSD = \frac{1}{\overline{C}} \sqrt{\frac{\sum_{i=1}^{i=n} (C_i - \overline{C})^2}{n-1}}$$

- Plot *RSD* versus mixing parameters (speed, power input, Froude No. etc. etc.).

- Look for parameter that “collapses” curves taken at each scale.

- Look for relationship between distribution and N_{JS}.

Distribution versus N_{JS}

- Some impellers achieve uniform dispersion when operating at N_{JS}, e.g. large PBT.
- Others have poor distribution, e.g. hydrofoils.
- Operation at “just suspended” point is no guarantee of uniform dispersion.
- An area of active research
  - CFD and BHRG FMP experiments

Example of C_i versus N

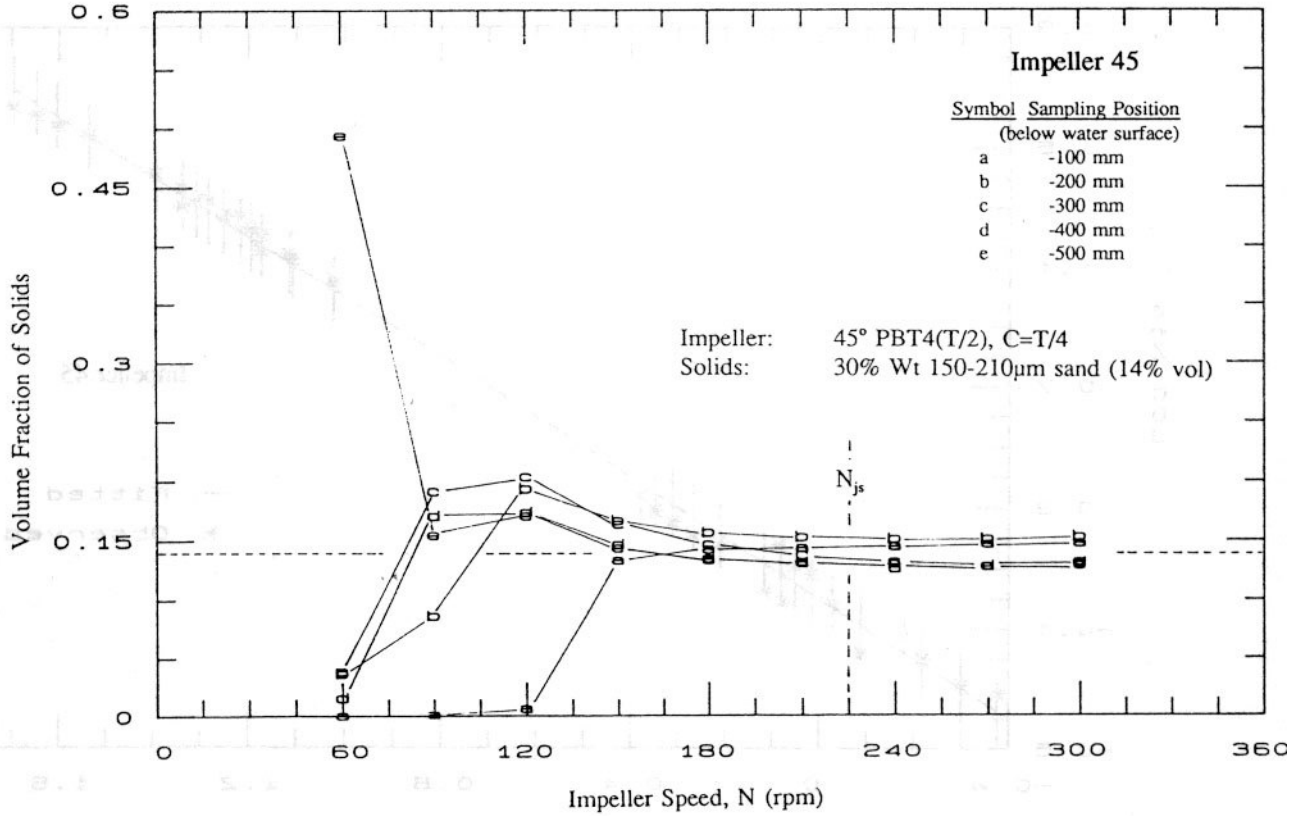


Fig 13 Plot of Solids Concentration against Speed at different Sampling Locations

RSD versus N

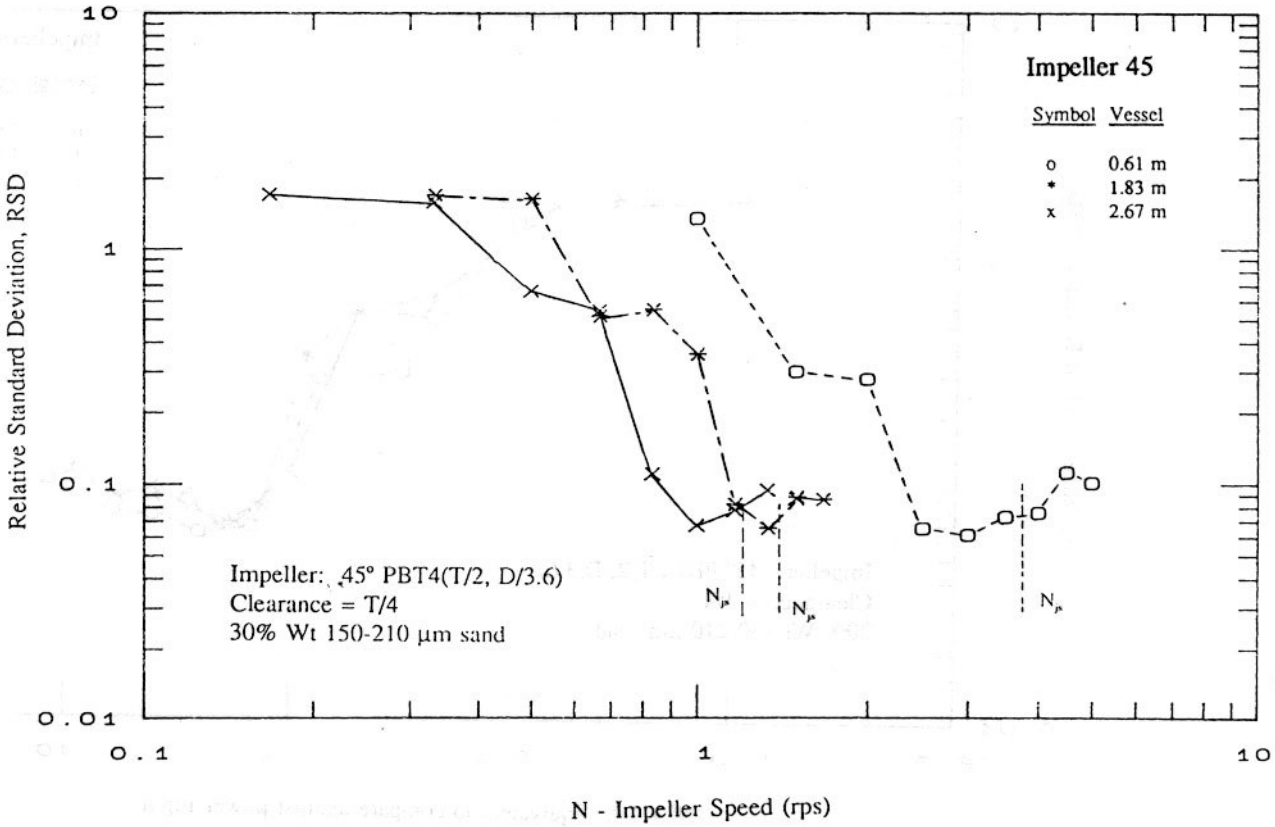


Fig 15 Plot of RSD against Impeller Speed for 3 Scales

RSD versus Po N^3 D^2

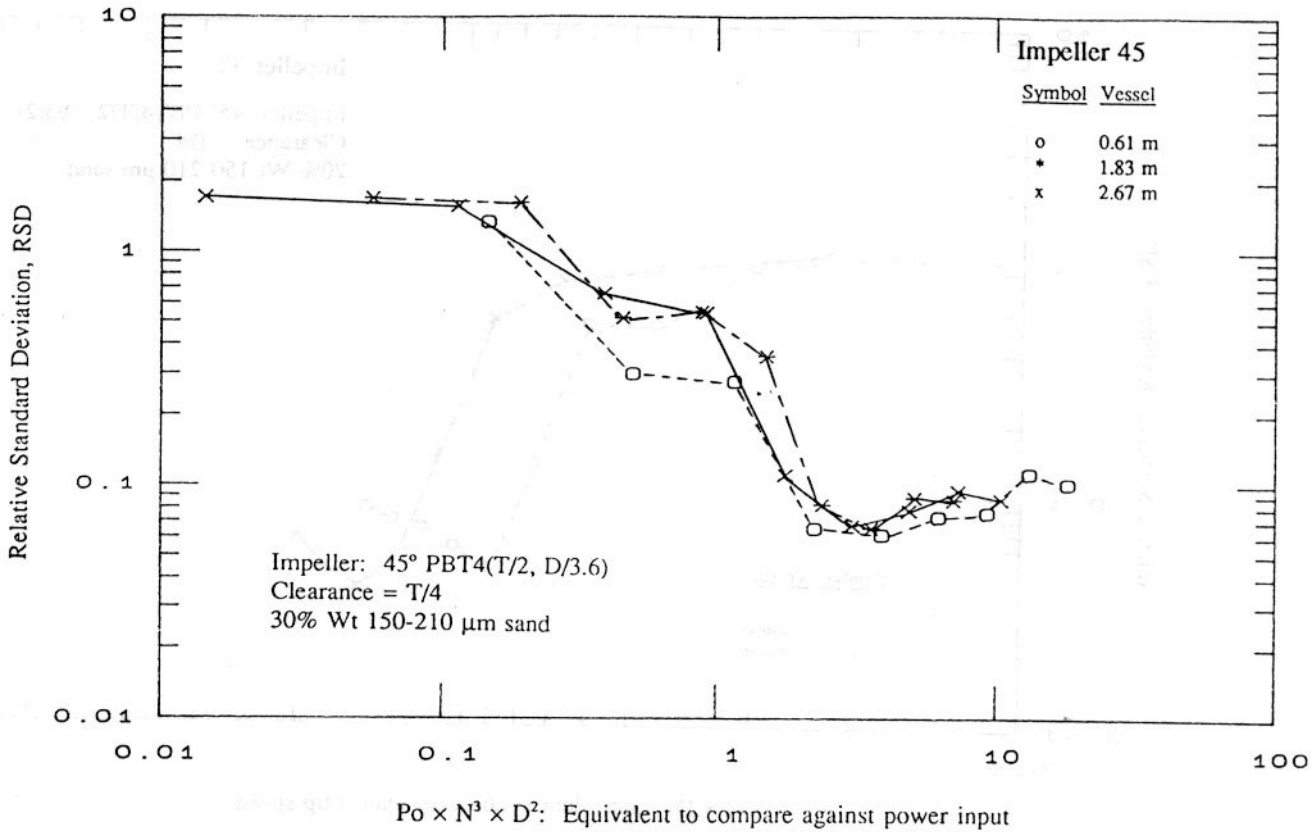


Fig 18 Solids Distribution Scale-up Data: comparing against power input

RSD versus N $D^{0.78}$ - Buurman's

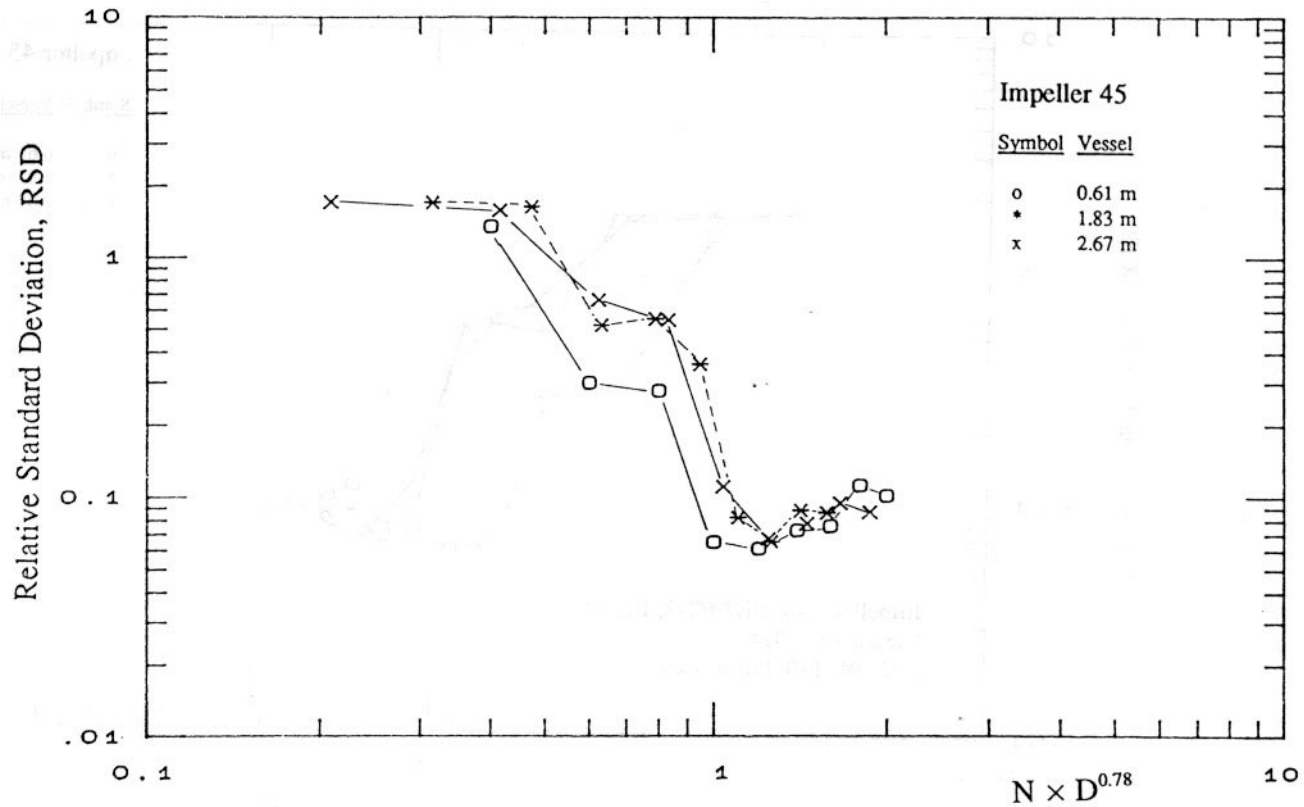


Fig 14 Solids Distribution Scale-up Data according to Buurman's Model ($N_{RSD} \propto D^{-0.78}$)

Plots of RSD - Summary

- *RSD* versus *N*:
  - N_{JS} decreases on scale-up.
  - No relationship exists for *RSD.*

- *RSD* versus $Po\ N^3\ D^2$:
  - Equal power input per unit mass.
  - Very good relationship between *RSD* at each scale.

- *RSD* versus $N\ D^{0.78}$:
  - Buurman's Froude No. model.
  - Good relationship, but not as good as equal power input per unit mass.
  - Seems to work well at larger scale.

Density

- Need to know fluid density for power calculations.

- When $N < N_{JS}$: $\rho_{SL} = \dfrac{1}{\dfrac{x}{\rho_S} + \dfrac{1-x}{\rho_L}}$ $\rho = \rho_L$.

- When $N \geq N_{JS}$: $\rho = \rho_{SL}$.

$$x = \frac{M_S}{M_{SL}}$$

Tickler Turbine

- Many vessels operate on a cycle - filling and emptying.

- If vessel contains slurry, low level impeller must be installed:
  - If not, particles will settle out once impeller is uncovered.

- Typical design:
  - Flat blade turbine: $0.33 > D / T > 0.25$
  - Clearance: Within 6 - 12 inches of vessel base.
  - PBT or Hydrofoil: $D / T = 0.50$
  - Clearance: Normal position.
  - Calculate N_{JS} for main impeller.

- Add power of each impeller.

ATTRITION / COMMUNITION

- Reducing particle sizes
  - May be good or bad
- Many particles are agglomerates of primary particles
- Many crystals are fragile
- Size reduction correlates with power per unit volume and time or specific energy
  - Batch and continuous

ATTRITION

- Dupont work
- Used agglomerates – carbon beads
- Breakage by fines coming off agglomerates
  - Common mechanism
- Rate process with time depending on local energy dissipation
- Similar findings on communition in media mills.

ATTRITION

- Do not reach equilibrium quickly
- Time times power
- Hours in a tank can be equivalent to one pass through a pump
- Linear relation

ATTRITION

- Fine particles caused by attrition often limit separation processes
  - Filtration and centrifugation
- Reduce attrition by de-energizing processes
  - Reduce energy input
  - Reduce contact time
    - Holdup in storage tanks
    - recycle

End of M7

M8
SOME COMMENTS ON SCALING
CFD FOR SOLVING MIXING PROBLEMS

GENERAL COMMENTS

- Nature of Mixing and Process Result
- Design mixer or choose operating conditions to achieve desired process result.
- Typically process result is a plateau
  - more mixing - speed and power does not improve the process
- Optimization is often not based on process result but rather on auxiliary conditions

DESIGN AND RATING

- DESIGN PROBLEMS
  - what equipment for a full scale facility
    - configuration
    - impeller type
    - impeller speed
    - power/motor
    - mechanical design

DESIGN AND RATING

- RATING PROBLEMS
  - Given this piece of equipment
  - will it do the desired job
    - required changes

TYPES OF ANSWERS

- PAPER
  - based on physical principles
  - physical properties
  - calculate from empirical theory based equations
  - long term goal
  - about 80 to 90 percent of cases
- Long Term Goal

TYPES OF ANSWERS

- SCALE UP
  - from small scale experiments predict large scale design and performance

SCALE UP VERSUS SCALE DOWN

- Scale up from arbitrary lab equipment to plant reactor which looks different
- Often full scale plant equipment is known or can be guessed with accuracy
- Mixing is very geometry dependent
  - HARD TO PREDICT
- Make lab equipment geometrically similar to plant. SCALE DOWN

SCALING

- Want all parameters on small scale to match large scale - IMPOSSIBLE
- Key parameters scale differently
  - solids suspension with tip speed
  - dispersion with power per unit volume
  - mix time with speed
- Must pick key variable for scaling
- Need to explore sped range on small scale
- Prefer data on two scales

SCALING

- Use geometric similarity
  - reduces scaling to speed
  - need to figure out which is key process result
  - then which fluid mechanic phenomena
- Key parameters
  - speed - position
  - tip speed - addition time
- Scale on that

SMALL SCALE EXPERIMENTS

- NOTE: Computational Fluid Mechanics is an experimental technique
- When?
  - Unusual configurations
  - Unusual process result
  - Unavailable basic data
  - Basic data changes during mixing

ANALYZING LAB EXPERIMENTS

- Philosophy
  - find out what controls
  - talk to chemists
  - define process results
  - run test on small scale to find out above
  - geometric similar equipment
  - vary speed - OBSERVE, OBSERVE
  - any effect - slope versus speed

ANALYZING LAB EXPERIMENTS

- Small scale
  - REAL FLUIDS
- Large Scale
  - Model fluids

SCALING

- Still somewhat of an art form
- Requires experience and knowledge of many fields
  - biochemistry
  - chemistry
  - reaction engineering
  - physical chemistry

ADVANCED TECHNIQUES

- Laser Doppler Anemometry (LDA)
- Computational Fluid Mechanics / Mixing

  CFD/CFM

WHAT WOULD WE LIKE TO KNOW?

- Velocity at very point
- Turbulence at every point

- In Principle could calculate
  - all forces
  - forces on second phase
  - map velocity fields
  - particles and their trajectories
  - concentration and paths

LASER DOPPLER ANEMOMETRY - LDA

- Experimental
  - one or two lasers
  - transparent tank
  - low concentration of tracker fine particles
  - measure vectors - fluctuations
- DPIV - laser sheet
- Digitized sequential pictures
  - lots of data
- Compress and present via computer graphics

COMPUTATIONAL FLUID MECHANICS

- Mathematical technique
- Mathematical modeling
- Assumptions
  - nature of turbulence
  - interphase forces
  - results only as good as assumptions
- Increasing but still limited applicability

COMPUTATIONAL FLUID MECHANICS

- Scale free modeling
- Needs confirmation
- LDA confirms CFD

COMPUTATIONAL FLUID MECHANICS

- Navier Stokes Equations
  - three dimensional partial differential equations
  - general
  - unsteady state
- Stress term
  - Laminar
  - constitutive equation

COMPUTATIONAL FLUID MECHANICS

- CONSTITUTIVE EQUATION
- relation between forces (stresses) and motion usually expressed as shear rate
- Rheological equations in 3D
- 27 element tensor
  - in simplest form - viscosity

COMPUTATIONAL FLUID MECHANICS

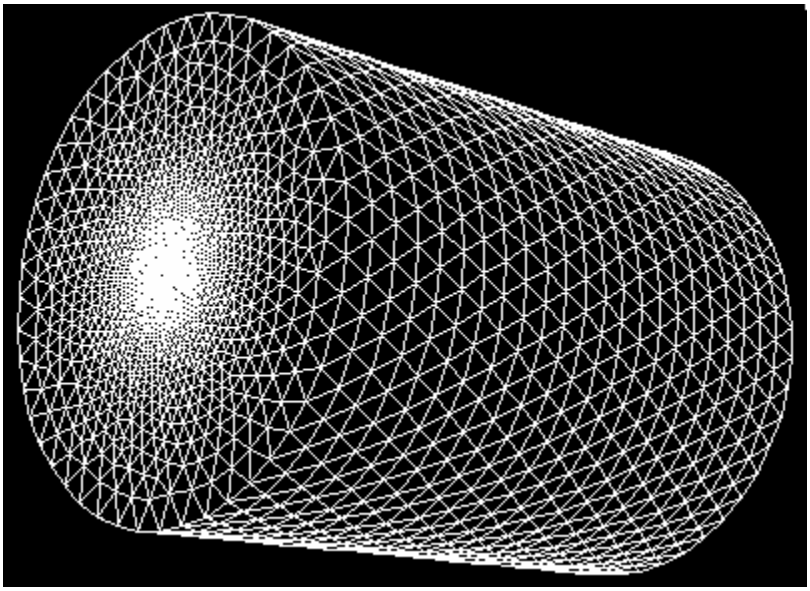
- TURBULENCE
- CONSITUTIVE EQUATIONS - many
- Concept of average and fluctuating velocities
    - V = Vave + V’
  - on averaging introduces extra stresses larger than laminar - turbulent stressses (Reynolds)
  - Turbulent viscosity(diffusivity)* local shear rate

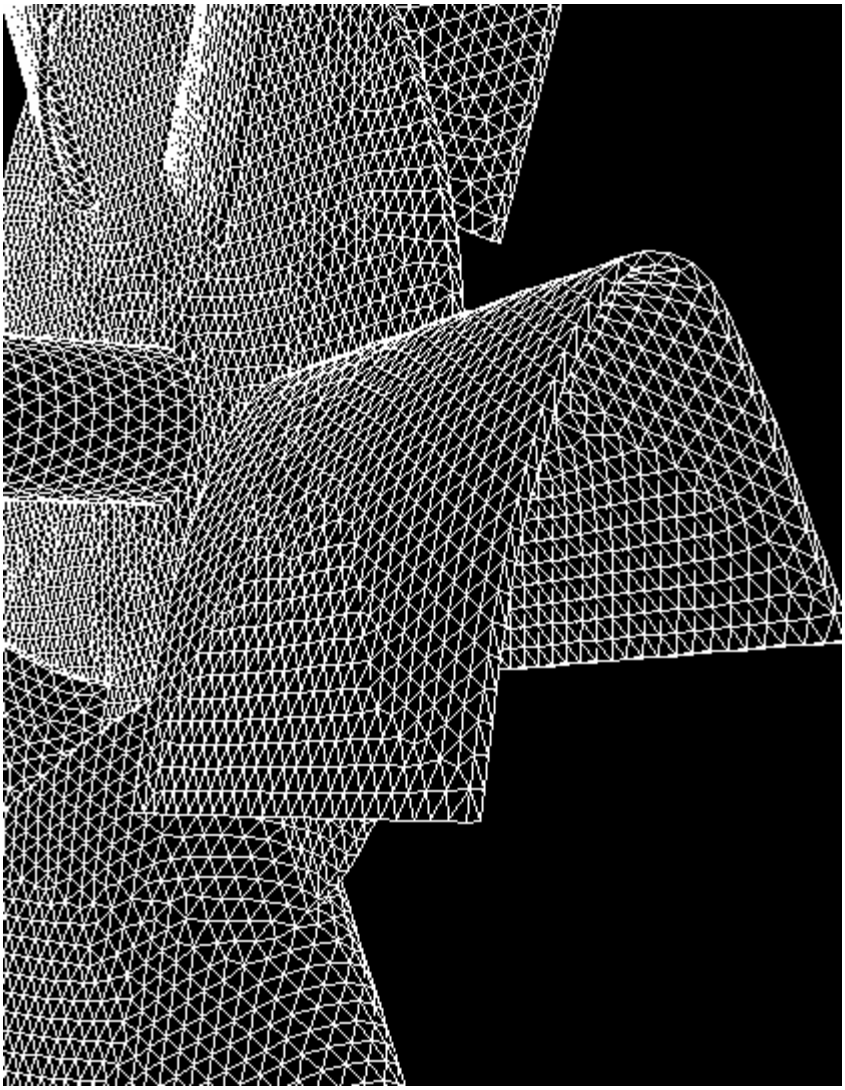
COMPUTATIONAL FLUID MECHANICS

- Methods of solving partial differential equations
  - finite volume
  - finite difference
  - finite element
  - boundary elements
  - body fitted coordinates - rectangular fits
  - mesh generation programs

COMPUTATIONAL FLUID MECHANICS

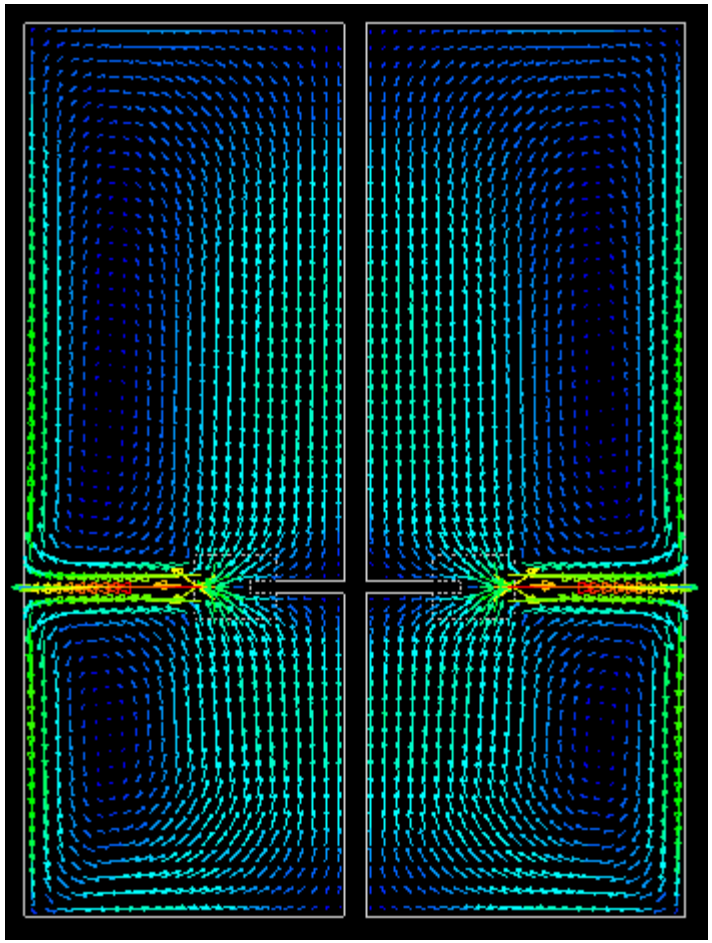
- Lots of mesh points at which variables are calculated (10,000 to 40,000)
- Lots of data
- Graphics display
  - same as for LDA
  - color required
- Particle tracking

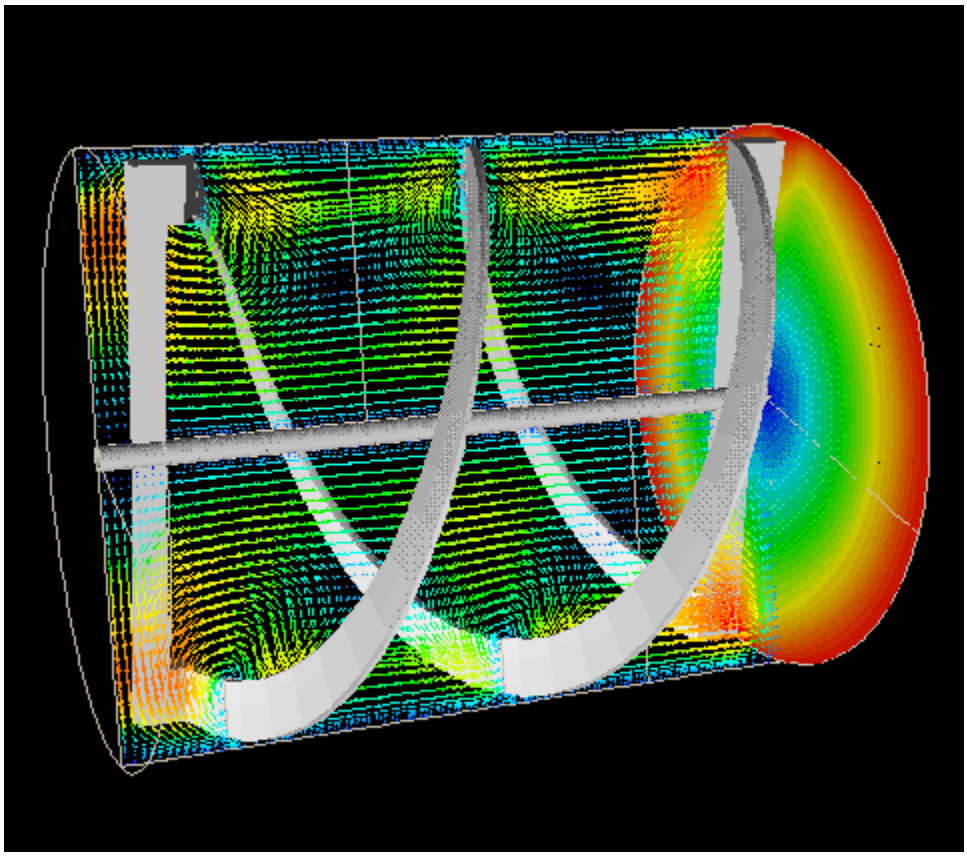




CFM FROM ANDRE BAKKER

http://www.bakker.org/cfm





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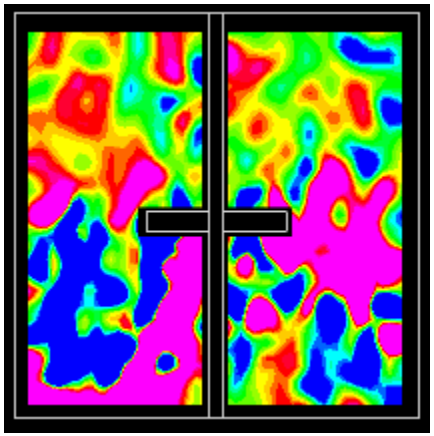
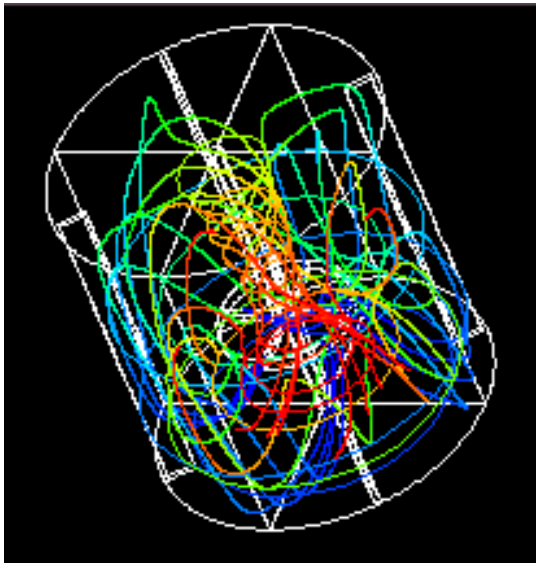
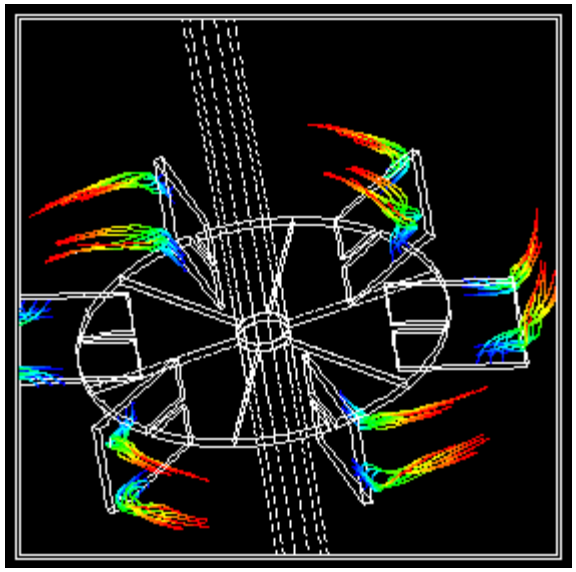






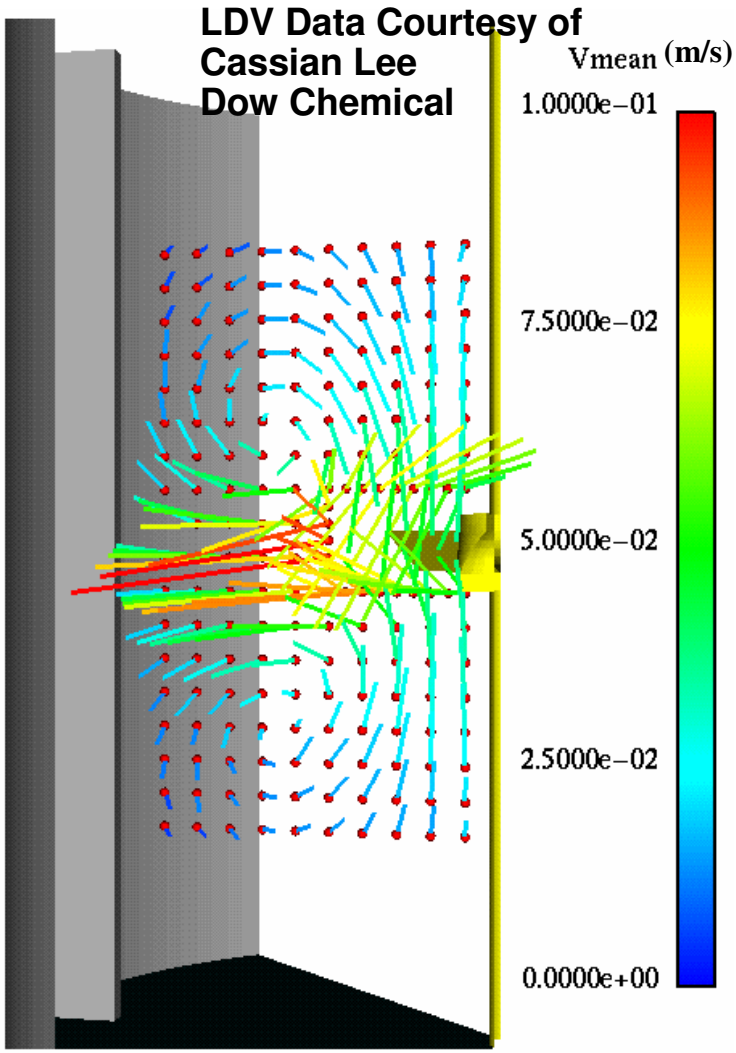
COMPUTATIONAL FLUID MECHANICS

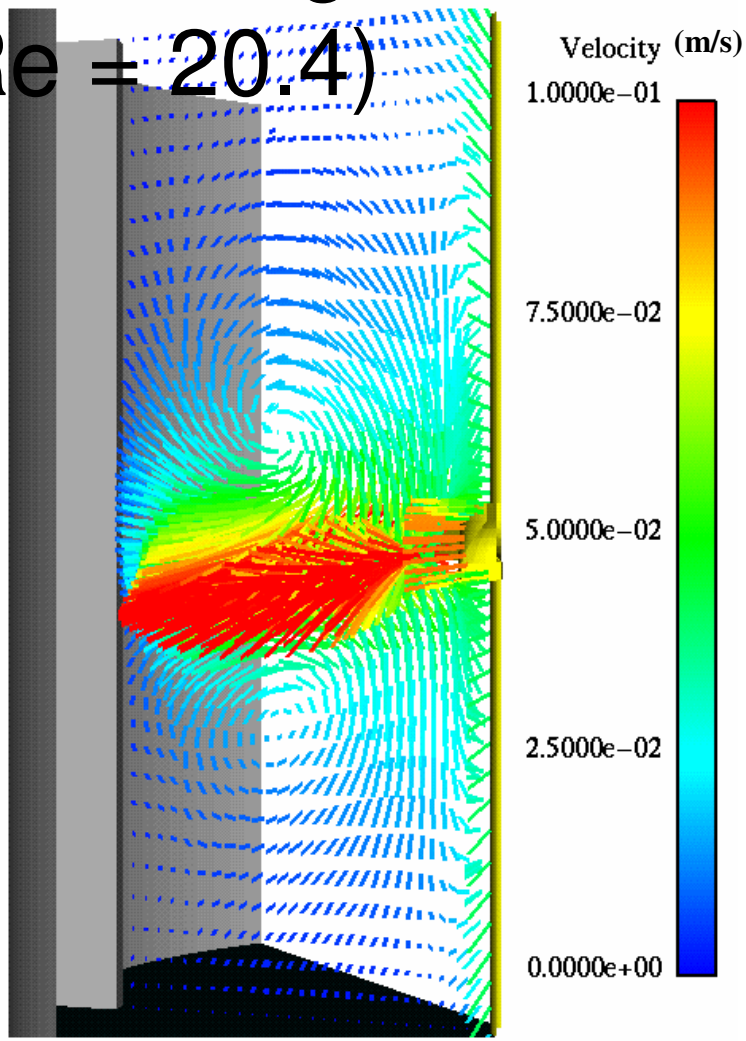
- MAJOR QUESTIONS
  - how to model turbulence
  - how to model impeller
  - how to handle multiphase
    - coupling equations
    - effect of dispersed phase on continuous

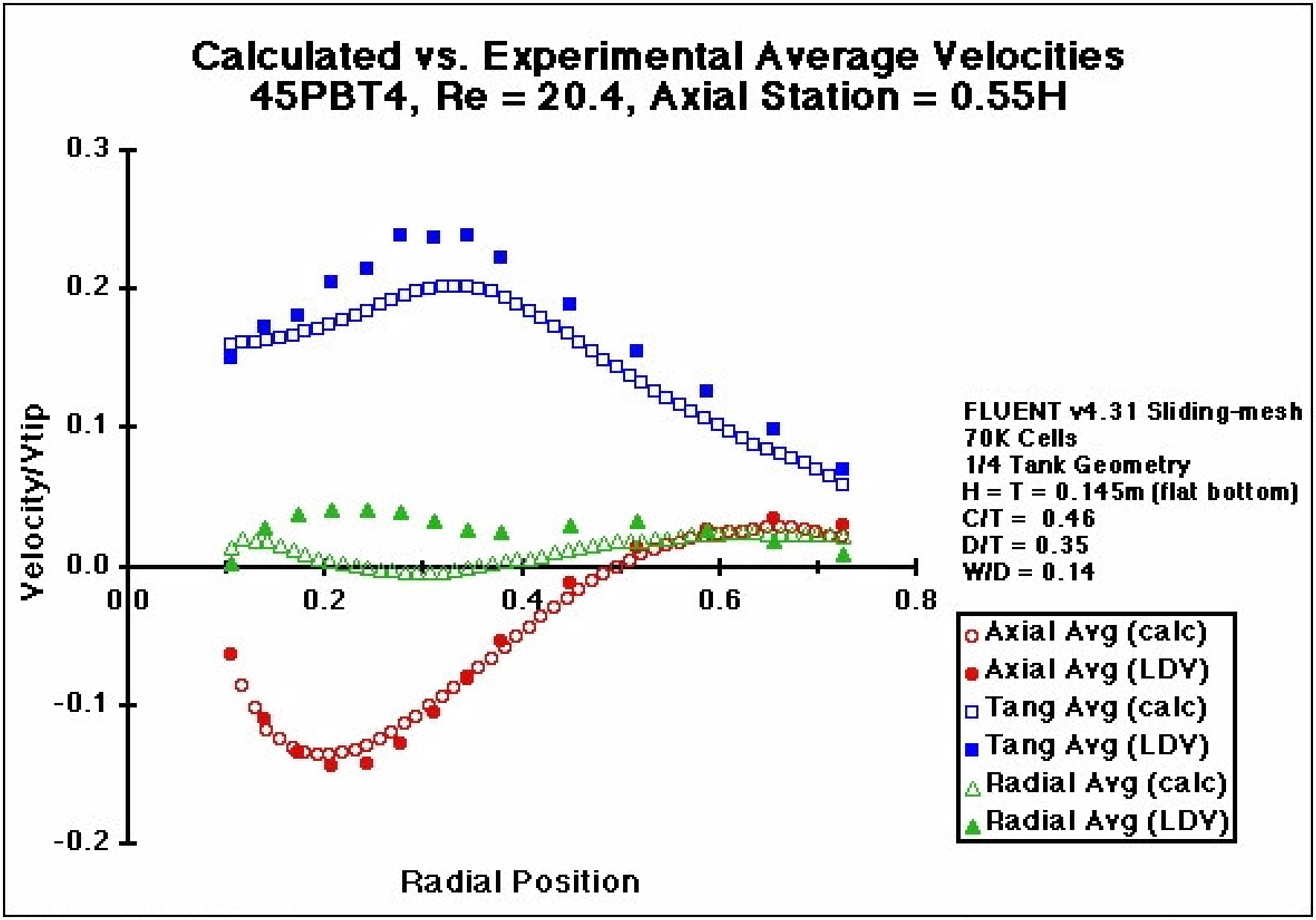
CFM FROM DICK LAROCHE

http://www.cpcfd.org/laroche/

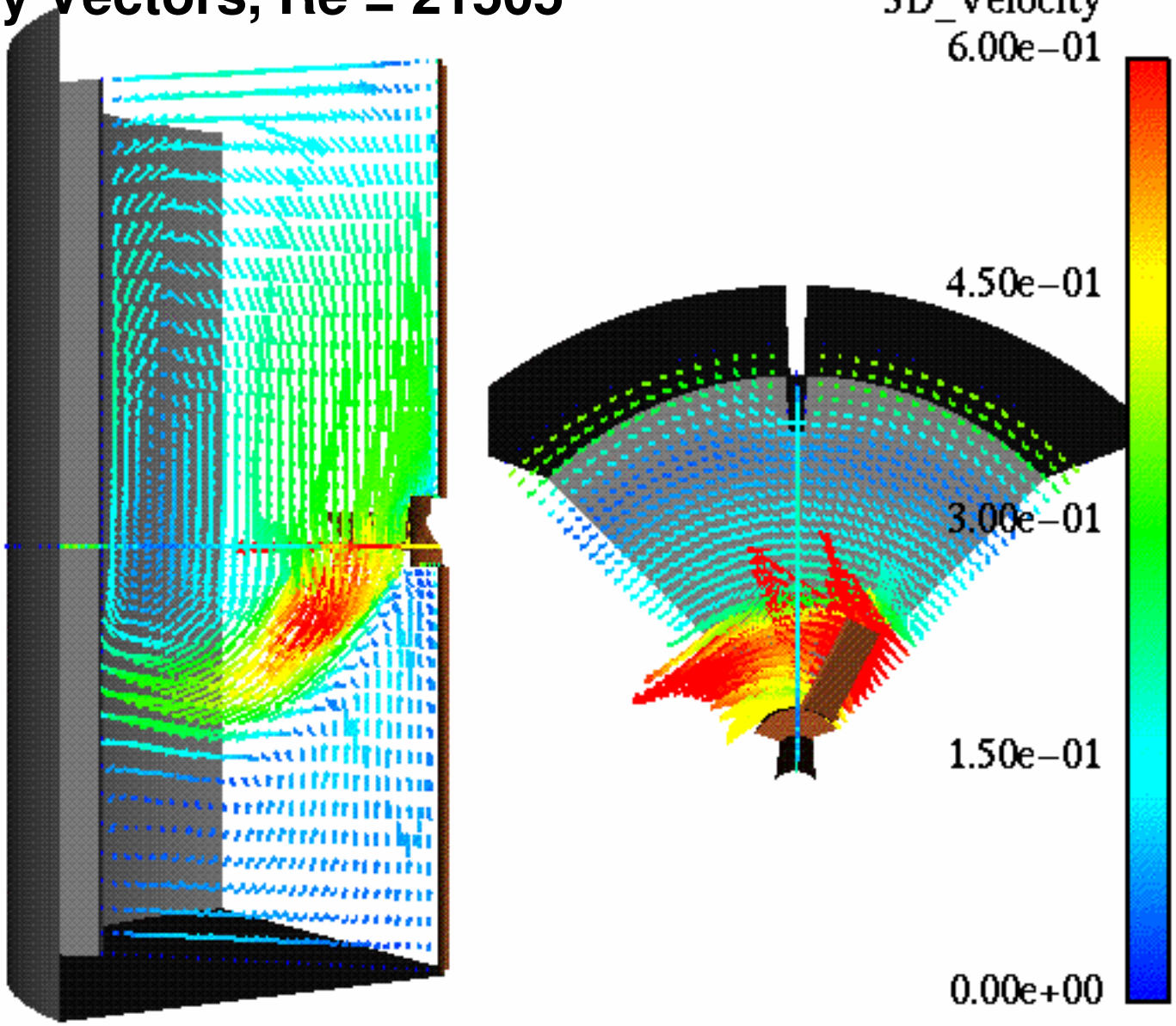
Experimental vs. FLUENT Sliding-Mesh (Re = 20.4)

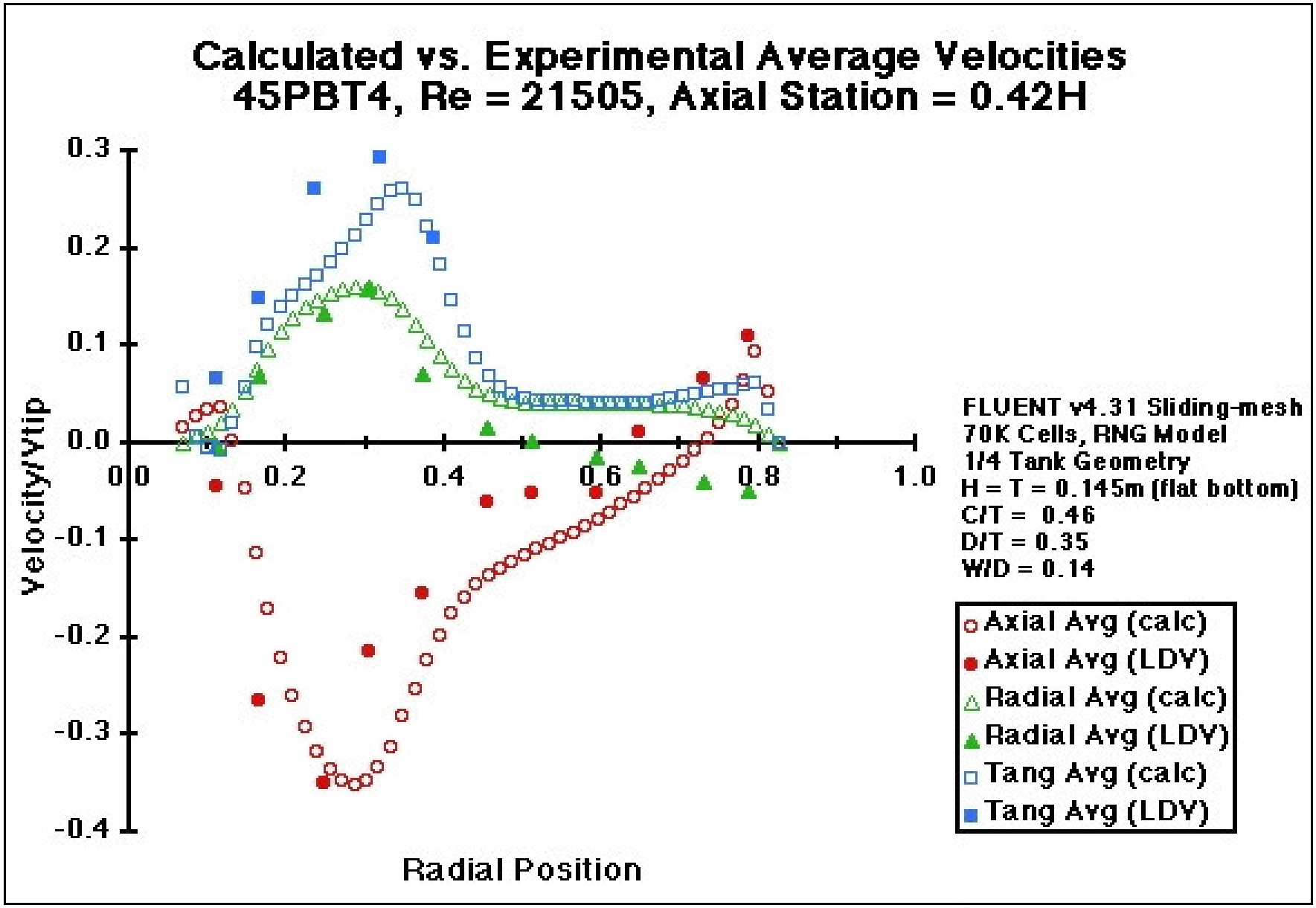





Calculated vs. Experimental Average Velocities
45PBT4, Re = 20.4, Axial Station = 0.55H
Velocity/Vtip
Radial Position
0.3
0.2
0.1
0.0
-0.1
-0.2
0.0
0.2
0.4
0.6
0.8
FLUENT v4.31 Sliding-mesh
70K Cells
1/4 Tank Geometry
H = T = 0.145m (flat bottom)
C/T = 0.46
D/T = 0.35
W/D = 0.14
Axial Avg (calc)
Axial Avg (LDV)
Tang Avg (calc)
Tang Avg (LDV)
Radial Avg (calc)
Radial Avg (LDV)

Velocity Vectors, Re = 21505

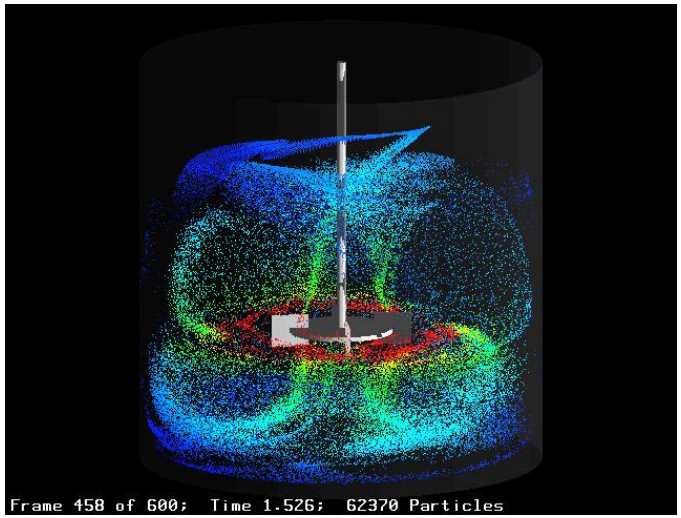



Calculated vs. Experimental Average Velocities
45PBT4, Re = 21505, Axial Station = 0.42H
Velocity/Vtip
Radial Position
0.3
0.2
0.1
0.0
-0.1
-0.2
-0.3
-0.4
0.0
0.2
0.4
0.6
0.8
1.0
FLUENT v4.31 Sliding-mesh
70K Cells, RNG Model
1/4 Tank Geometry
H = T = 0.145m (flat bottom)
C/T = 0.46
D/T = 0.35
W/D = 0.14
Axial Avg (calc)
Axial Avg (LDV)
Radial Avg (calc)
Radial Avg (LDV)
Tang Avg (calc)
Tang Avg (LDV)

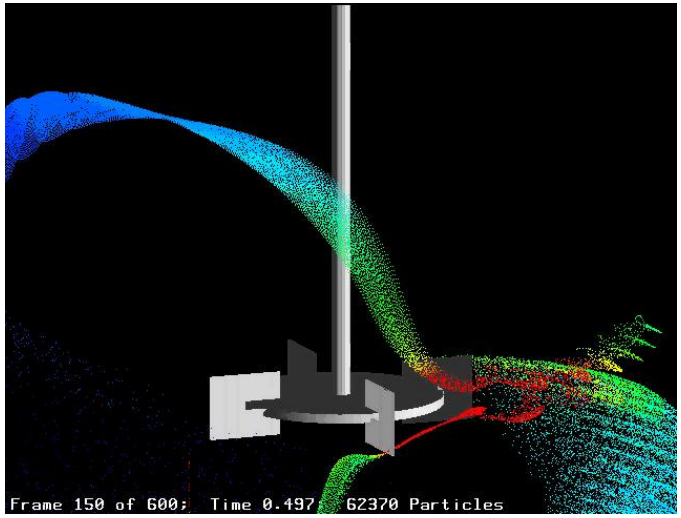
Lagrangian Particle Tracking for Laminar Flows

$$\frac{D\boldsymbol{x}}{Dt} = \boldsymbol{u}$$

x is the massless particle position
u is the fluid velocity vector

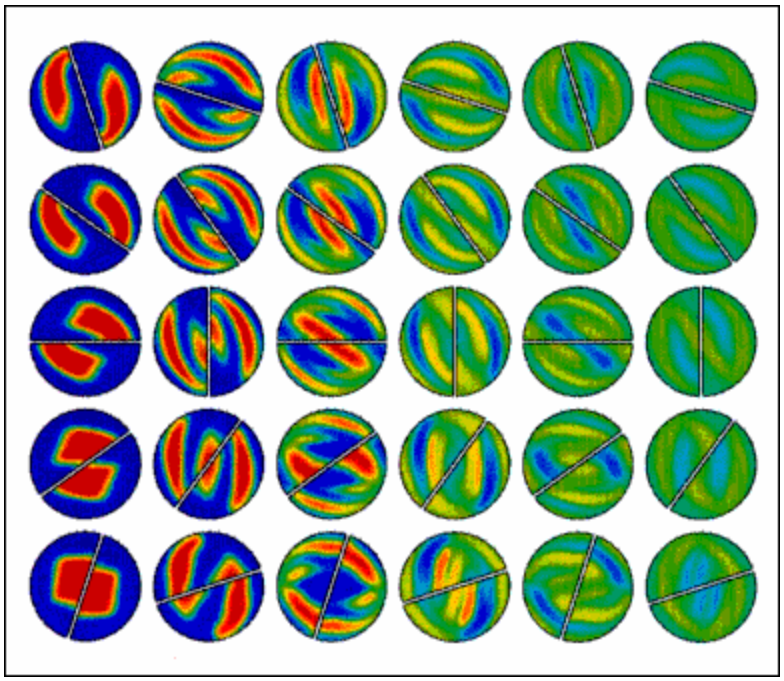


- Tracking 10K to 1000K "massless"particles
- Reveals flow structures
- Statistics for process dynamics



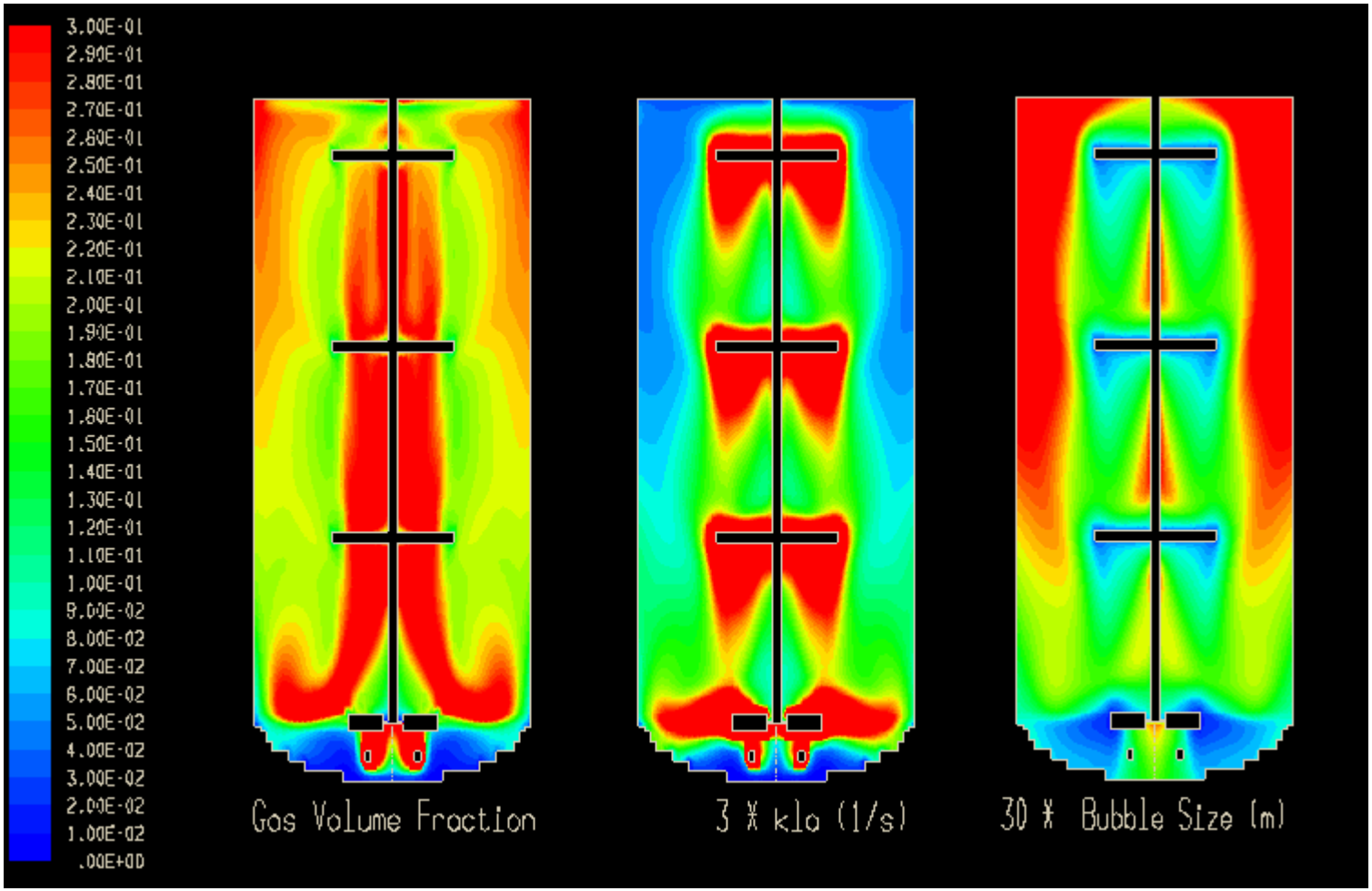
COMPUTATIONAL FLUID MECHANICS

- SOME SUCCESSFUL USES
    - stagnation in laminar flows - Tanguy
    - caverns around impellers
    - jet mixing - Forney
    - Draft tubes - Strand
- TOUGHER PROBLEMS
  - Free surfaces
  - high swirl
  - high non symmetry
  - multi phase



CFM FROM ANDRE BAKKER

http://www.bakker.org/cfm



SOLID SUSPENSION AND CFD

- Must use approximations
- Two fluid phase models
- Can not describe discrete phase
- Couple through drag laws
- May not handle turbulent effects of particles well
- Very useful qualitative.

SOLIDS SUSPENSION AND DISTRIBUTION

- Can not duplicate turbulence mechanism for solids re-suspension
- Can qualitatively and perhaps quantitatively describe distribution above Njs – see work of Bechtel SF on WTP
  - Some assumptions to handle conditions
  - GO TO ATTACHED PDF FOR EXAMPLES

ORGANIZATIONS

- NORTH AMERICAN MIXING FORUM NAMF
  - Industrialists and academics interested in mixing
  - self election
  - newsletter with announcements
  - conferences
    - leave a business card with Etchells
- Web site - www.Mixing.net/namf

ORGANIZATIONS

- BRITISH HYDROMECHANCIAL RESEARCH GROUP (BHRG)
- Research consortium
- about $30,000 per year per group
- Fluid Mixing Processes - FMP
- Tank and pipeline
- Contract Research
- David Brown - www.BHRGroup.co.uk

ORGANIZATIONS

- University of Maryland Rotor-Stator Consortium
- Prof. Rich Calabrese

CONFERENCES

- UK WORKING PARTY – Fluid Mixing 8 April 10-12 2006 King's College London www.fluidmixing8.org/

- AMERICAN INSITITUE OF CHEMICAL ENGINEERS - Nov. each year-
  - 2008 Philadelphia
  - NAMF sponsors about six sessions (9 mixing sessions)
- 13th EUROPEAN MIXING CONFERENCE
  - 2009 London
- NAMF BIANNUAL CONFERENCE
  - 2010 Victoria BC Canada
  - no proceedings –
  - Contact – http://mixing.net/namf/conferences/
- ISMIP – Industrial Mixing – Canada June 2008

NEW BOOK

- Handbook of Industrial Mixing
  - editors: Ed Paul, Suzanne Kresta, Victor Atiemo-Obeng
  - Wiley June 2003

  - with an all star cast

NOMENCLATURE

- A - area
- C – off bottom height
  - Sometimes concentration
- D – impeller or pipe diameter
- d or dp – particle diameter
- g – gravity acceleration
- H – liquid height
- N – impeller rotational speed
- P- power
- Q - flow
- S – density ratio
- T – tank diameter
  - Occasionally temperature
- U and V – velocity
- X – 100 times the weight ratio
- Z – distance traveled
- ρ – density
  - S – solid L - liquid
- μ - viscosity
- λ – eddy length
- σ – surface or interfacial tension
- Є – rate of energy dissipation per unit mass
  - Sometimes Pm
- Θ – mixing time
- Φ – volume fraction
- v – kinematic viscosity – viscosity divide by density

DIMENSIONLESS NUMBERS

- Reynolds – NRe or Re – $\rho^* D^2 * N/\mu$
- Power – Po or Npo – $P / (\rho * N^3 * D^5)$
- Fourier – Fo - $\mu * \theta/\rho/T^2$
- Weber Nwe or We -
- Froude – Fr – $N^2 * D/g$
- Flow – Nq or NQ or Fl – $Q/(N * D^3)$

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U.S. DEPARTMENT OF
ENERGY

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352
1-888-375-PNNL (7665)
www.pnl.gov

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PNNL-18751

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under Contract DE-AC05-76RL01830

Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Volume III

GL Smith
AP Poloski
MW Rinker
RL Demmer
AW Etchells, III
BE Lewis Jr.
SL Marra

July 2009

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PNNL-18751

Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

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Prepared for
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Pacific Northwest National Laboratory
Richland, Washington 99352

(1) Idaho National Laboratory, Idaho Falls, Idaho
(2) AWE3 Enterprises, Philadelphia, Pennsylvania
(3) Oak Ridge National Laboratory, Oak Ridge, Tennessee
(4) Savannah River National Laboratory, Aiken, South Carolina

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Appendix A.3

Site Presentations

Hanford Sludge Characterization

Adam Poloski

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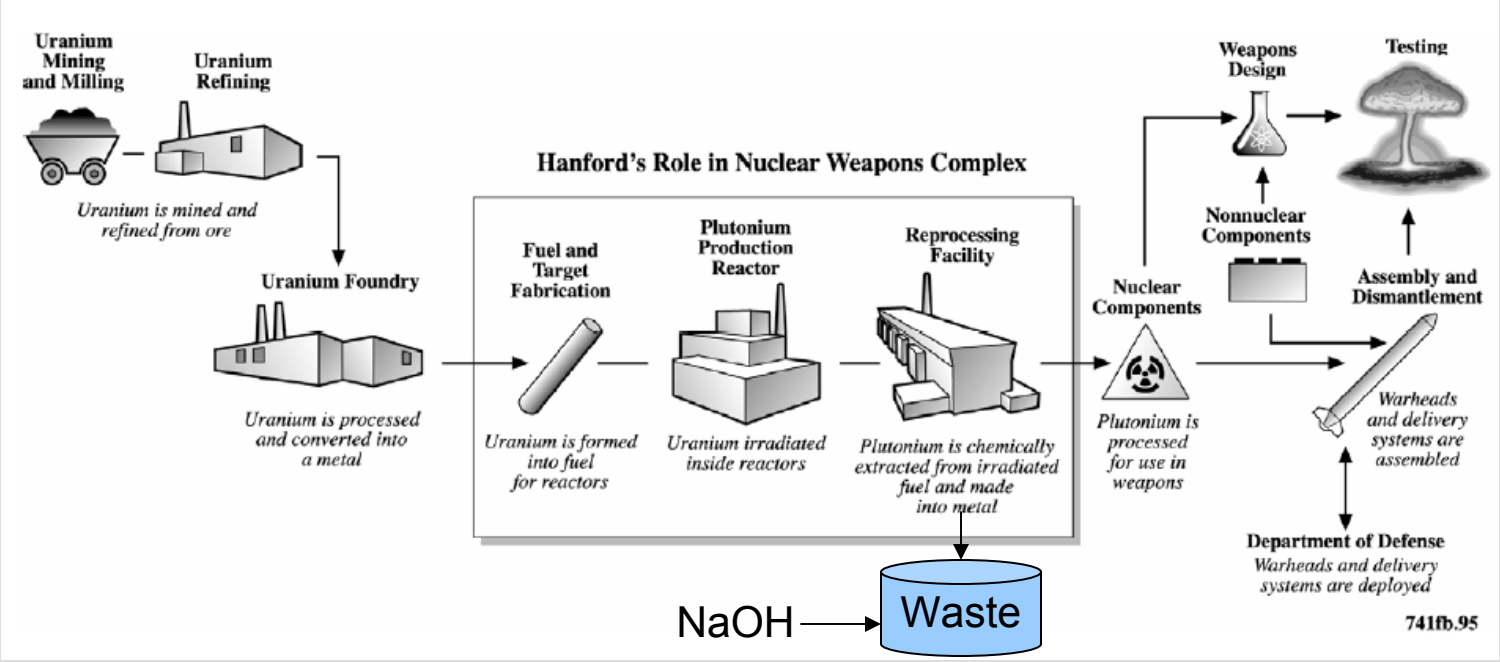
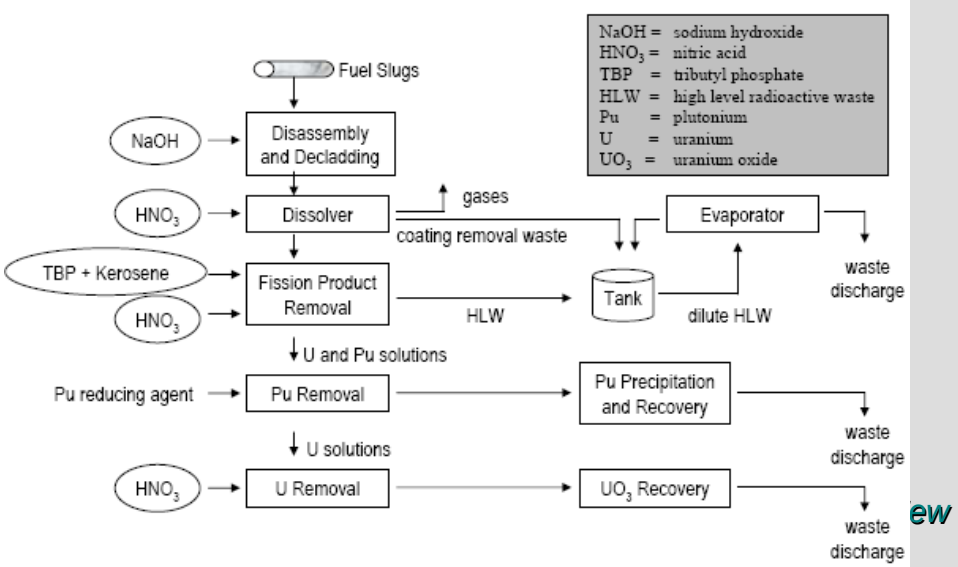
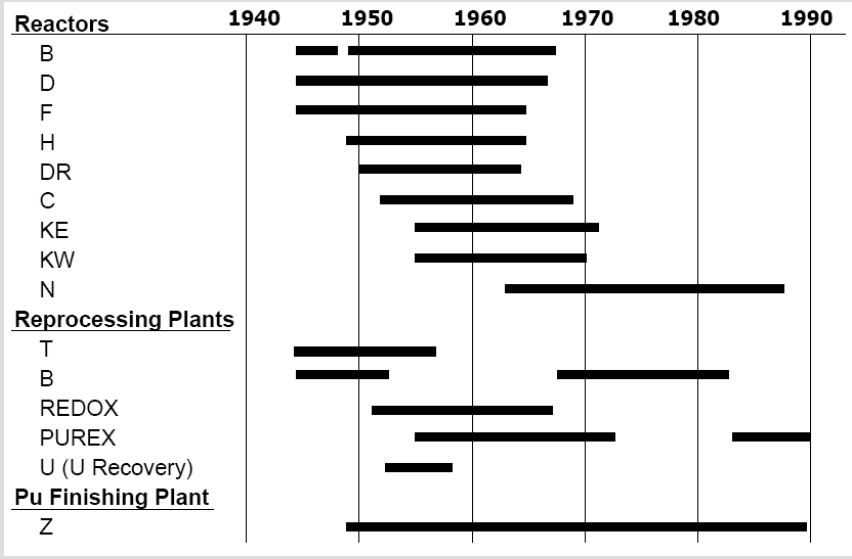
Hanford Waste Generation Processes







ew

Waste Types

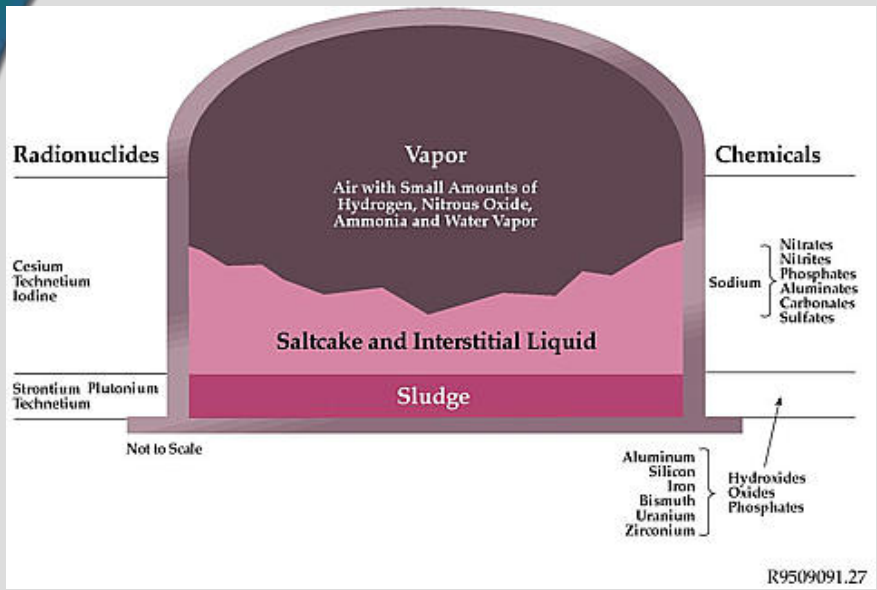
- Several waste types produced due to combinations of varying reactor fuels and reprocessing plant technologies
- Several in-tank treatment processes lead to further waste types
- FeCN process
- CEM, DE

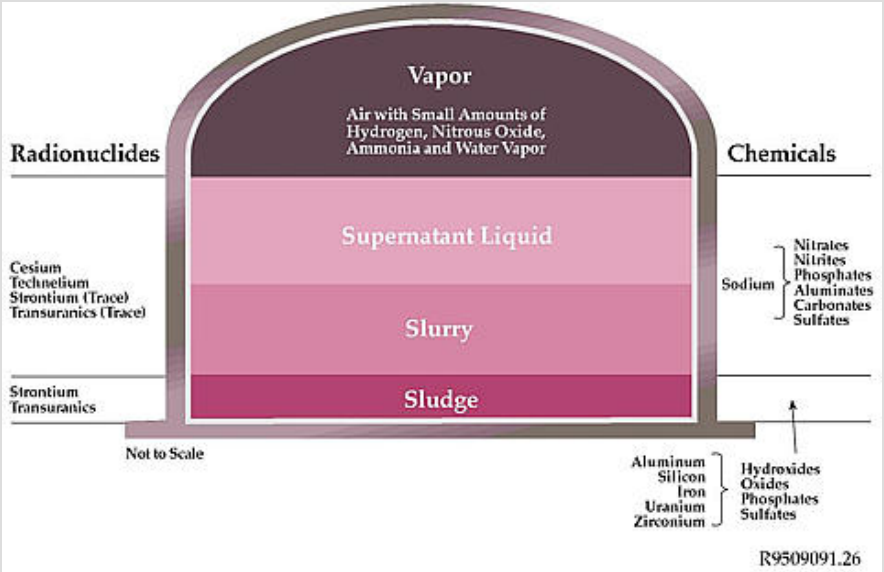
Table 3.1. List of Waste Type Definitions

Waste Type	Definition
1C	$BiPO_4$ first cycle decontamination waste (1944-1956)
1CFeCN	Ferrocyanide sludge from in-farm scavenging of 1C supernatants in TY-Farm (1955-1958)
224	lanthanum fluoride process "224 Building" waste (1952-1956)
2C	$BiPO_4$ second cycle decontamination waste (1944-1956)
A1-SltCk	Saltcake from the first 242-A Evaporator campaign (1977-1980)
A2-SltSlr	saltcake from the second 242-A Evaporator campaign (1981-1994).
AR	Washed Plutonium-Uranium Extraction (PUREX) sludge (1967-1976)
B	high-level acid waste from PUREX processed at B Plant for Sr recovery (1967-1972)
BL	low-level waste from B Plant Sr and Cs recovery operations (1967-1976)
CEM	Portland Cement
CSR	Cesium recovery, supernatant from which Cs has been removed
CWP	PUREX cladding waste (1956-1960 and 1961-1972)
CWR	REDOX cladding waste, aluminum clad fuel (1952-1960 and 1961-1972)
CWZr	zirconium cladding waste (PUREX and REDOX)
DE	diatomaceous earth
HS	hot semi-works ^{90}Sr recovery waste (1962-1967)
MW	$BiPO_4$ process metal waste (1944-1956)
OWW3	PUREX organic wash waste (1968-1972)
P1	PUREX HLW (1956-1962)
P2	PUREX HLW (1963-1967)
P3	PUREX HLW (1983-1988)
PFeCN	Ferrocyanide sludge from in-plant scavenged supernatant (1954-1958)
PL2	PUREX low-level waste (1983-1988)
R (boiling)	boiling REDOX HLW (1952-1966)
R (non-boiling)	non-boiling REDOX HLW (1952-1966)
R-SltCk	Saltcake from self-concentration in S- and SX-Farms (1952-1966)
S1-SltCk	Saltcake from the first 242-S Evaporator campaign using 241-S-102 feed tank (1973-1976)
S2-SltSlr	Saltcake from the second 242-S Evaporator campaign using 241-S-102 feed tank (1976-1980)
SRR	HLW transfers (late B Plant operations)
T2-SltCk	Saltcake from the second 242-T Evaporator campaign using 241-TX-118 feed tank (1965-1976)
TBP	tributyl phosphate waste (from solvent based uranium recovery operations)
TFeCN	ferrocyanide sludge produced by in-tank or in-farm scavenging
TH	PUREX waste from processing of thoria targets
Z	Z Plant waste

Battelle

Hanford Tank Configurations





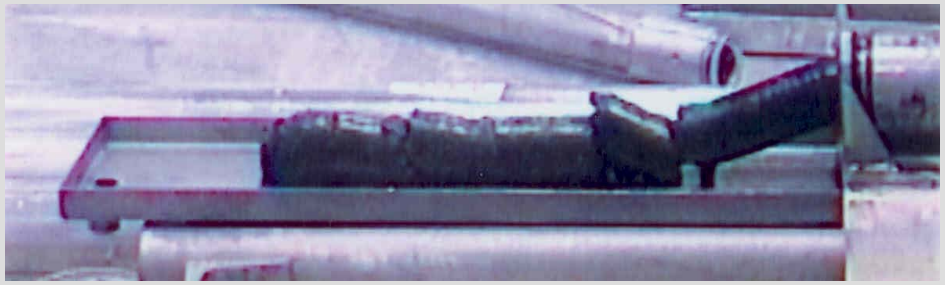
- 177 tanks
  - Single shell (top) and double shell (bottom)
- 53 million gallons of waste
  - 25,000 MT solids
- Composition of sludge contained in 149 single shell tanks
  - 37 Mgal total waste volume; 13 Mgal sludge; 23 Mgal saltcake
  - 17,000 MT solids
- Multiphase suspensions
  - Solid, Liquid, Gas
- nearly every element on Periodic table present
- Heterogeneous: radially and axially
  - Mixing conditions
  - Temperature gradients

A.3.4

Hanford Tank Waste Sampling and Characterization

Estimated Shear Strength: 2,000-4,000 Pa

Hanford Tank B-203, Core 122, Segment 10



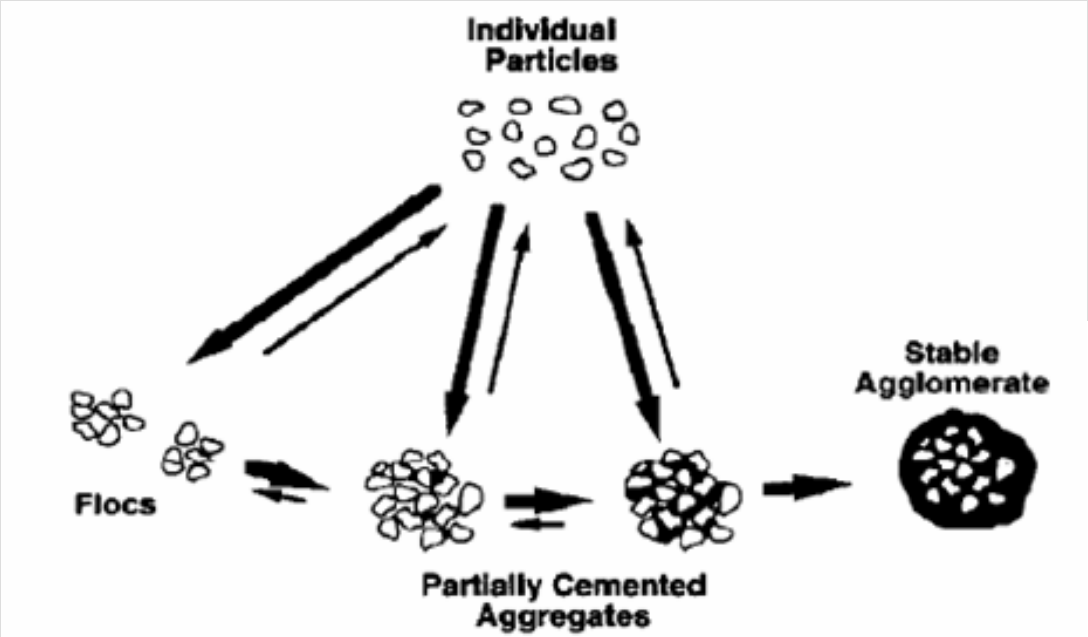
Hanford Tank B-204 Core 114, Segment 8

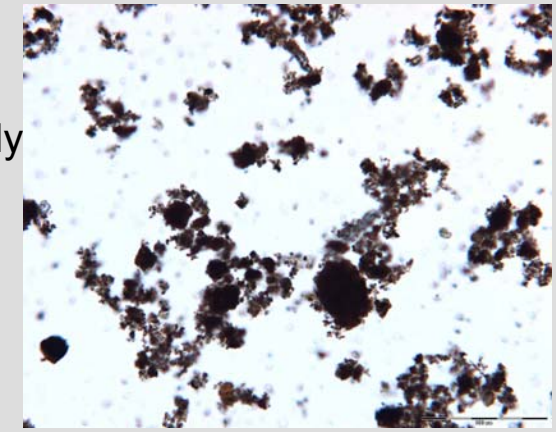
A.3.5

- Over 20-30 year period, samples from the tanks have been taken and characterized
  - Chemical analysis
    - Anion, cation, and organics
  - Rheological analysis
    - Flow curves
    - Shear strength
  - Physical Properties
    - Sedimentation rates
    - Apparent density
    - Fraction dissolved and undissolved solids
- Key questions:
  - What are the size and density of the sludge particles?
  - How fast does the supernatant layer develop?
  - What is the viscosity of the supernatant layer?
  - What are the flow behaviors of the sludge layer?

How big and dense are the sludge particles? Particle Types

- Individual Particles
  - Crystals
- Flocs
  - Primary particles bound by electrostatic forces
  - Multi-component
  - Low density
  - Easily broken down by moderate shear forces
- Partially Cemented Aggregates
  - Coagulated aggregates
  - Can be chemically bound together
  - Fractal Density
- Stable Agglomerates
  - Agglomerates that eventually become monolithic
  - High density





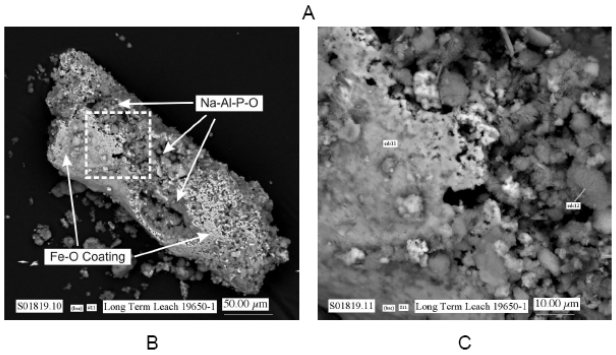


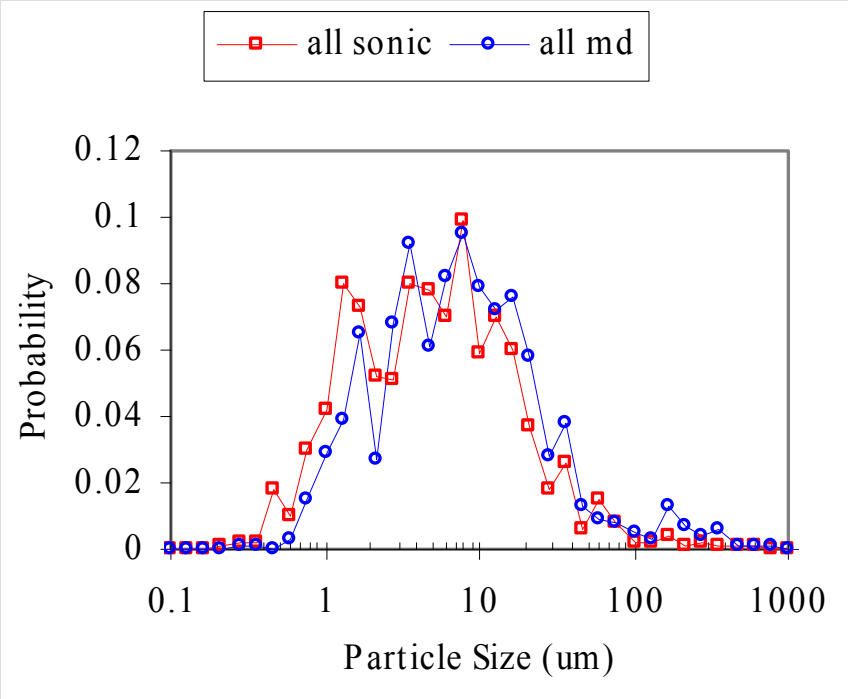
FIGURE S-8. Backscattered-electron SEM micrographs of leached C-204 sludge samples showing preferential dissolution features in an Fe oxide aggregate (micrograph A) and Fe-oxide coating (micrographs B and C). [Micrograph C shows at higher magnification the region marked by the dashed-line square in micrograph B.]

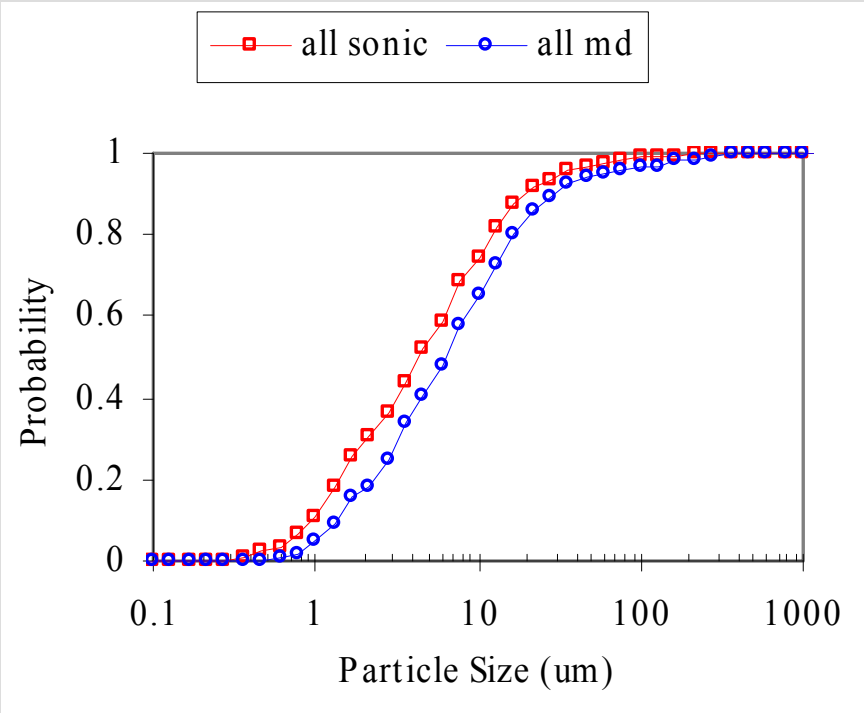
What are the size and density of the sludge particles?

Particle Size Distributions

- "Minimal disturbance" (md) PSD
  - Data collected at minimum turbulence/disturbance conditions
  - Crystals, flocs, cemented aggregates, stable agglomerates
- "Sonicated" (sonic) PSD
  - Data collected during ultrasonic treatment of the sample
  - Crystals, cemented aggregates, stable agglomerates

Particle Size Distributions





Quantile	1%	5%	25%	50%	75%	95%	99%	100%
Sonicated[a]	0.39	0.70	1.63	4.39	10.1	33.4	112	774
Minimal Disturbance[b]	0.65	1.00	2.80	6.31	14.0	58.6	256	1000

(a) Combined data from 18 sludge tanks.
(b) Combined data from 19 sludge tanks.

A.3.8

Degree of Particle Size Characterization

- Several PSD data sets discarded due to instrument limitations
- Several types remain uncharacterized
  - TBP
  - R (non-boiling)
  - DE

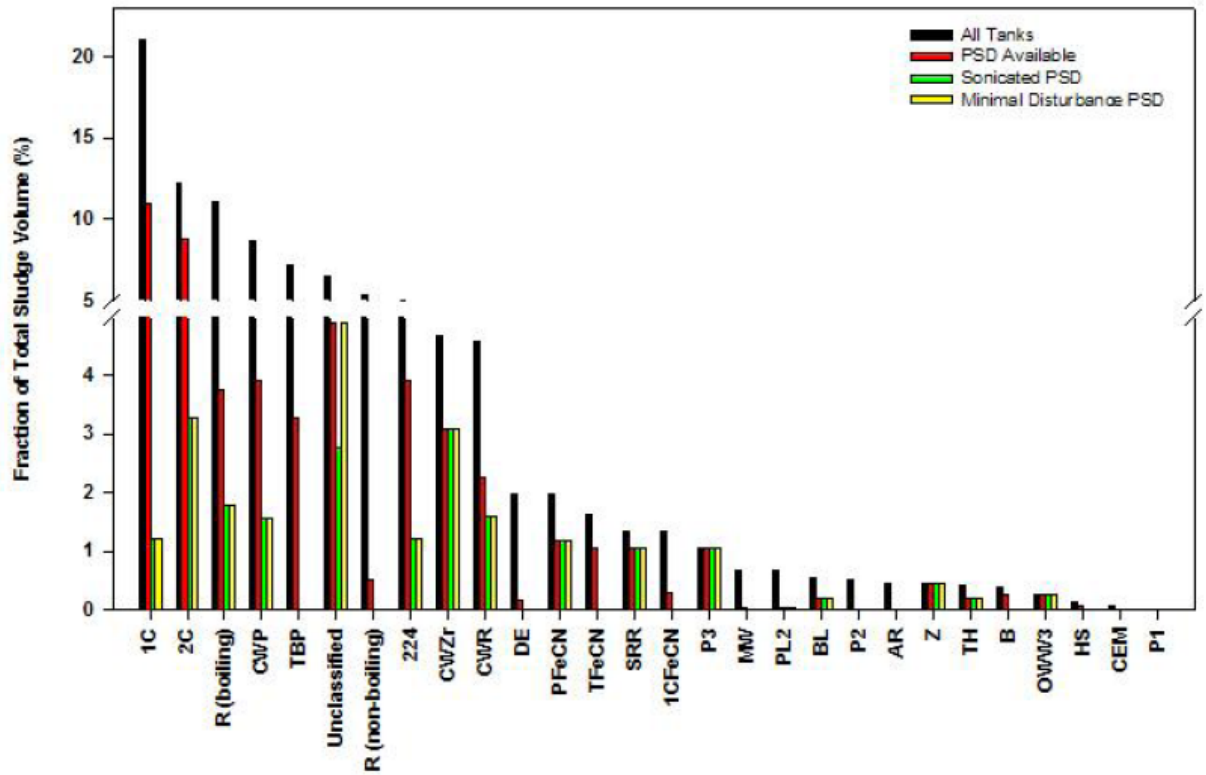
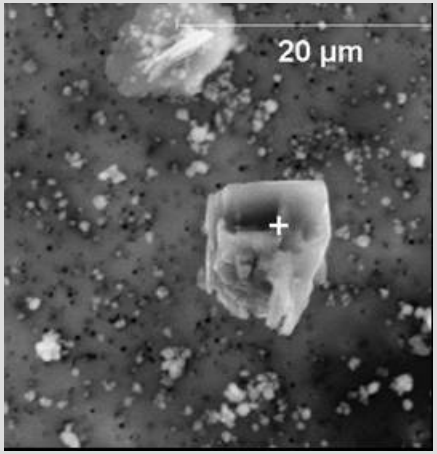


Figure 3.1.1. Volumetric Significance of Composite PSD Analysis. Here, the volume fraction of total sludge solids by waste types (black bars) is compared to the volume fraction with PSD measurements available (red bars), regardless of quality, and the volume fraction down selected for sonicated and minimal disturbance data sets (green and yellow, respectively).

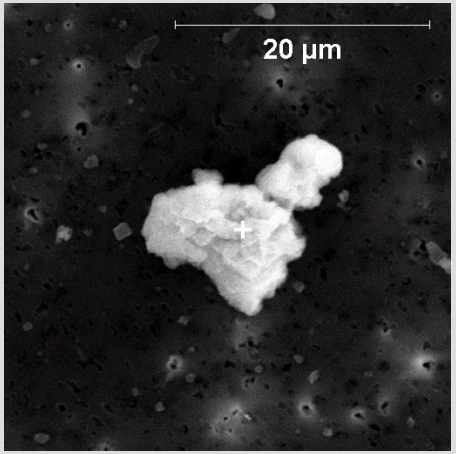
What are the size and density of the sludge particles?

Scanning Electron Micrographs

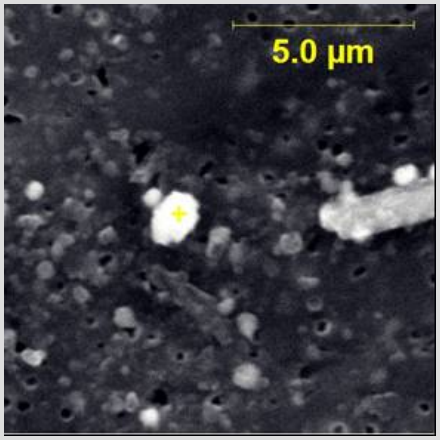
- Maximum Particle and Agglomeration Size
  - Image analysis from SEM and XRD
  - Expert panel elicitation



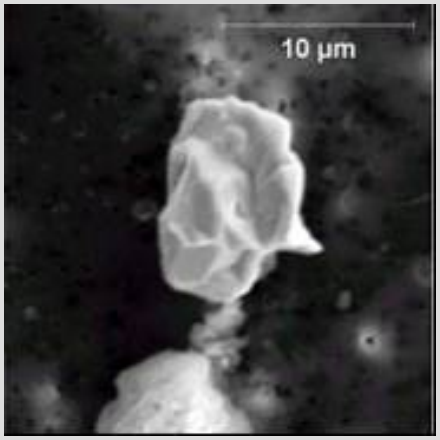
Gibbsite, C-104



$Na_2U_2O_7$, SY-102



Fe_2O_3, AY-102



PuO_2, SY-102

What are the size and density of the sludge particles?

Saltcake species

- The following slides visually show the presence of large (100-500 μm) particles in Hanford saltcake
- Not considered in PSDD assessment
- These species may reprecipitate in the WTP
  - Broad PSD
  - Broad range of particle shapes
  - Broad range of particle densities

500 microns

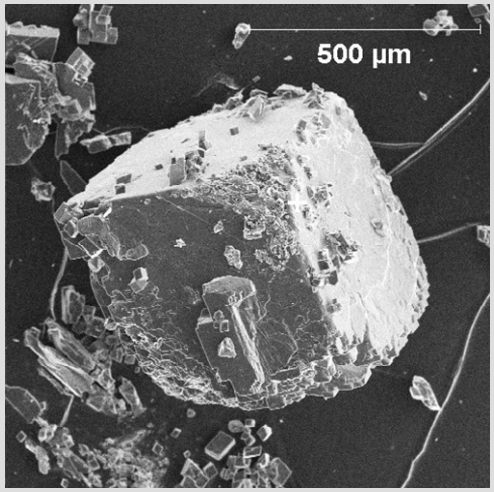


Figure 4.1.2-1. High magnification secondary electron image of original solids from Tank S-112.

500 microns

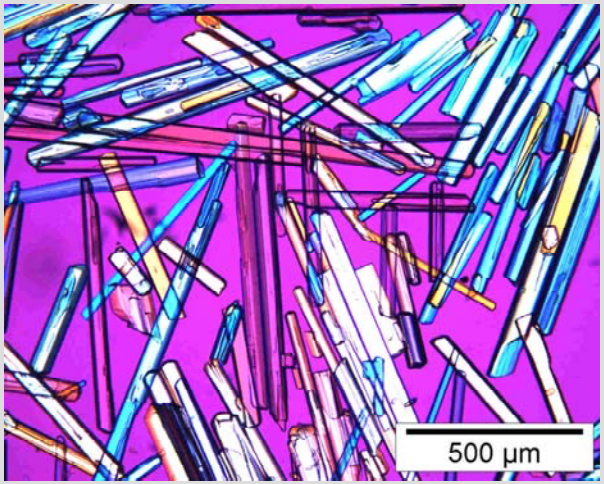


Figure 4.4.2-1. $Na_3PO_4 \cdot 12H_2O$, Tank U-107.

Crystals formed by dissolving U-107 saltcake at 50 °C, then cooling the solution to 23 °C; some of the crystals are thick enough to display first-order interference colors. (See also Figure 3.1.3-3.)

Crossed polars with Red I compensator, 4x objective.

100 microns

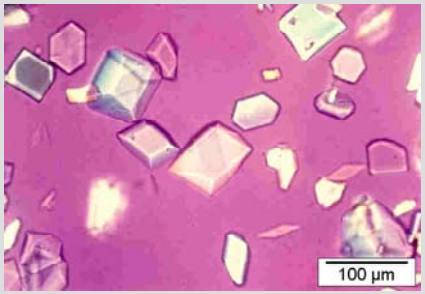


Figure 4.4.2-1. $Na_3PO_4 \cdot 6H_2O$ in tank waste simulant.

Lower hydrate crystal habit, which forms at higher temperatures, is much more compact, and does not form gels or plug pipes the way the 12-hydrate does. The transition temperature, typically ~40-50 °C, depends on ionic strength of solution and other factors.

Crossed polars with Red I compensator, 16x objective.

50 microns

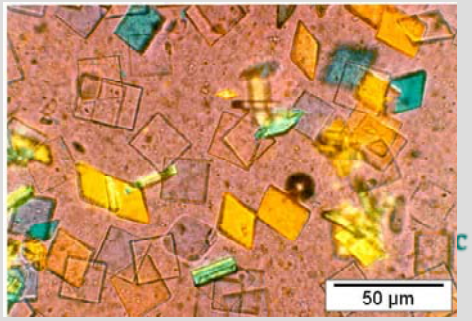


Figure 4.5.1-1. $NaAl(OH)_4$ from a previous simulant, showing the "thin quadratic platelet" form of the crystals (Fricke and Jucaitis 1930). Platelets that lie flat appear to be isotropic because the optic axis is parallel to the light path, but plates tilted slightly appear blue/yellow, revealing the birefringence.

Simplified List of Solid-Phase Compounds

Solid-Phase Compound	Crystal Density (g/mL)	Volume Fraction	Maximum Observed Particle Size (μm)
$Al(OH)_3$, Gibbsite	2.42	0.515	20
$(NaAlSiO_4)_6 \bullet (NaNO_3)_{1.6} \bullet 2H_2O$	2.365	0.166	8
AlOOH, Boehmite	3.01	0.106	0.05
$NaAlCO_3(OH)_2$	2.42	0.095	4.2
Fe_2O_3	5.24	0.041	1.6
$Ca_5OH(PO_4)_3$	3.14	0.020	0.1
$Na_2U_2O_7$	5.617	0.016	15
ZrO_2	5.68	0.011	50
Bi_2O_3	8.9	0.0081	10
SiO_2	2.6	0.0069	100
$Ni(OH)_2$	4.1	0.0055	0.5
MnO_2	5.026	0.0054	10
CaF_2	3.18	0.0023	15
$LaPO_4 \bullet 2H_2O$	6.51	0.0013	3
Ag_2CO_3	6.077	0.000094	4
PuO_2	11.43	0.000013	40

What are the size and density of the sludge particles?

PSDD Modeling

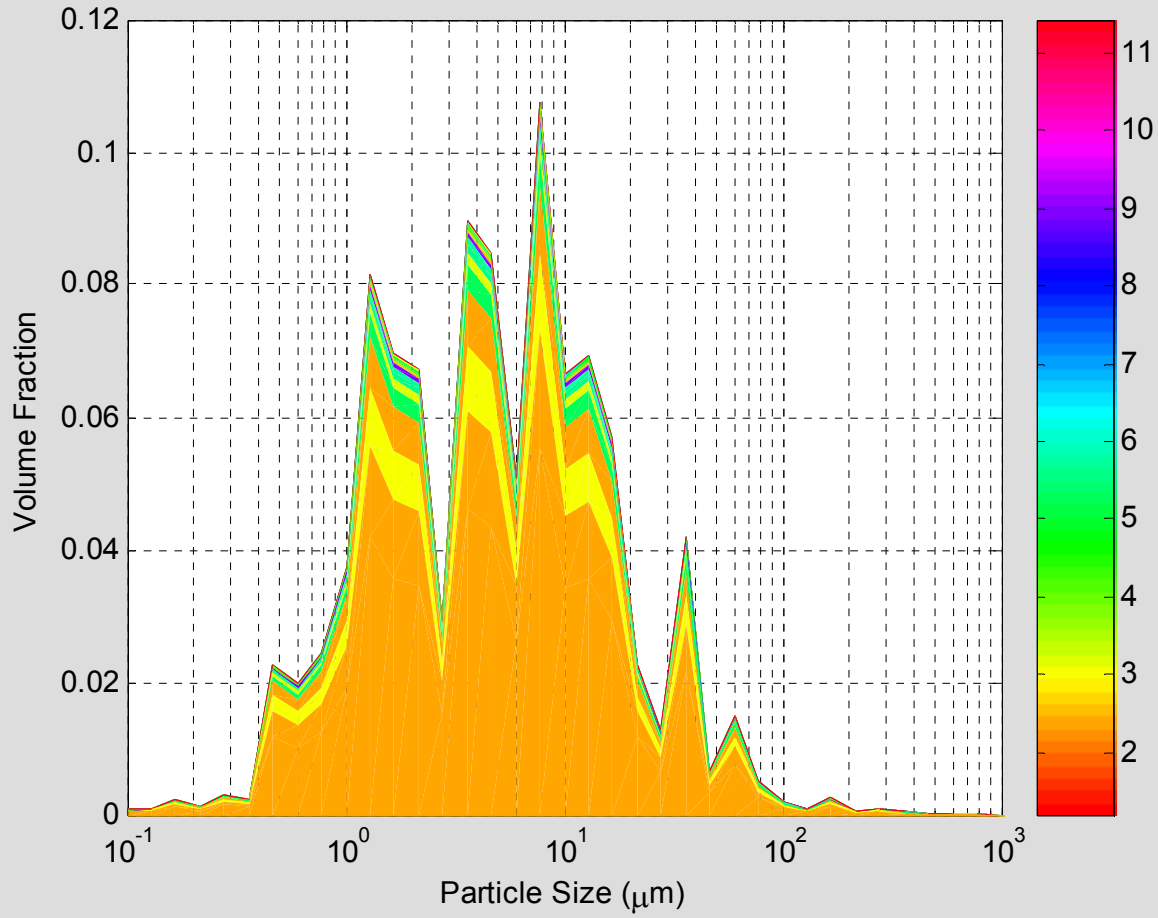
- Monte Carlo simulation approach used.
  - Input parameters:
    - PSD (repeated realizations of composite PSDs)
    - Crystal density
    - Volume fraction
    - Primary particle size (affects Cases 2 and 4)
    - Fractal Dimension
- Representative PSDD is calculated at centroid for respective input distributions.
- Composite PSD must be reproduced and particulate mass must be conserved.

1. Sonicated PSD
   - Crystals, cemented aggregates, and stable agglomerates are assigned crystal density
2. Sonicated PSD
   - Crystals are assigned crystal density
   - Density of cemented aggregates and stable agglomerates assigned via fractal relation
3. Minimal disturbance PSD
   - Crystals, flocs, cemented aggregates, and stable agglomerates are assigned crystal density
4. Minimal disturbance PSD
   - Crystals are assigned crystal density
   - Density of flocs, cemented aggregates, and stable agglomerates assigned via fractal relation

What are the size and density of the sludge particles?

Monte Carlo Modeling Results

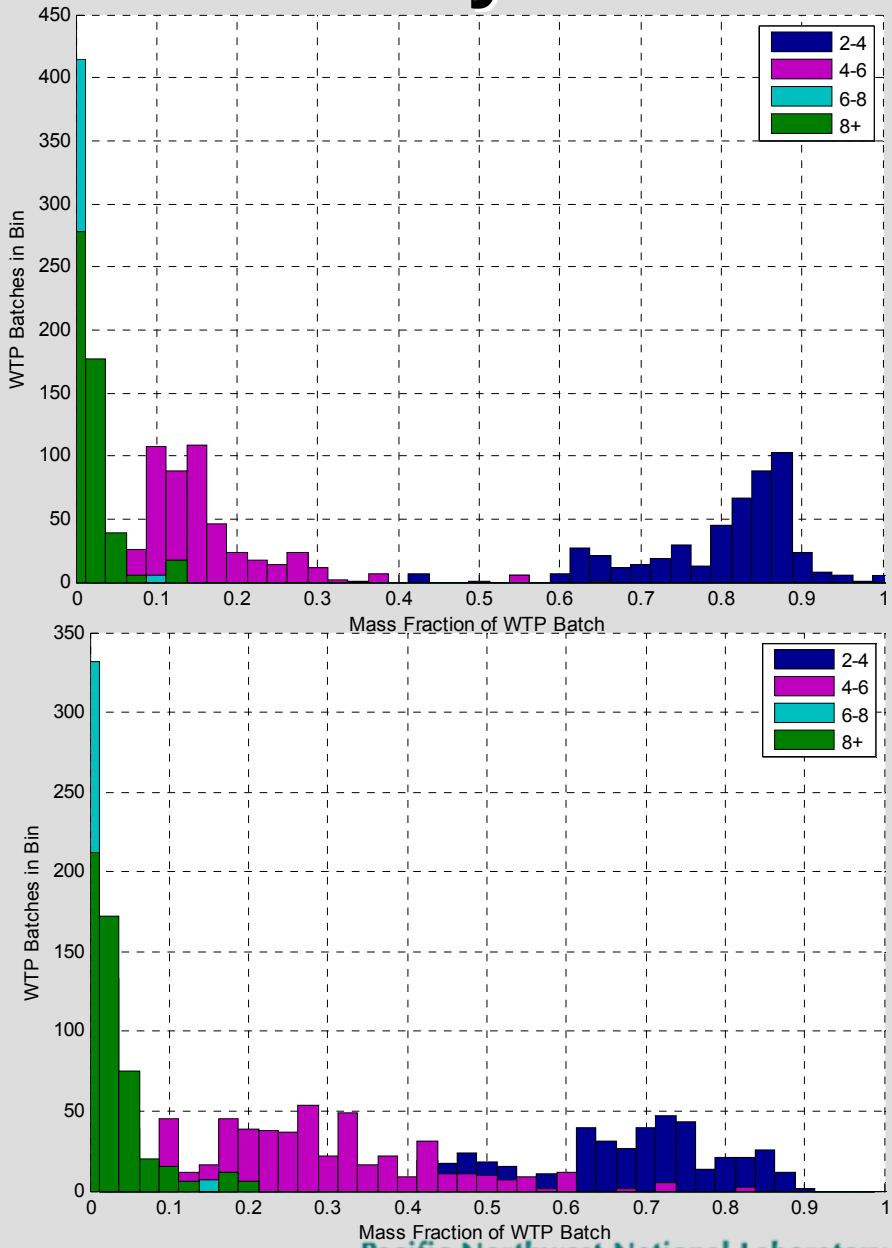
- Feasible solutions to this problem were calculated for each realization while maintaining mass balance and particle size distribution
- Particle size & density relationships for each simulation case were modeled
- Results can be applied to slurry transport and mixing designs
- Example for Case 1 shown on left (colorbar axis is particle specific gravity)
- Particles range from 1 to 100 microns over a density range of 2.5 to 11 g/cc
  - Dilute conditions → settling slurry
  - Concentrated conditions → discrete particles settling in a non-settling matrix



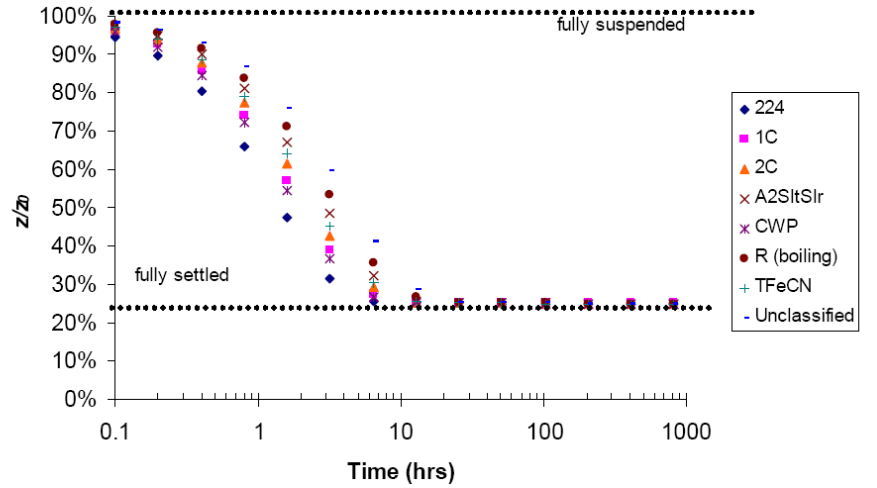
What are the size and density of the sludge particles?

Waste Feed Variability

- 518 WTP batches over 20-30 year processing period
- Colors are groupings of particle density
- Pre-leaching (top)
- Post-leaching approximation (Gibbsite and Boehmite removed; bottom)
- High amount of particles with density of 2-4 g/cc
- 30-80 wt% of slurry contains particles with 4-6 g/cc
- 10-20 wt% of slurry contains particles with 8-10 g/cc
- Particle size and rheological properties will also significantly vary over time
  - Avoid designing to averages and design to anticipated but extreme conditions



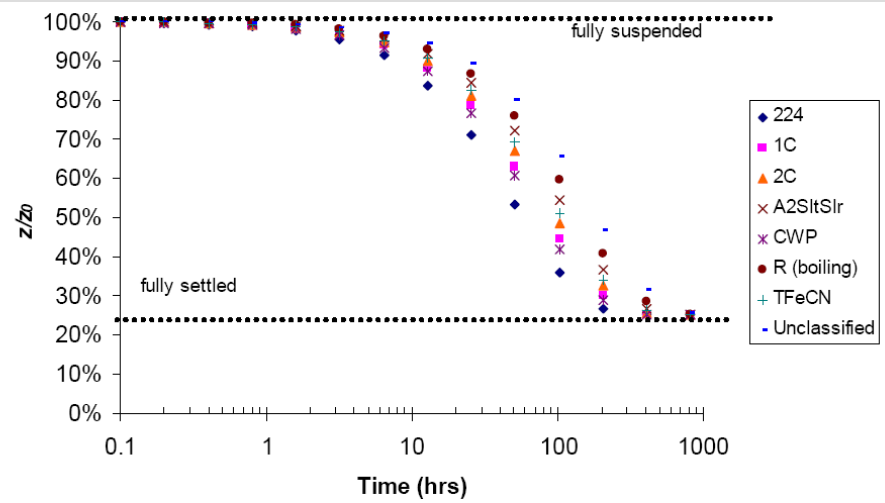
Effect of Process Scale on Sedimentation Time



a) initial interface height is $z_0 = 10$ cm; initial solids concentration of the suspended sample is $\phi = \frac{\phi_{max}}{4}$

- Compressive settling regime observed
- Sedimentation completed within 10 hours at small scale

- Sedimentation completed within 1,000 hours at medium scale

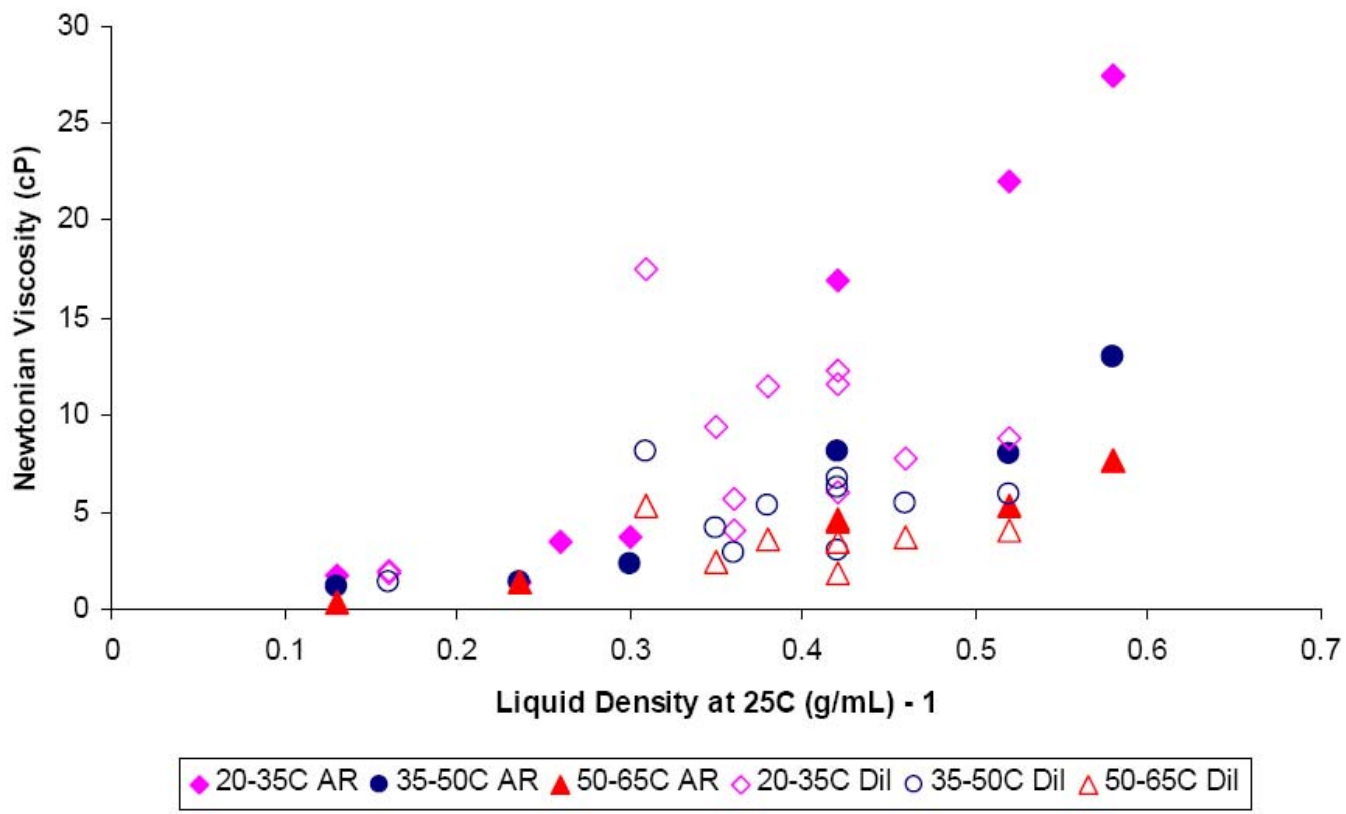


b) initial interface height is $z_0 = 4$ m; initial solids concentration of the suspended sample is $\phi = \frac{\phi_{max}}{4}$

How fast does the supernatant layer develop?

Supernate Viscosity Data

- as-received (AR)
- diluted with Hanford process water (Dil)
- Viscosity ranges from 2-3 cP to 20-30 cP depending on dissolved solids concentration and temperature



(a) Data grouped according to dilution and temperature.

A.3.17

What is the viscosity of the supernatant layer?

Shear Strength Model

- Nomenclature to differentiate between direct measurement techniques and extrapolation from a flow curve
  - static measurements termed "shear strength"
  - Dynamic extrapolations from a flow curve termed "yield stress"
- Thixotropic (time dependent) property
  - Rebuilding of gel structure with time
- Measured with shear vane technique
- Data from AZ-101 pretreated HLW sludge
- Behaves similar to mineral suspensions where full shear strength reached in 24-48 hours while quiescent
- Only data available on restructuring of Hanford HLW sludge

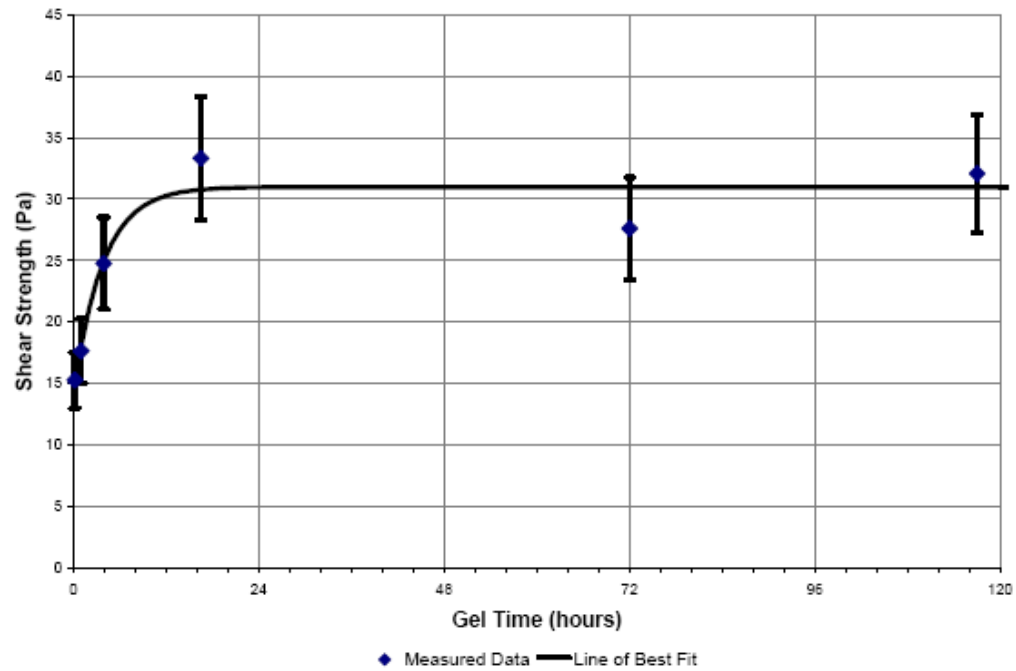


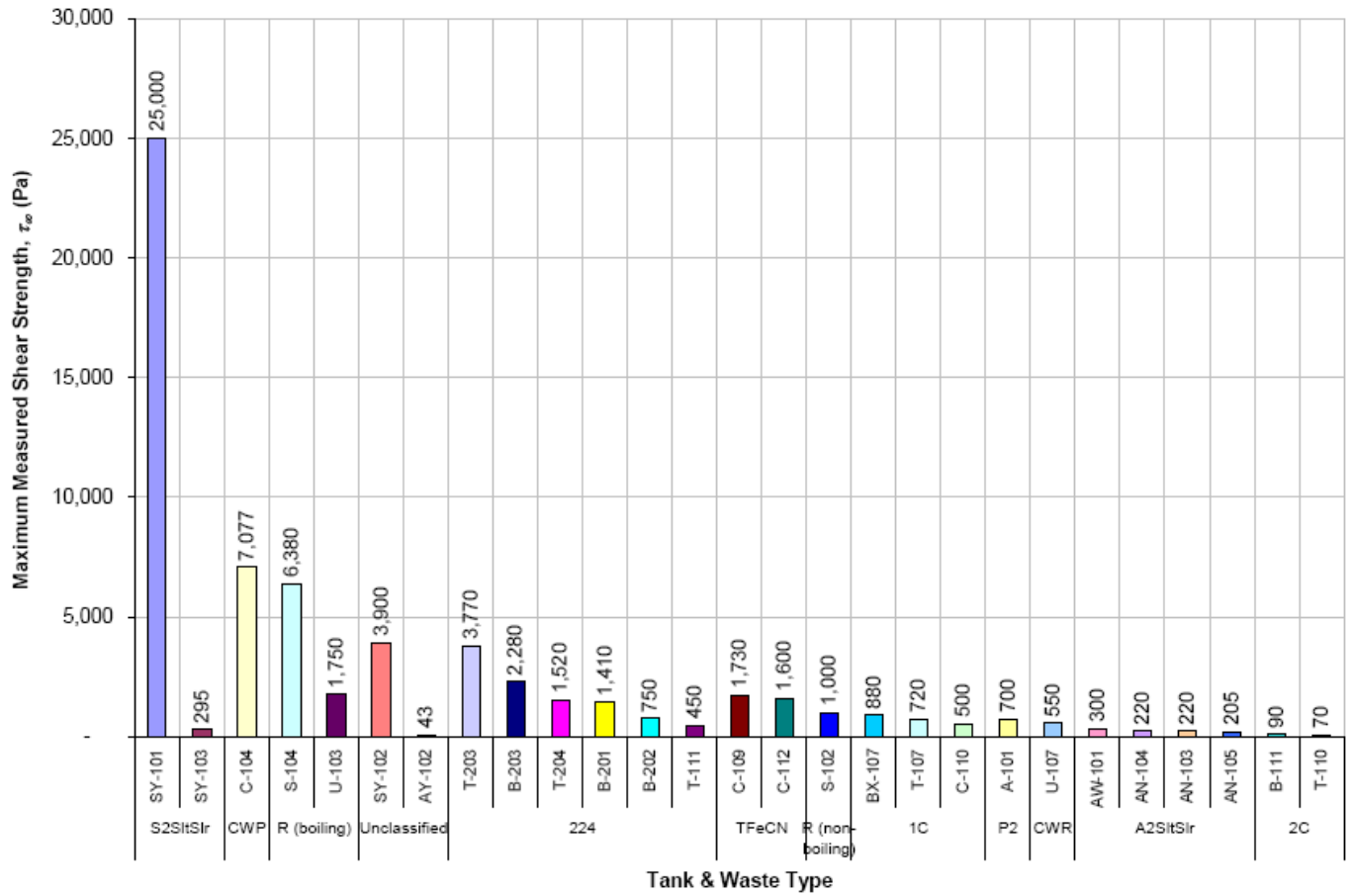
Figure 11.1. Shear Strength as a Function of Gel Time for HLW Pretreated Sludge

A.3.18

What are the flow behaviors of the sludge layer?

Shear Strength Data

- Measured with shear vane technique and penetrometer (in-situ)
- Large range of shear strength values
- SY-101 had large volume of retained gas and should be proportional to a rebuilding structure (shear strength)
- In several tanks the falling ball system was fully supported by a sludge layer (>9,000 Pa)

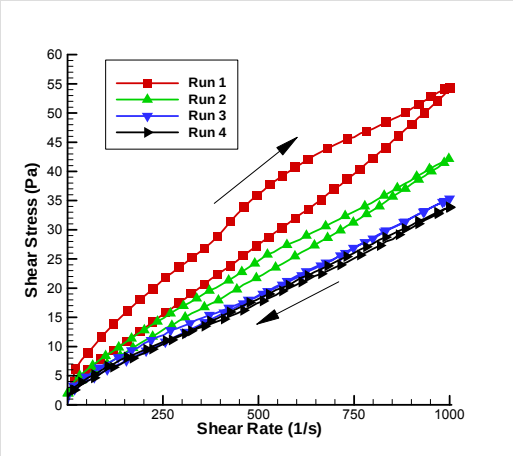


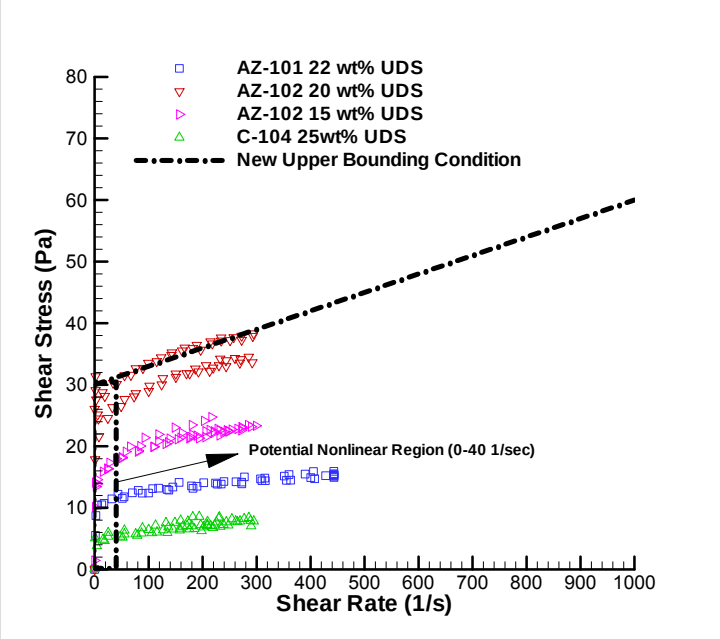
a) Maximum measured shear strength for each Hanford waste tank

What are the flow behaviors of the sludge layer?

Flow Curve Observations

- Ramp up and ramp down (top)
- Hysteresis loops indicate thixotropy (top)
- Runs repeated until equilibrium curve reached
- Viscoplastic behavior observed
- Bingham plastic model used
  - Two parameter model
  - Conservative design
  - Yield stress in model
  - infinite shear rate predicted
- Additional modeling can be performed on thixotropic decay parameters





What are the flow behaviors of the sludge layer?

Maximum Measured Bingham Consistency (Plastic Viscosity) Values

- "Consistency" nomenclature used to eliminate the term viscosity when discussing non-Newtonian fluids
- Measurements taken under a wide range of dilution conditions
- Maximum values typically represent sediment rheology
  - Important design information for plant restart

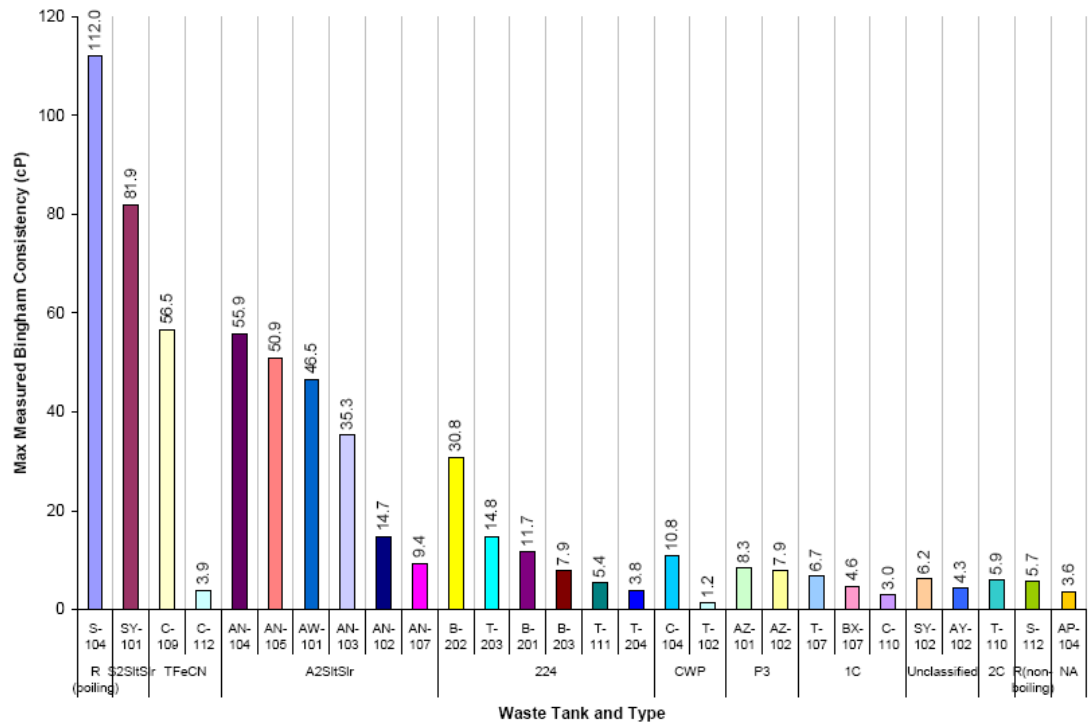


Figure 12.3. Maximum Measured Bingham Consistency for Various Hanford Tanks and Waste Types

What are the flow behaviors of the sludge layer?

Maximum Measured Bingham Yield Stress Values

- Measurements taken under a wide range of dilution conditions
- High range of maximum value represent sediment rheology under gravitational conditions
  - Important design information for plant restart
- Several of the thicker materials could not be accurately measured in viscometer due to wall slip
  - Excessive diluent was added and maximum values are significantly reduced

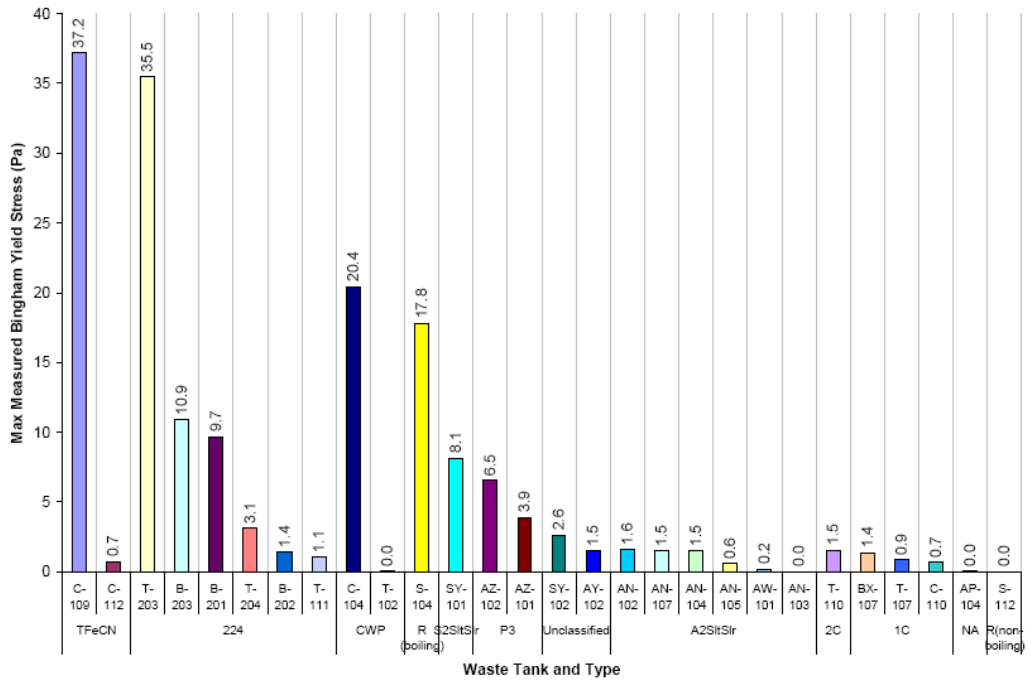


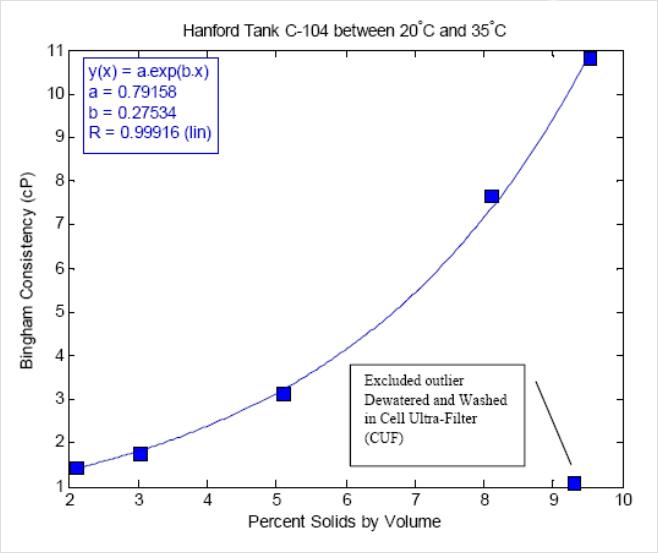
Figure 12.4. Maximum Measured Bingham Yield Stress for Various Hanford Tanks and Waste Types

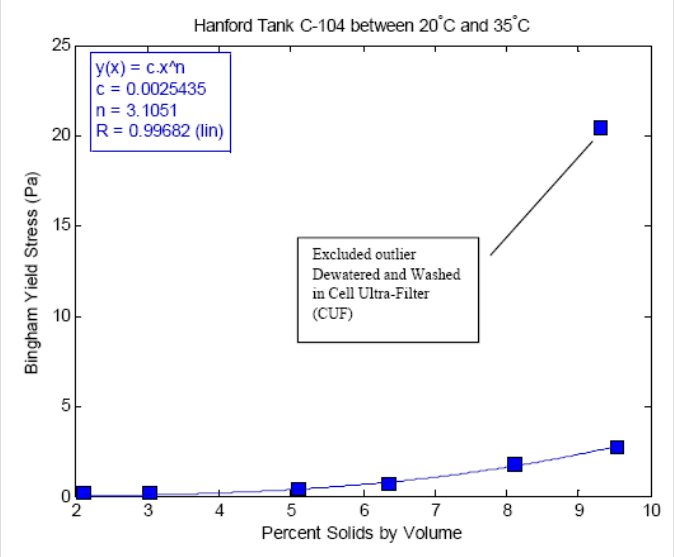
What are the flow behaviors of the sludge layer?

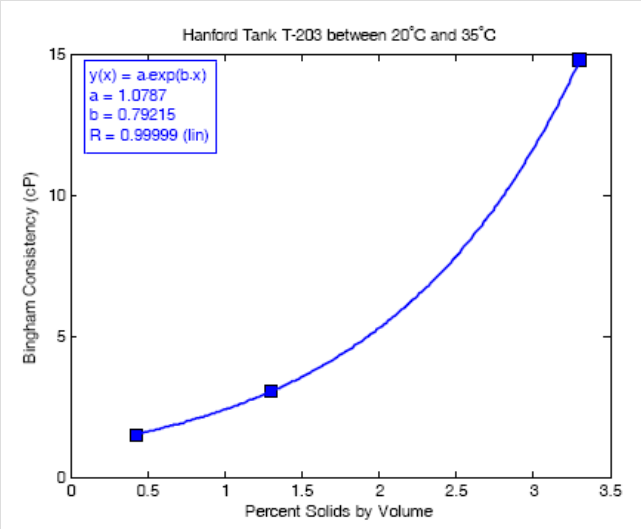
A.3.22

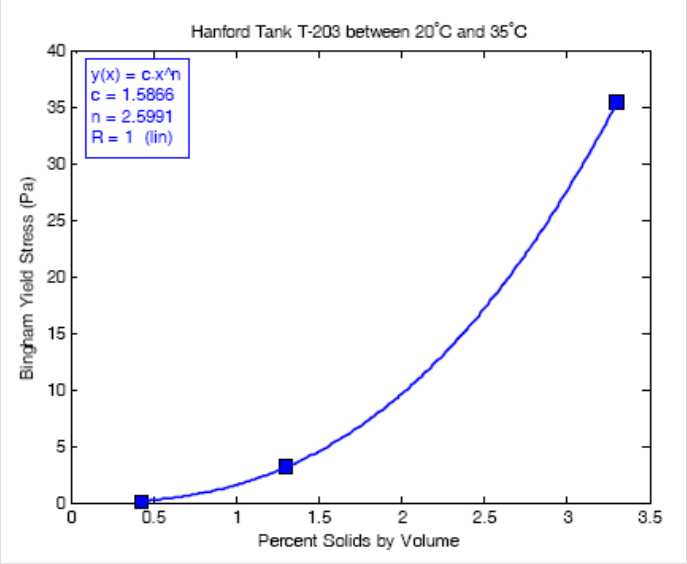
Bingham parameters as function of solids loading

- Empirical equation used to describe data
- Experimental data typically contains 2-3 points
- eliminates possibility of using 3+ parameter models
- Maximum packing fraction varies from tank to tank
  - Top ~10 vol%
  - Bottom ~5 vol%
- Very few of these correlations could be constructed
- Waste tanks should be further rheologically characterized







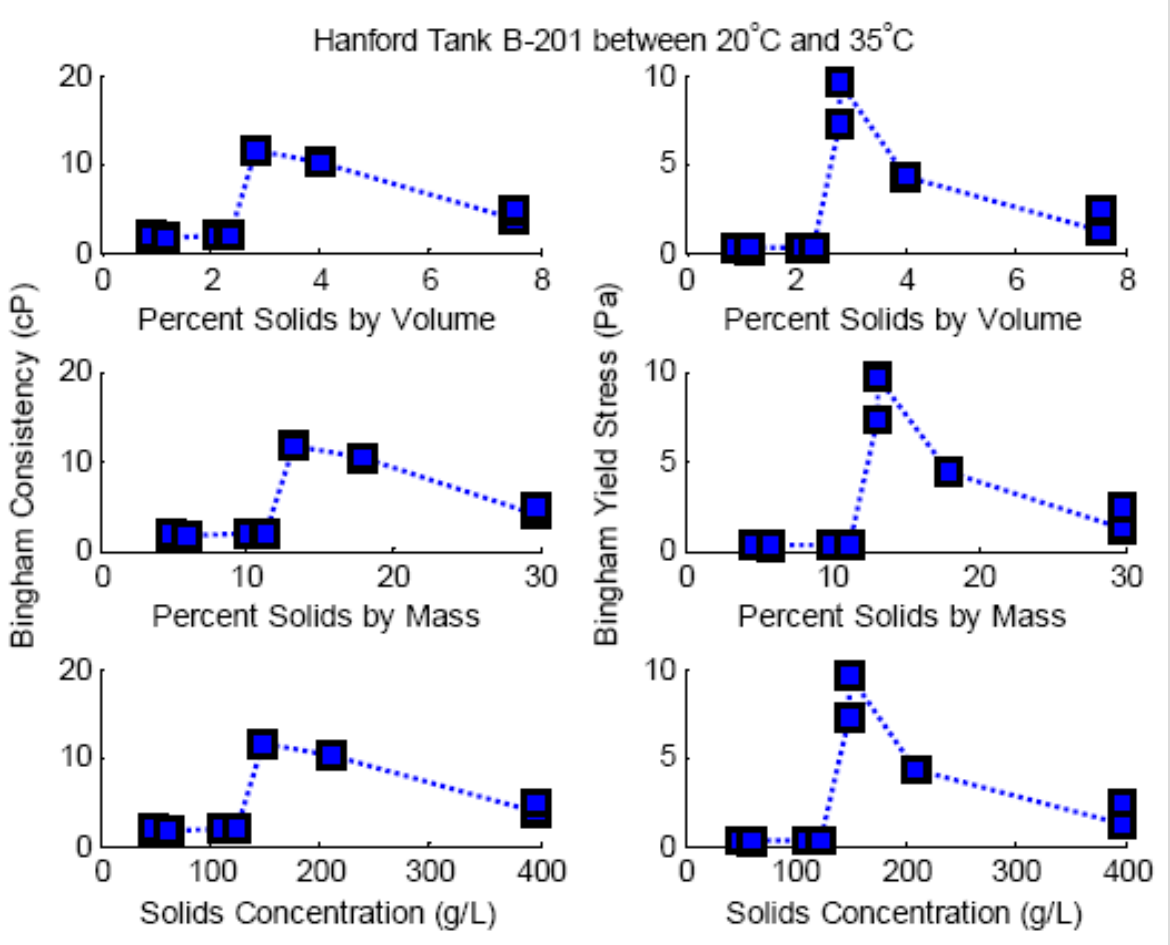


What are the flow behaviors of the sludge layer?

Water Dilution Chemical Effects

- As dilution with water ↑
- Zeta potential →0
- pH↓ can result in precipitation
- Particle size↕ competing effects of dissolution and precipitation
- Undissolved solids concentration↓

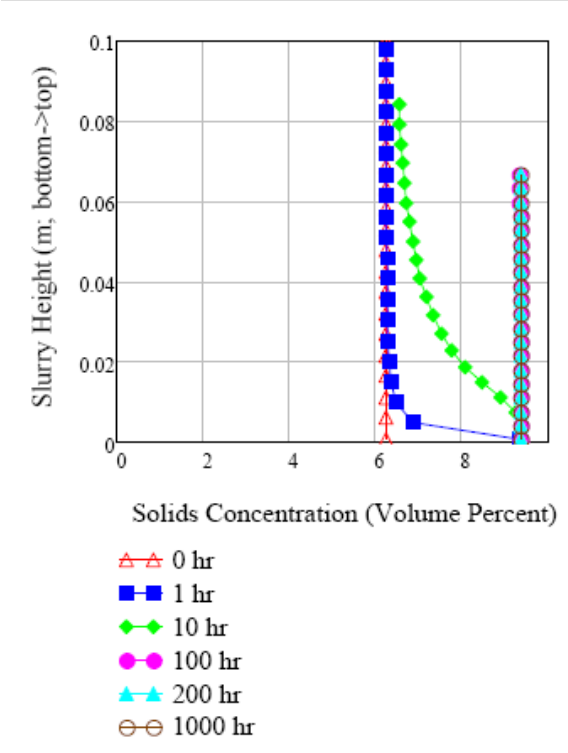
- Resulting rheological properties ↕
  - Maxima can exist during water dilution steps
  - Waste retrieval

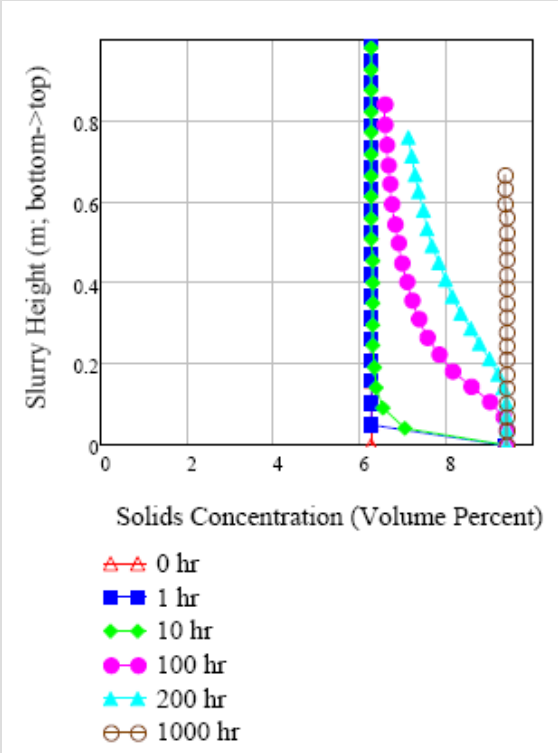


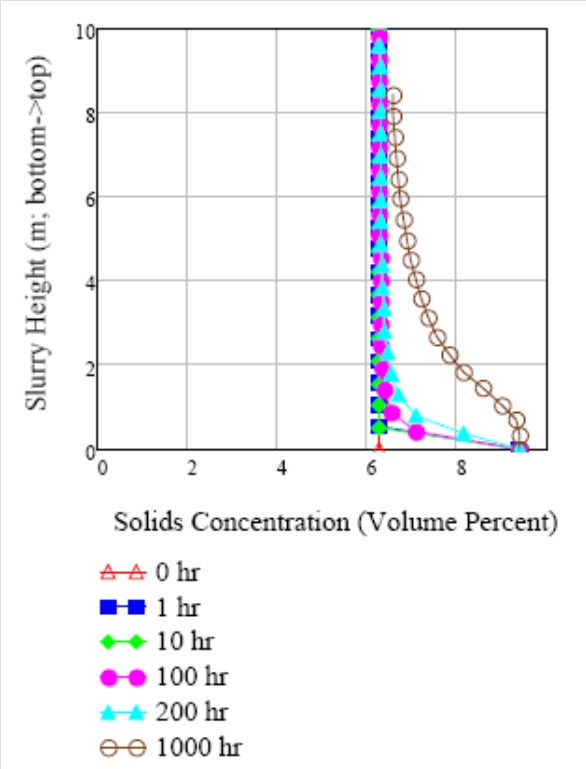
A.3.24

Effect of process scale on sedimentation times

- Calculated sedimentation concentration profiles
  - 0.1 m (left)
  - 1 m (center)
  - 10 m (right)
- Solids concentration profiles develop at a slower rate with increasing process height
- Bottom of tank has highest solids concentration for longest period of time



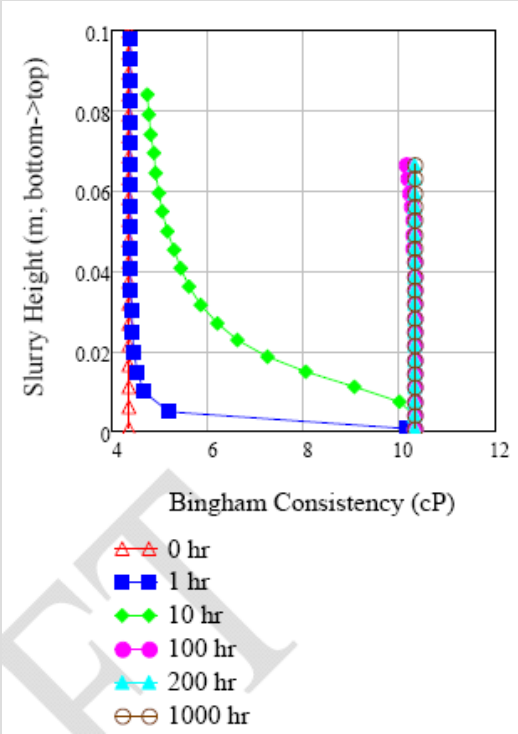


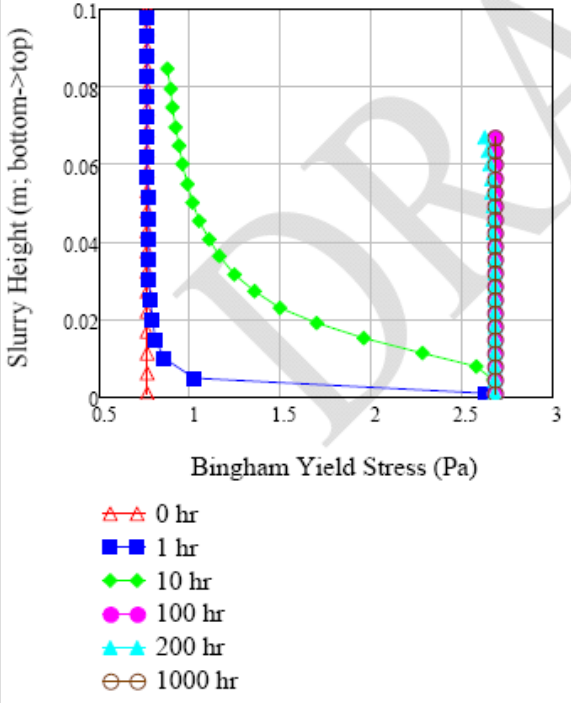


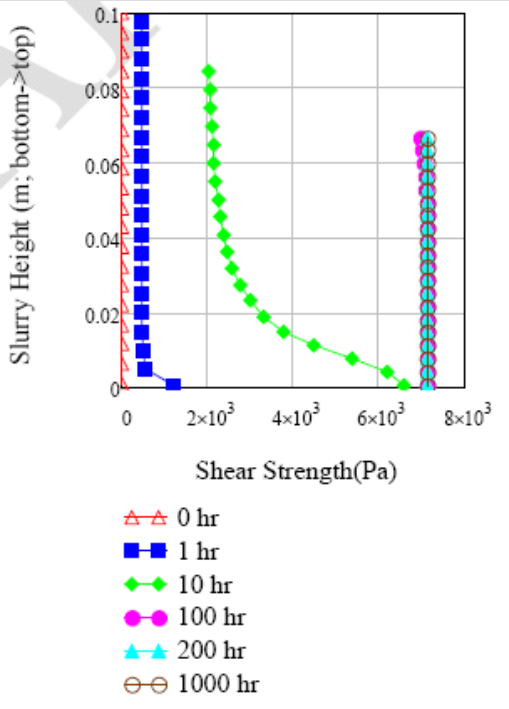
A.3.25

Transient rheological behavior during sedimentation

- 0.1 m starting slurry height
- Fully settle between 10-100 hrs
- Shear strength model account for solids increase and gel time separately
- Thick layer develops from the bottom towards the top with time







Degree of Physical Property Characterization

- Characterization effort was opportunistic rather than comprehensive
- Several types remain uncharacterized in several areas
- Quality of data gathered over 20 year period varies widely with different test objectives, research staff, equipment…

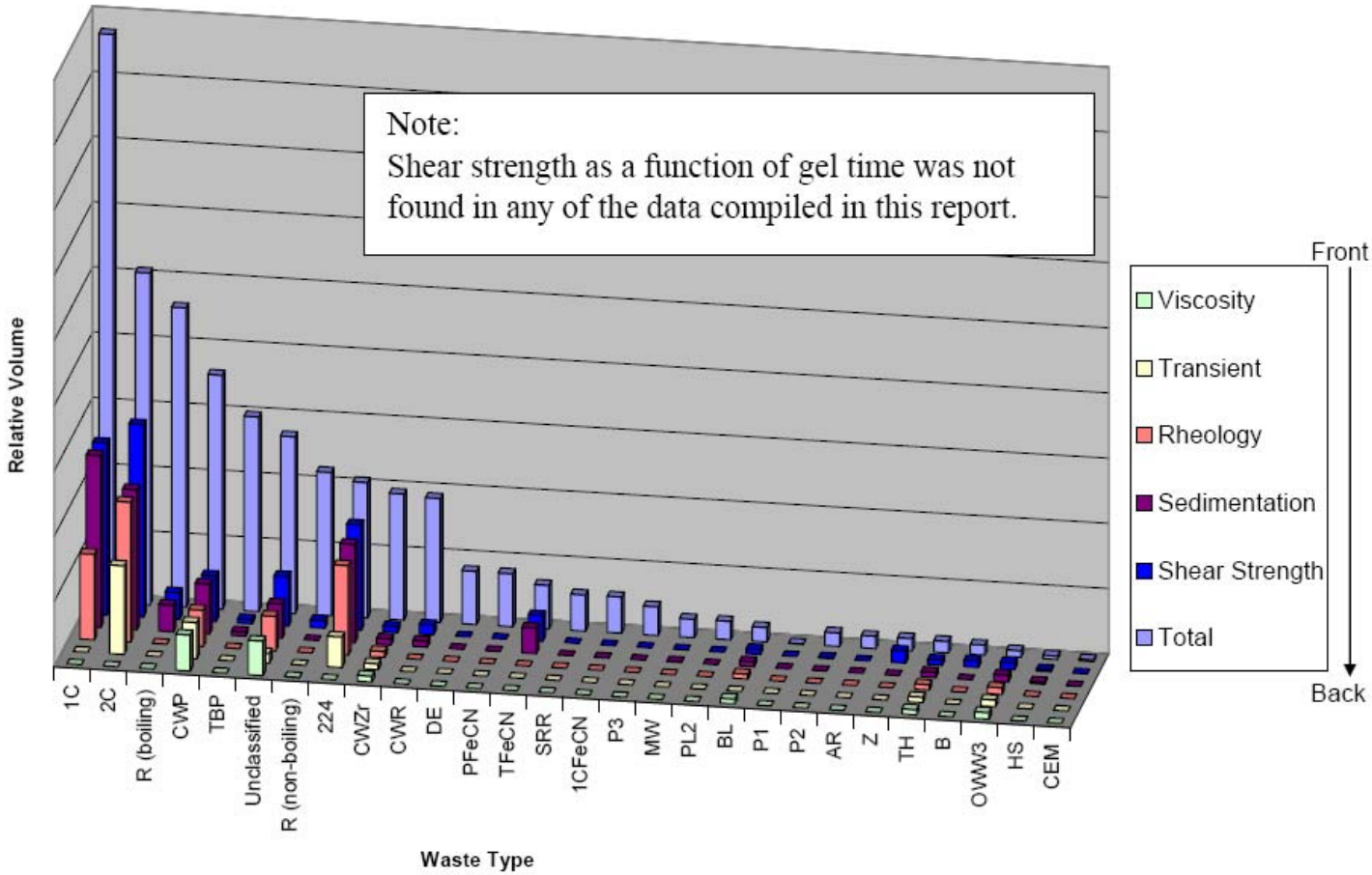


Figure S.6. Relative Volume of Waste Types Modeled Based on Waste Tank Data Available for Liquid Viscosity, Sedimentation, Shear Strength, Rheology, and Transient Modeling Compared with the Total Volume of Each Sludge Waste Type

Summary

Table 14.1. Range of Rheological Parameters and Regrowth Times at Typical Process Scales

Category	Heel Shear strength	Slurry/Sediment Heel Bingham Yield Stress	Slurry/Sediment Heel Bingham Consistency	Supernatant Viscosity
Min[a]	40 Pa	0 Pa	1 cP	1 cP
Median[a]	700 Pa	1.5 Pa	8 cP	8 cP
Max[a]	25,000 Pa	40 Pa	110 cP	30 cP
Tank heel property after 10 hours of sedimentation in process piping (0.1 m sludge height)	25,000 Pa	40 Pa	110 cP	n/a
Tank heel property after 100 hours of sedimentation in a medium-scale vessel (1 m sludge height)	25,000 Pa	40 Pa	110 cP	n/a
Tank heel property after 1000 hours of sedimentation in a large-scale vessel (10 m sludge height)	25,000 Pa	40 Pa	110 cP	n/a

(a) Statistics performed on all compiled data discussed in this report.
n/a – not applicable.

A.3.28

- Particle Size and Density
  - Distributions were decoupled and a set of possible distribution attained via Monte Carlo simulation
- Sedimentation Data
  - Obtained empirical sedimentation models for Hanford sludge
  - Reaches fully settled configuration in the range of several hours to several days with 10 hours as typical value
- Flow Curve Data
  - Bingham parameters were modeled
  - Coupled with sedimentation data to obtain transient equation
  - Can reach values well above 30 cP, 30 Pa within 10 hours
- Shear Strength Data
  - Coupled with sedimentation and Bingham yield stress model for obtain transient equation
  - Can reach values well above 10,000 Pa within 10 hours
- WTP throughput may be rheologically limited due to high rheological properties at design solids concentrations
  - This situation should be anticipated integrated into the baseline plan
- More information at http://www.pnl.gov/rpp-wtp/

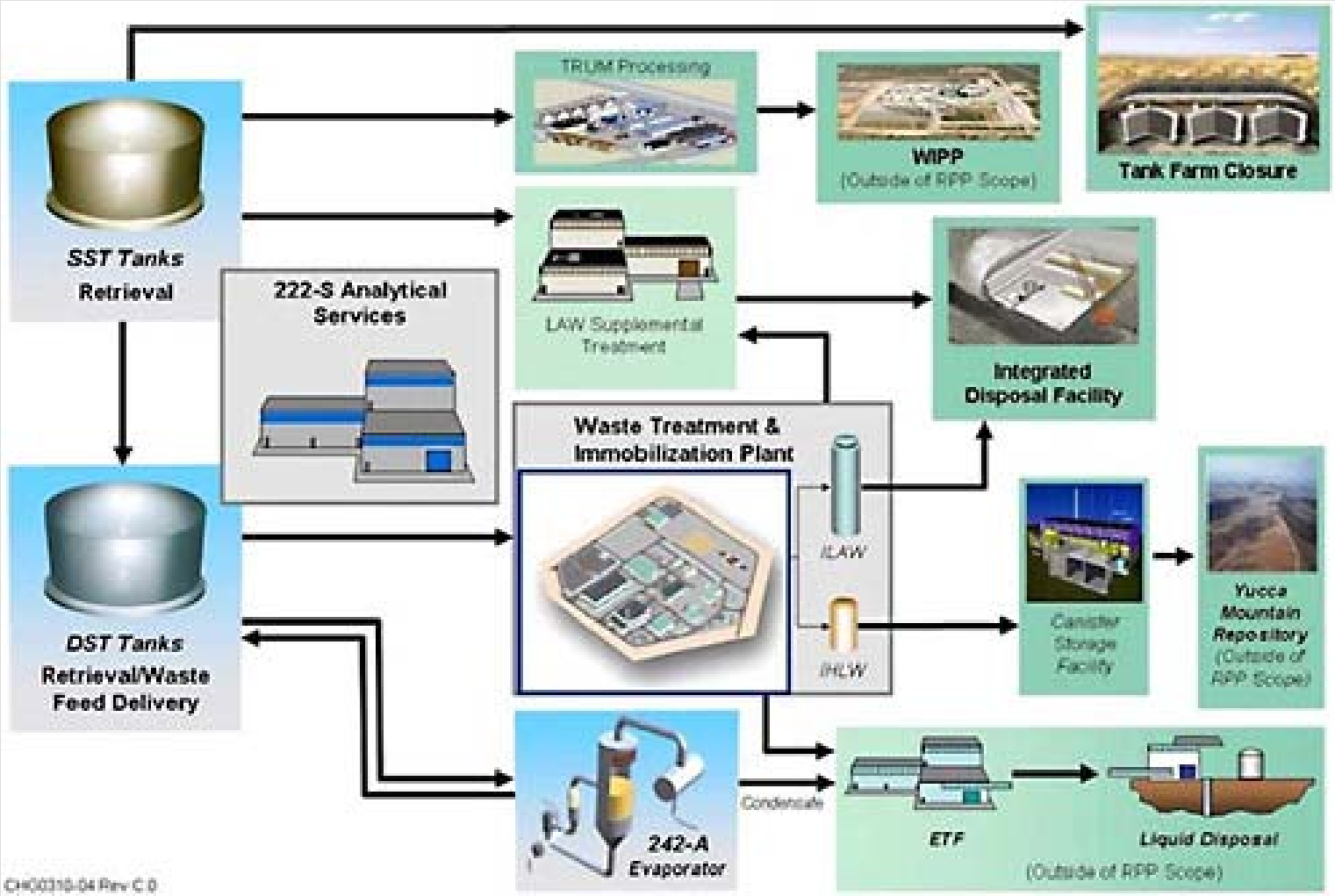
Hanford Sludge Processing

Adam Poloski

Battelle

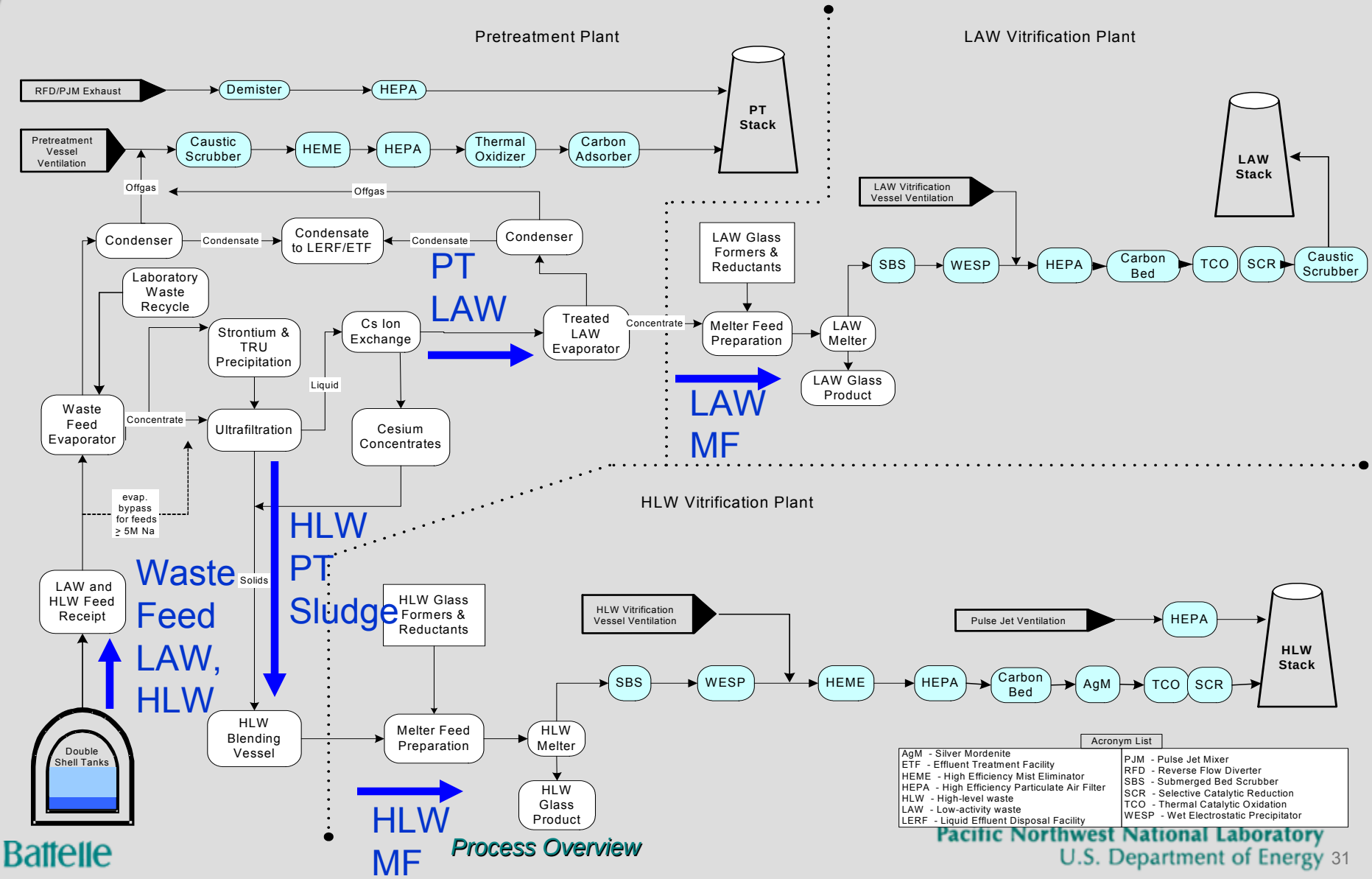
Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

Simplified Hanford Waste Process Flow Diagram



A.3.30

Simplified WTP Process Flow Diagram



Process Overview

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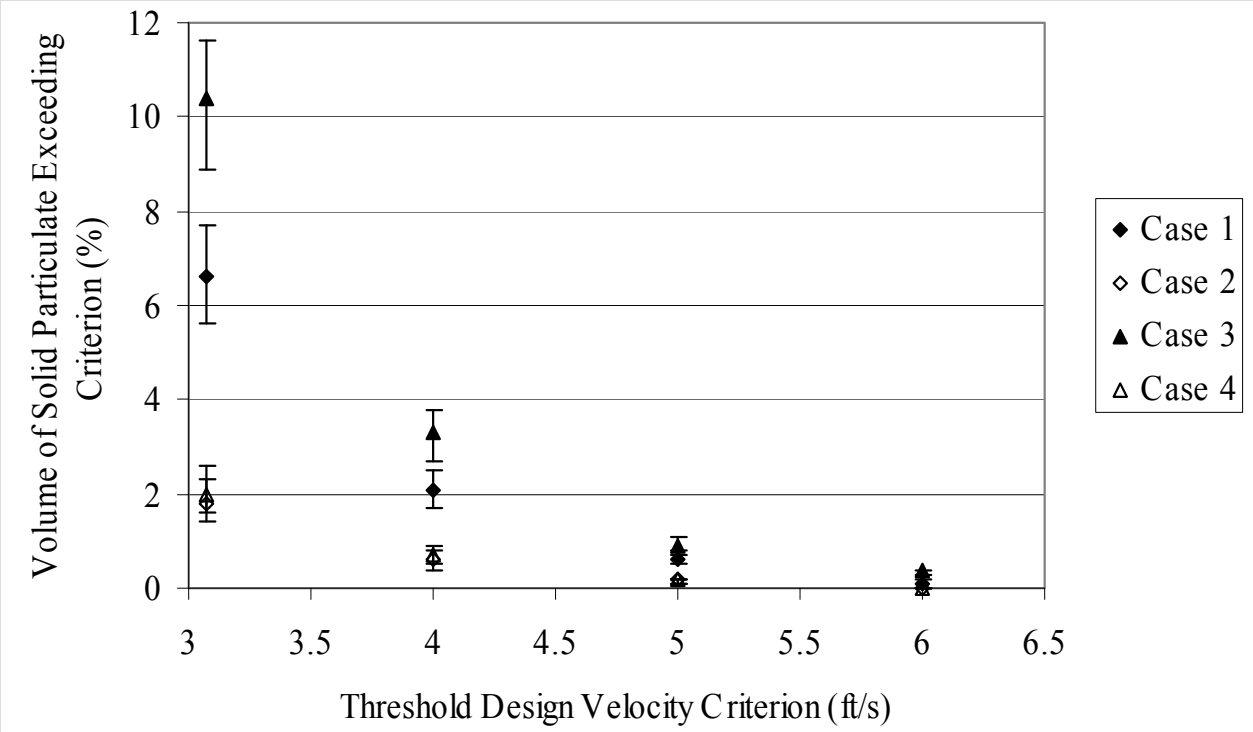
U.S. Department of Energy

Waste Transport – Critical Velocity Calculations

- PSDDs from Monte Carlo results are evaluated
- Oroskar & Turian equation is design basis
- Flow Conditions
  - Pipe diameter = 3 in.
  - Liquid density = 1.2 g/mL
  - Liquid viscosity = 2 cP
  - Mass fraction of solids in flow = 0.154
- Up to 3% of sludge by volume may form a stationary bed in process piping at 4 ft/sec
  - 3.1 ft/sec is WTP design velocity
  - 4 ft/sec with design margin (+30%)
  - Represents 400,000 gallons of sludge
- Experimental flow loop program in place to test Hanford size/density particle scenarios in carrier slurries
  - Complex process geometries
  - line unplugging of chemical precipitates

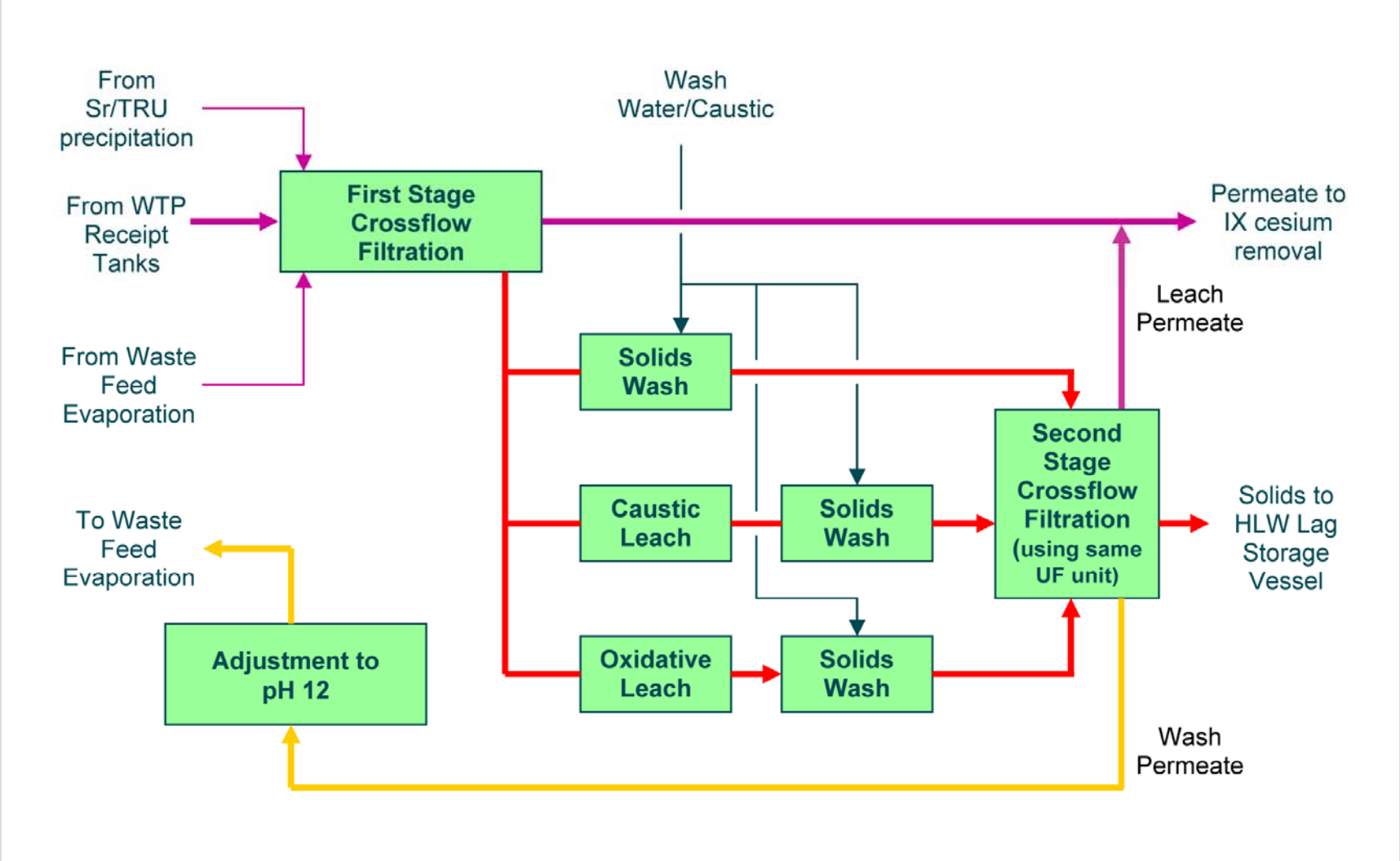


A.3.32

Critical Velocity Calculations, Case 1 at 3.1 ft/s

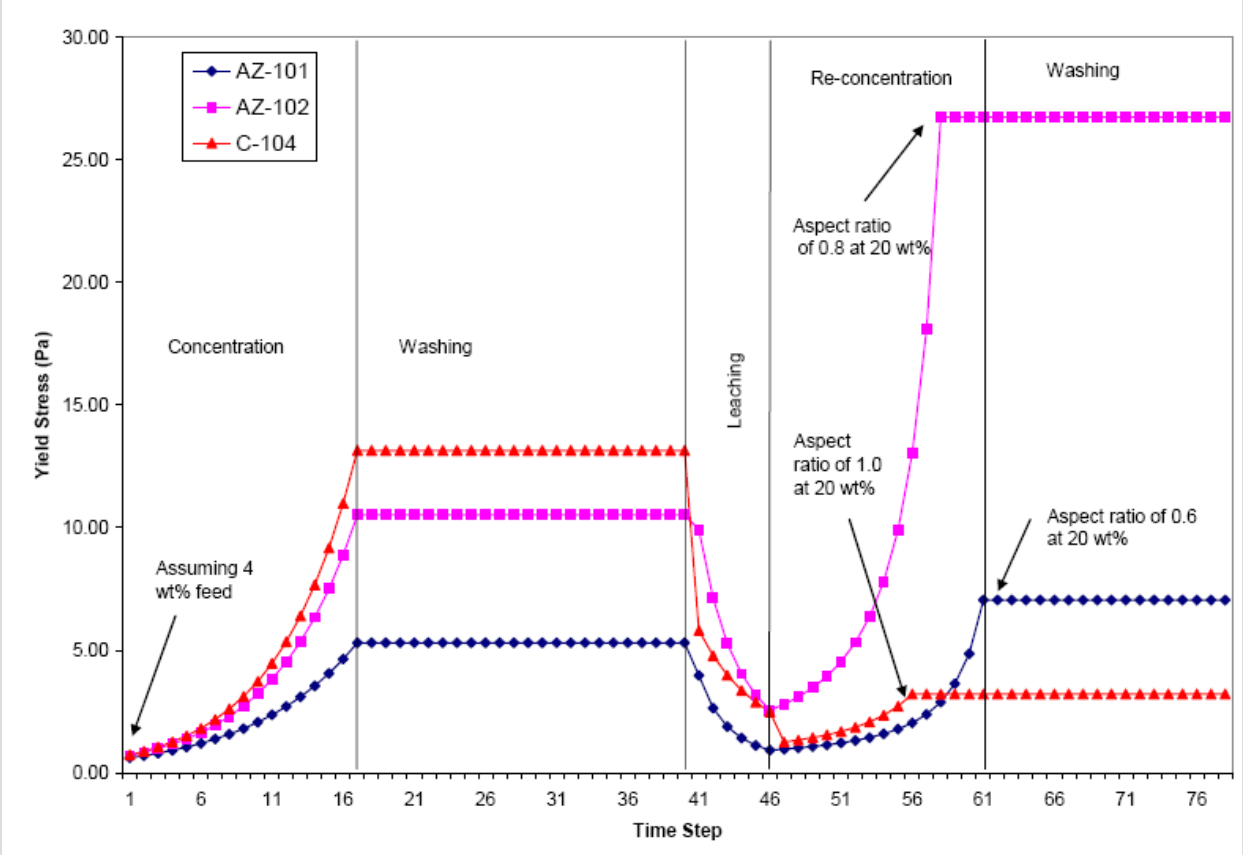
Solid-Phase Compound	Density, g/mL	Representative Case 1 PSDD Volume %	Percent Exceedance
$Al(OH)_3$, Gibbsite	2.42	51.5	1%
$(NaAlSiO_4)_6 \bullet (NaNO_3)_{1.6} \bullet 2H_2O$	2.365	16.6	1%
AlOOH, Boehmite	3.01	10.6	4%
$NaAlCO_3(OH)_2$	2.42	9.5	1%
Fe_2O_3	5.24	4.1	69%
$Ca_5OH(PO_4)_3$	3.14	2.0	7%
$Na_2U_2O_7$	5.617	1.6	74%
ZrO_2	5.68	1.1	74%
Bi_2O_3	8.9	0.81	100%
SiO_2	2.6	0.69	1%
$Ni(OH)_2$	4.1	0.55	31%
MnO_2	5.026	0.54	64%
CaF_2	3.18	0.23	7%
$LaPO_4 \bullet 2H_2O$	6.51	0.13	89%
Ag_2CO_3	6.077	0.0094	81%
PuO_2	11.43	0.0013	100%

Leaching & Filtration PFD



Filtration and Leaching – Predicted Bingham Yield Stress Changes During Leaching and Filtration

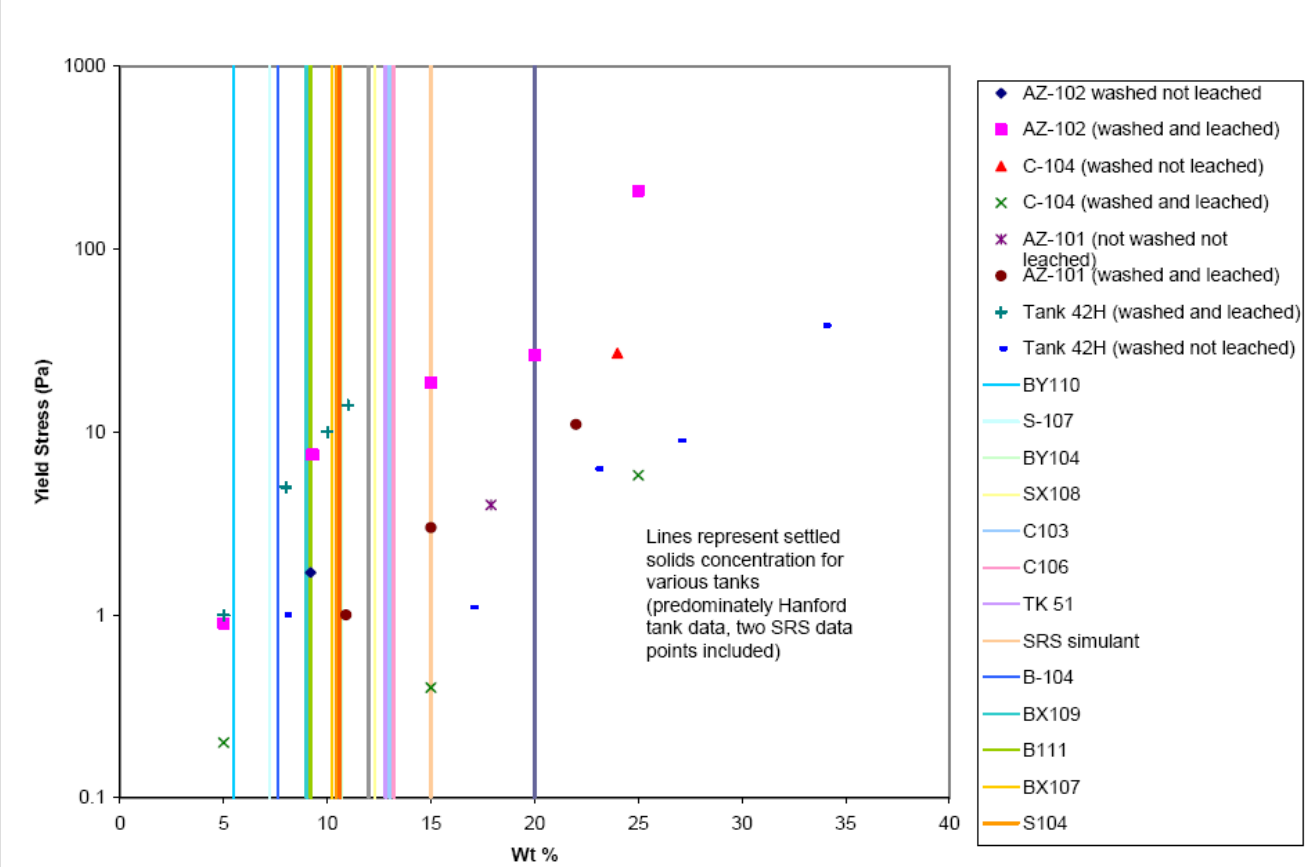
- This figure is based on the following set of assumptions.
  - That the feed is initially concentrated to 20 wt% UDS at 17,000 gallons
  - That washing has little impact on yield stress
  - That 5,000 gallons is added during leaching
  - That the changes in rheology caused by leaching occur immediately, before any significant solids dissolution occurs
  - That all the observed dissolution occurs during the leaching process
  - That the waste is concentrated to 20 wt% UDS after leaching.
- Data not available on how rheological properties will change through this process
  - PSD, shape, ionic strength, pH, all vary throughout this process



A.3.35

Comparison of gravitational sedimentation to ultrafilter concentration

- Target solids concentration is 20 wt% during filtration & leaching
- Assumes that maximum wt% solids under gravitational settling conditions ∝ maximum packing fraction
- Dewatering past point achievable due to gravitational sedimentation
- Estimate that 20-30% of HLW waste tanks may exceed 30 Pa yield stress bound during filtration and leaching
- Values of 100 Pa yield stress observed during leaching/filtration
  - Under BNFL
  - Recently under M-12
- Small scale: Moyno pump and mechanical agitators
- Process scale: Centrifugal pump and PJMs
- Large
- More data needed to reduce risk



Process Mixing

- Mixing and suspension of particles in Newtonian and non-Newtonian fluids
- Mechanical agitators, pulse jet mixers (PJMs for bottom of tank), and air spargers (top of tank) utilized for mixing
- Data indicates that sufficient mixing can occur in non-Newtonian fluids with this technique
- Ongoing experimental programs to investigate Newtonian systems
  - Cloud heights
  - Just suspended velocities
  - Blend times

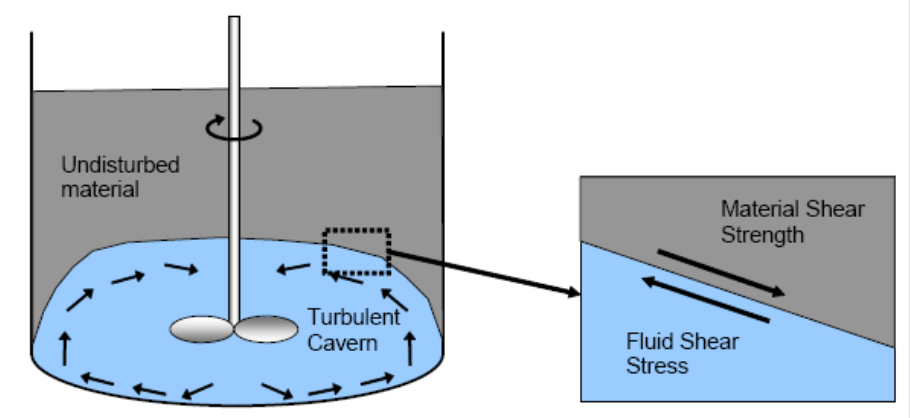


Figure 1.2. Illustrating Cavern Formation in Non-Newtonian Waste

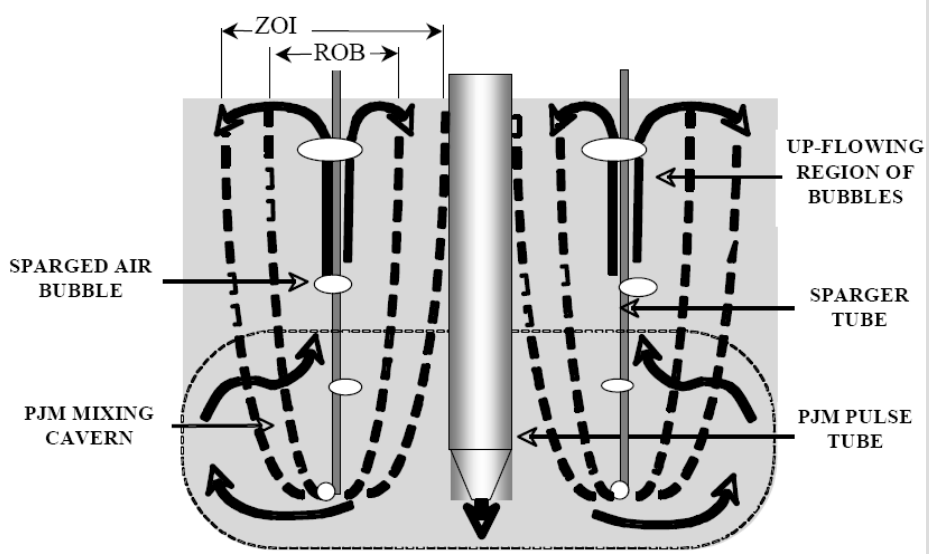


Figure 1.1. Illustration of Hybrid PJM/Sparger Mixing Concept

A.3.37

Gas Retention and Release

- Flammable gases are generated due to thermolysis and radiolysis
- During outages gas holdup will increase in non-Newtonian fluids
- Experimental programs have investigated gas retention and release dynamics under these scenarios
- Antifoam agent changed hold-up values
- Experimental results indicate that
  - Retrained gas released quickly after mixing
  - Minimal buildup of retrained gas through "intermittent operation"

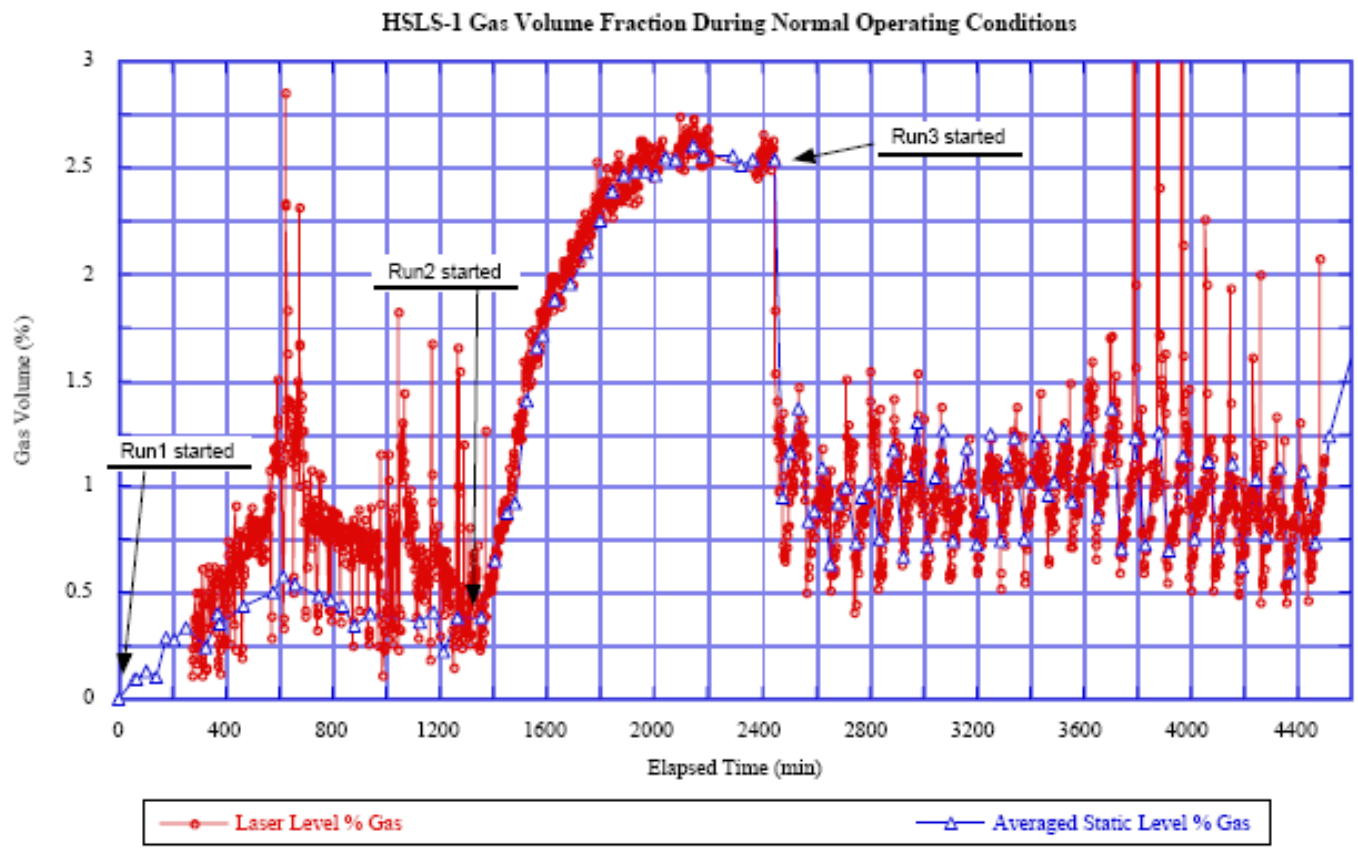


Figure 6.8. Gas Volume Fraction During HSLS-1

A.3.38

Pretreated LAW

- Viscosity of actual waste permeate after washing and leaching
- Limit of 15 cP placed for pumping through IX bed
- IX resin handling issues
  - Saturated conditions exist while processing through IX bed
  - IX resin swells under varying chemical conditions

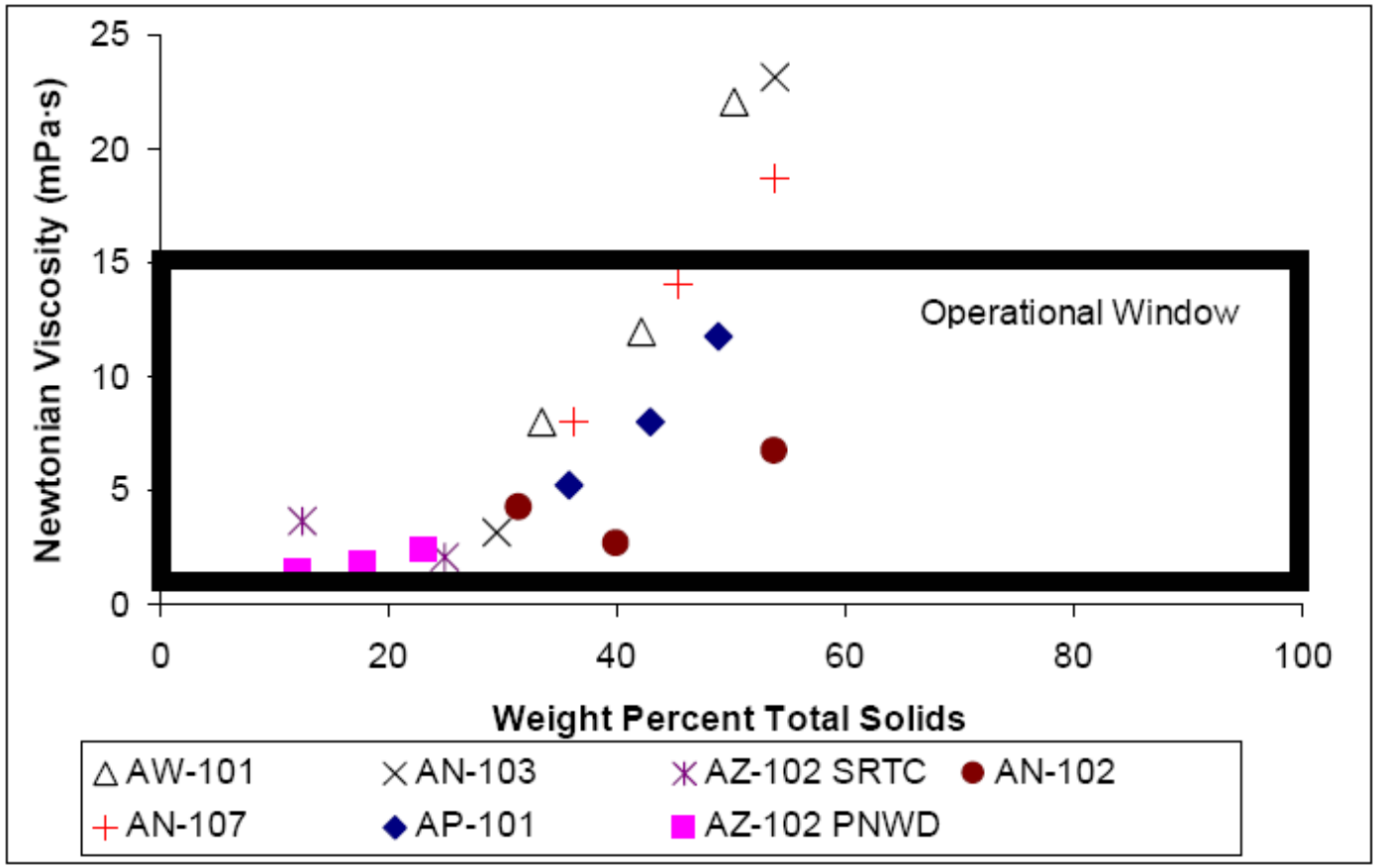


Figure 4.14. Comparison of Actual Data to Proposed Bounding Conditions as a Function of Weight Percent Total Solids for Pretreated LAW at 25°C

A.3.39

Melter Feeds

- Glass Former Chemicals added to pretreated waste feeds prior to vitrification
- Dry solids handling issues very different from slurry handling
- SRNL performed a large amount of work in this area

Table 4.1. Settling Information on Glass-Former Chemicals

No.	Name	Particle Size (Mesh; M)	Estimated Particle Density (g/mL)	Free Settling Velocity in Water[a] (m/s)	Reference
1	Kyanite	<325 M (44 μm)	3.61	0.0027	http://webmineral.com/data/Kyanite.shtml
	Alumina A2	<325 M (44 μm)	3.7	0.0027	http://www.reade.com/Products/Oxides/alumina.html
2	Boric Acid	>20 M (841 μm)	1.51	0.061	http://www.sqm-mx.com/q_industriales/productos/pdf/boric_acid_msds.pdf
3	10 M Borax	>8 M (2380 μm)	1.71	0.19	http://webmineral.com/data/Borax.shtml
4	Na_2CO_3	<100 M (149 μm)	2.54	0.015	http://webmineral.com/data/Natrite.shtml
5	Wollastonite	<325 M (44 μm)	2.84	0.0018	http://webmineral.com/data/Wollastonite-1A.shtml
6	Fe_2O_3	<325 M (44 μm)	5.3	0.0042	http://webmineral.com/data/Hematite.shtml
7	Li_2CO_3	<200 M (74 μm)	2.11	0.0032	http://www.chemetalllithium.com/
8	Olivine	<200 M (74 μm)	3.32	0.0062	http://www.webmineral.com/data/Olivine.shtml
9	SiO_2	<200 M (74 μm)	2.65	0.0045	http://www.u-s-silica.com/prod_info/PDS/Mill_Creek/MiCSCS752000.PDF
10	Rutile -94	Airfloated <80M (177 μm)	4.25	0.036	http://www.webmineral.com/data/Rutile.shtml
11	ZnO	1 μm	5.6	0.0000025	http://www.zinccorp.com/TD%20for%20Kadox%20920.pdf
12	$ZrSiO_4$	<325 M (44 μm)	4.65	0.0037	http://www.webmineral.com/data/Zircon.shtml
13	Sugar	NA	1.58	NA	http://www.alfa.com/CGI-BIN/LANSAWEB?WEBEVENT+L0422B63C7F078000D26B011+ALF+ENG

(a) Note that the free settling values are calculated values using Equation 4.4.

A.3.40

LAW Melter Feed Rheology

- Rheology dominated by large quantities of glass former chemicals
- Properties tend towards a characteristic curve
- Processing limits established at 100 cP and 20 Pa yield stress

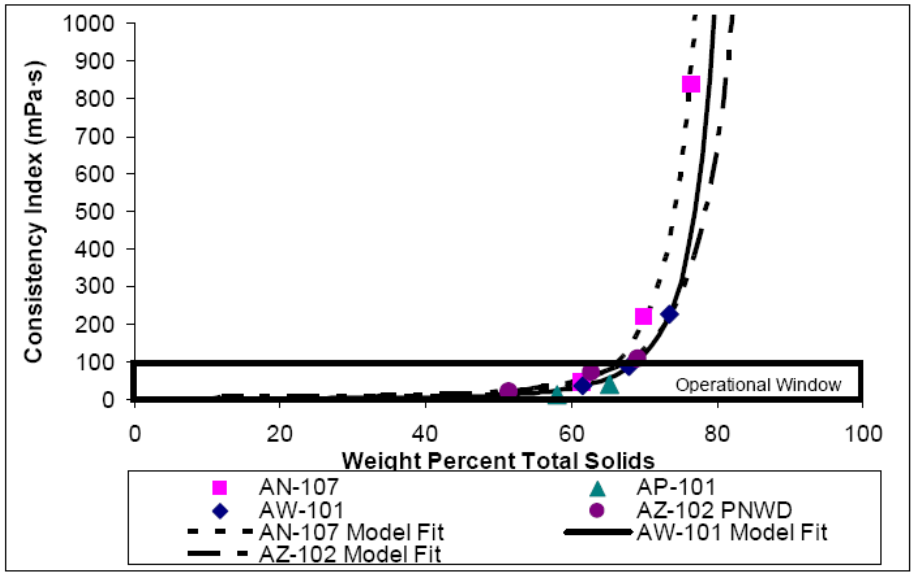


Figure 4.17. Bingham Consistency Index as a Function of Weight Percent Total Solids for LAW Melter Feed at 25°C

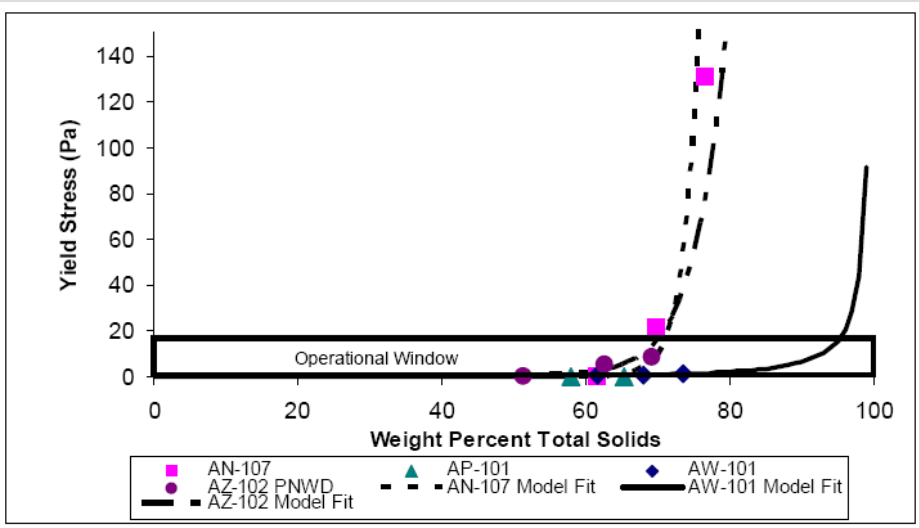


Figure 4.18. Bingham Yield Index as a Function of Weight Percent Total Solids for LAW Melter Feed at 25°C

A.3.41

Affects of aluminate precipitation on pretreated LAW

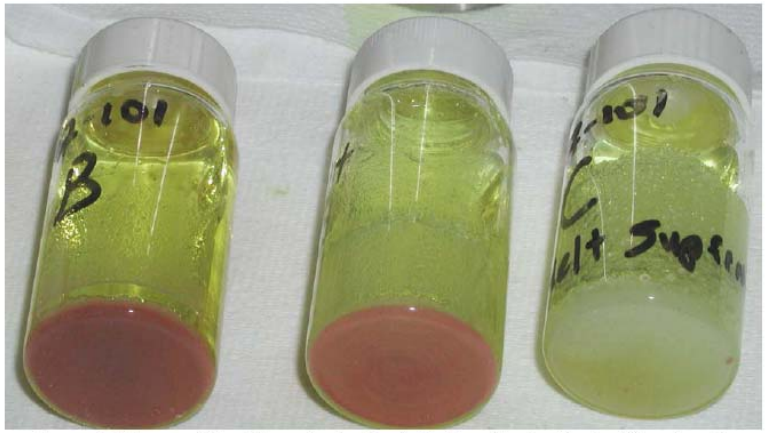
- Boric acid addition
- pH drops
- Aluminate precipitation
- Particle bridging results in high shear strengths

Table 5.2. Summary of Shear Strength Data at 40°C

Sample	Temp.	Shear Strength (Pa)		
		Run 1[a]	Run 2	Run 3
1.75 M Na	40°C	2,430	>11,600	>18,600
2,75 M Na	40°C	>11,600	>11,600	n/a
3.75 M Na	40°C	950	>11,600	n/a

n/a – not applicable

a, samples partially agitated



note: precipitates are white solids; red coloration is due to residual melter feed from decanting

Figure 5.5. Crystalline Precipitate Observed from Decanted AZ-101 Envelope B Melter Feed

a) Settled solids layer when tilted to one side.

b) Settled solids layer before mixing.

c) Settled solids layer during mixing.

Figure 5.6. High Shear Strength of AZ-101 Envelope B Melter Feed During Mixing

HLW Melter Feed

- Rheological properties of actual HLW melter Feed
- Similarly dominated by large quantities of GFCs

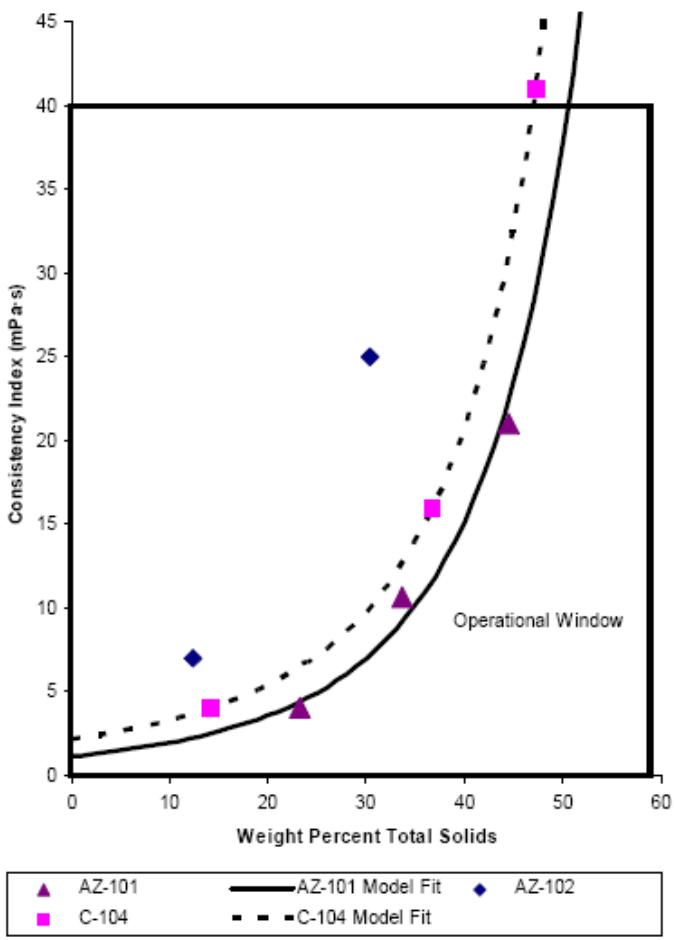


Figure 4.15. Bingham Consistency Index as a Function of Weight-Percent Total Solids for HLW Melter Feed at 25°C

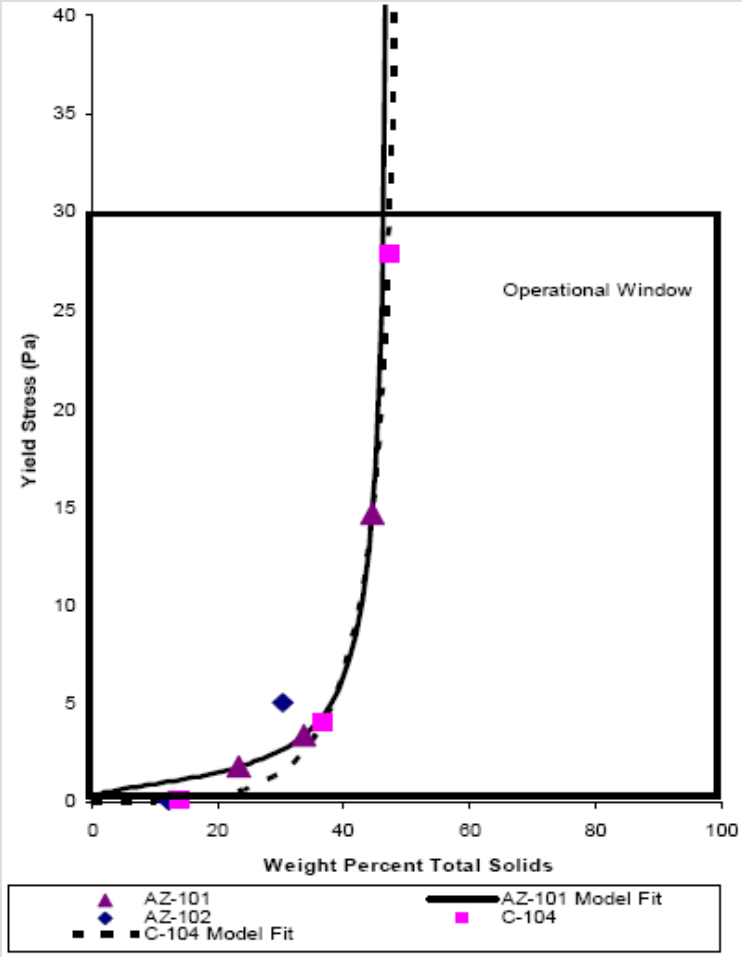


Figure 4.16. Bingham Yield Index as a Function of Weight-Percent Total Solids for HLW Melter Feed at 25°C

A.3.43

LAW Slurry Stream Summary

Table 3.4. Summary of Appendix A Database

Property	LAW Pretreated Waste	LAW Melter Feed
Chemical Composition	varies (see Appendix A)	varies (see Appendix A)
pH	~14	7–14[a]
Particle-Size Distribution (PSD)	n/a	<164 μm
Heat Capacity	n/a	n/a
Bulk Density	1.1–1.5	1.7–1.9
Supernatant Liquid Density	1.1–1.5	1.2–1.6
Vol % Settled Solids	0%	50%–100%
Settling Rate	n/a	n/a
Centrifuged Solids Density	n/a	n/a
Vol % Centrifuged Solids	n/a	n/a
Wt % Centrifuged Solids	n/a	n/a
Wt % Oven Dried Solids	n/a	n/a
Wt % Total Dried Solids	20%–60%	60%–75%
Wt % Undissolved Solids	n/a	n/a
Shear Stress Versus Shear Rate ambient and 40°C	Newtonian	Bingham Plastic
Shear Strength	n/a	n/a
Wt % total oxide	15%–30%	25%–35%

a - addition of acidic GFCs will drop the pH of the LAW pretreated waste this drop in pH can precipitate amphoteric species such as aluminum hydroxide. Aluminum hydroxide precipitation can result in relatively high rheological parameters and processing difficulties.

A.3.44

HLW Slurry Stream Summary

Table 3.3. Summary of Appendix A Database

Property	HLW Pretreated Waste	HLW Melter Feed
Chemical Composition	varies (see Appendix A)	varies (see Appendix A)
pH	~12[a]	7–12[b]
PSD[c]	<50 μm	<105 μm
Heat Capacity	n/a	n/a
Bulk Density	1.1–1.2	1.1–1.5
Supernatant Liquid Density	~1.0	~1.0
Vol% Settled Solids[c]	10%–90%	20%–90%
Settling Rate	n/a	n/a
Centrifuged Solids Density	n/a	n/a
Vol% Centrifuged Solids	n/a	n/a
Wt% Centrifuged Solids	n/a	n/a
Wt% Oven Dried Solids	n/a	n/a
Wt% Total Dried Solids	5%–36%	10%–61%
Wt% Undissolved Solids	6%–35%	38%–54%
Shear Stress Versus Shear Rate ambient and 40°C	Newtonian or Bingham Plastic	Bingham Plastic
Shear Strength	n/a	n/a
Wt% total oxide	7%–15%	25%–40%

n/a not available
(a) expected pH after washing in 0.01 M NaOH
(b) expected pH after boric acid GFC addition
(c) See Appendix B

A.3.45

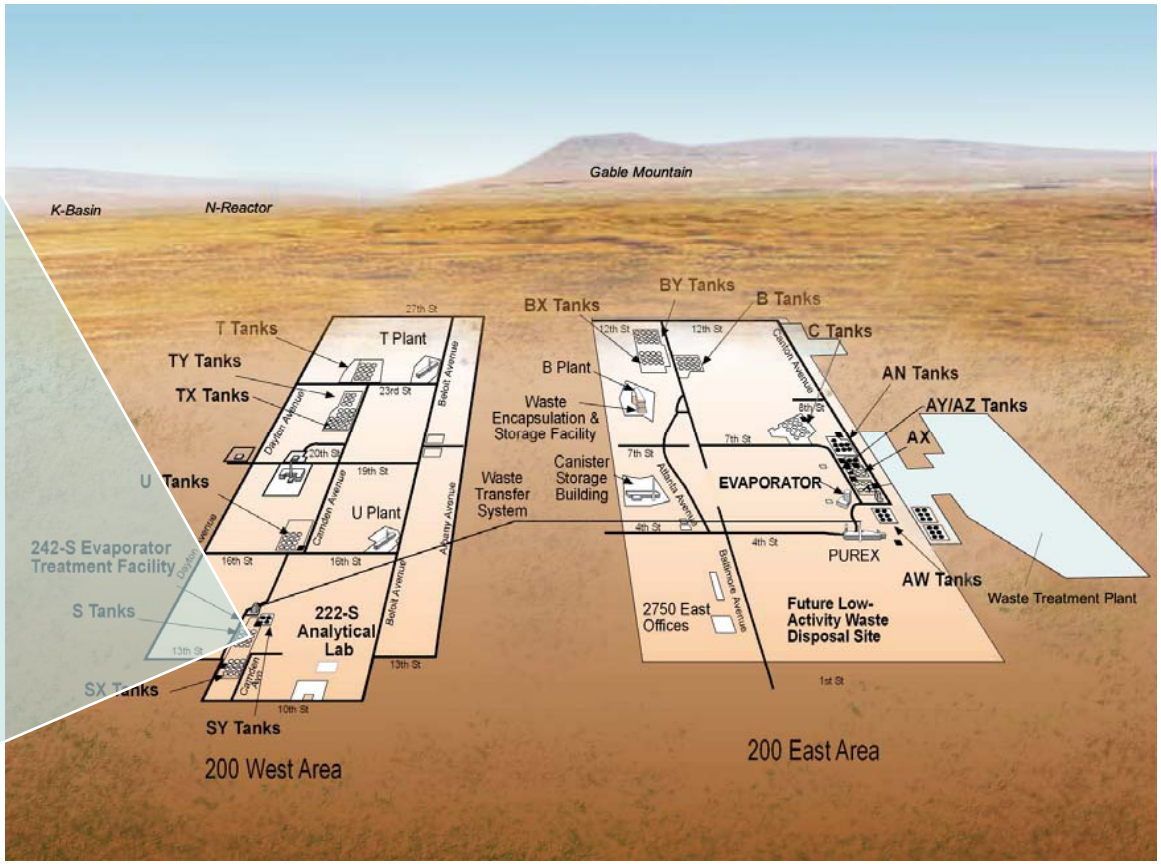
Summary

- Waste processing will significantly change all important slurry attributes
  - PSD, density, solids concentration, ionic strength, temperature, pH, zeta potential, particle shape
- Each major waste stream should be fully characterized under these conditions and data supplied to design team
- Waste feed batching complicates this process
- Very limited data exists on the affects of:
  - waste processing on slurry properties
  - slurry properties on waste processing

S-102 Event on July 27, 2007 -and- Lessons to be Learned Relating to Sludge/Slurry Processing

S-Farm

S-102

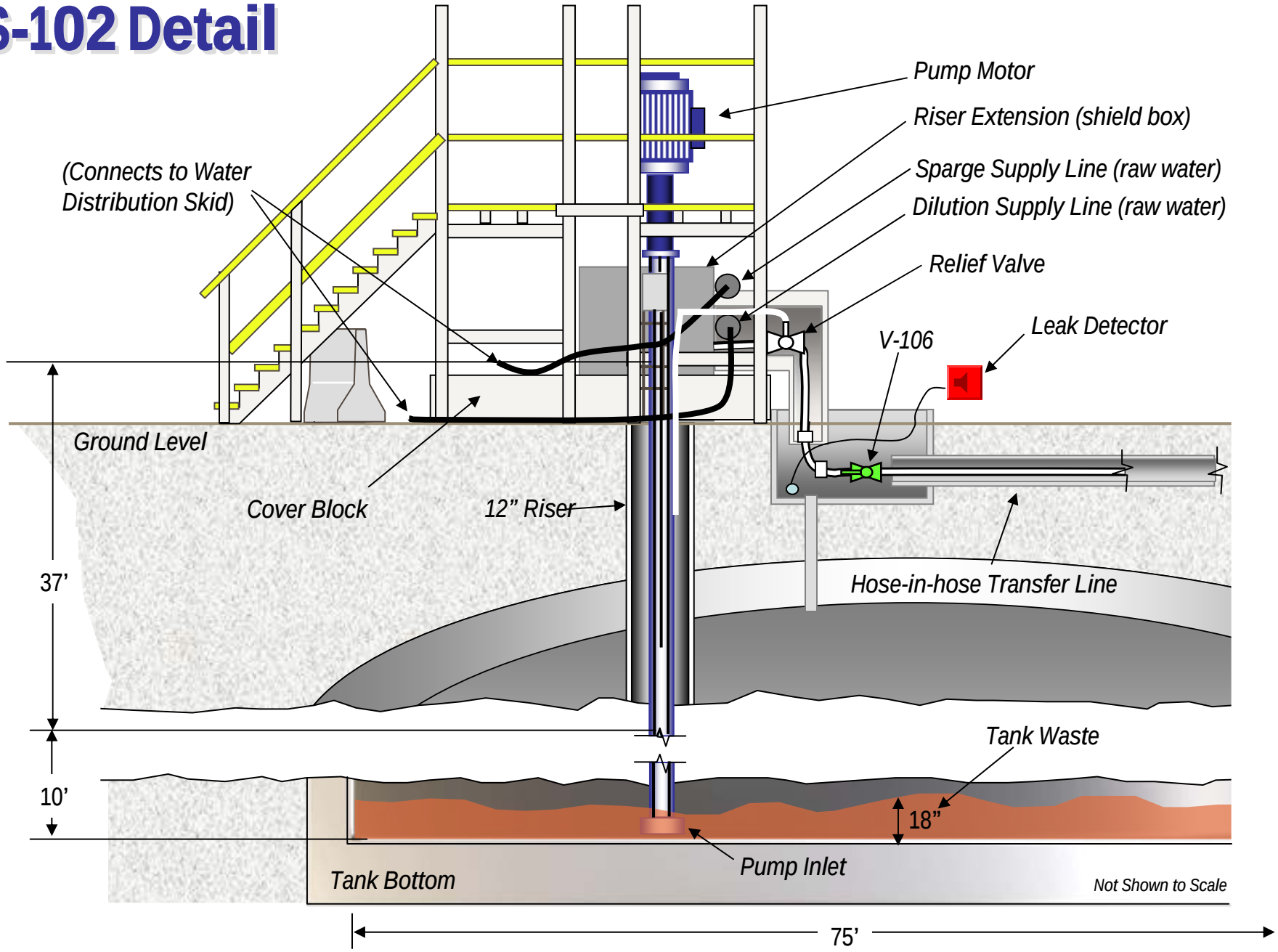


200 Area Tank Farms

S-102 Estimated Major Constituents as of 1 Aug 2007

Constituent	Mass (kg)	Percent of total
Nitrate	26925	28.11%
Sodium	24150	25.21%
Aluminum	19725	20.59%
Phosphate	6143	6.41%
Nitrite	5948	6.21%
Uranium	3278	3.42%
Oxalate	2745	2.87%
Carbonate	2430	2.54%
Total organic carbon	1065	1.11%
Other	3390	3.54%
Total	95798	100.00%

S-102 Detail

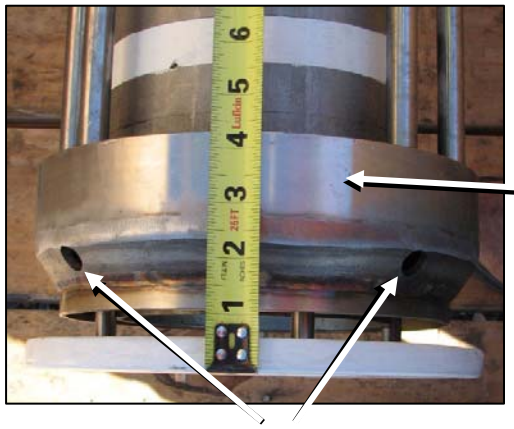

Pump Motor
Riser Extension (shield box)
Sparge Supply Line (raw water)
Dilution Supply Line (raw water)
Relief Valve
Leak Detector
V-106
(Connects to Water Distribution Skid)
Ground Level
Cover Block
12" Riser
Hose-in-hose Transfer Line
37'
10'
Tank Waste
18"
Pump Inlet
Tank Bottom
Not Shown to Scale
75'

Pump Inlet Nozzle

Pump Rotor

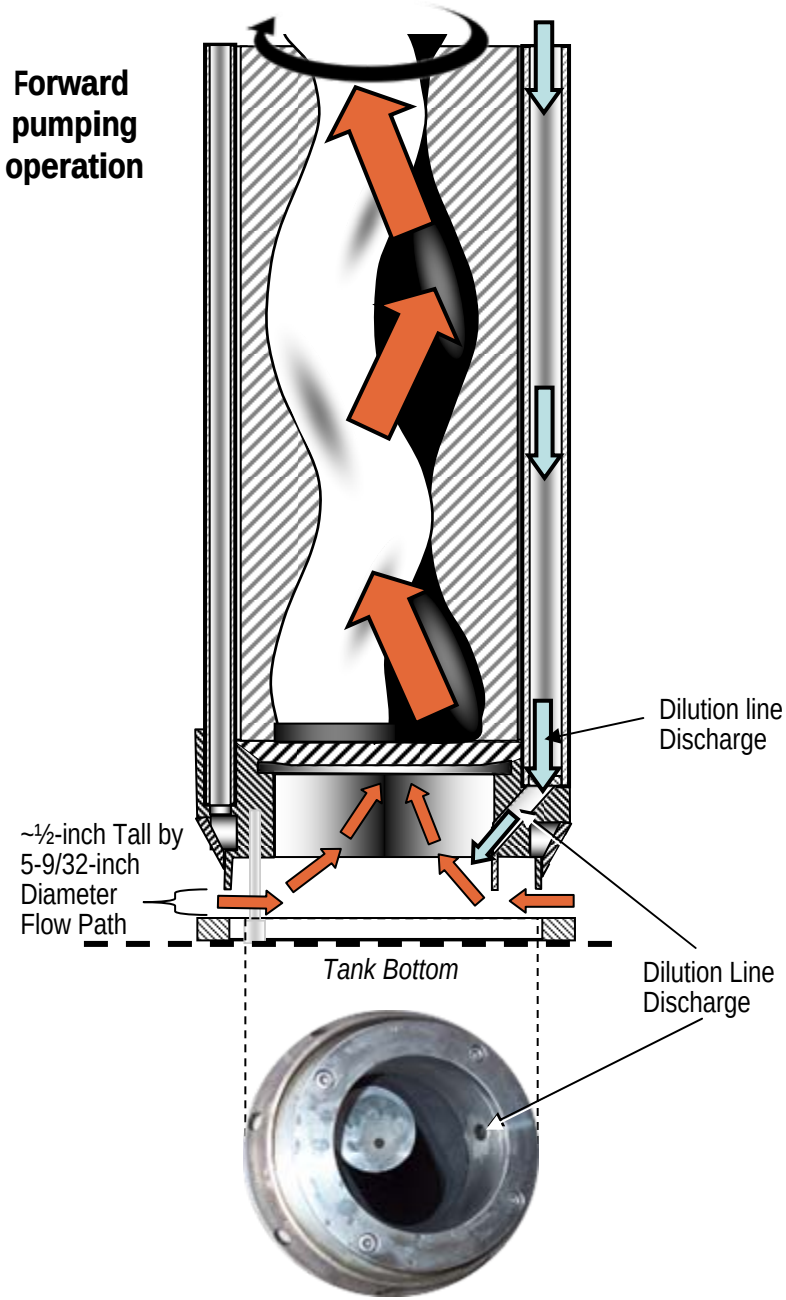
Dilution Water Supply Port

About 1 cubic foot of Gibsite settles to pump outlet after motor trip

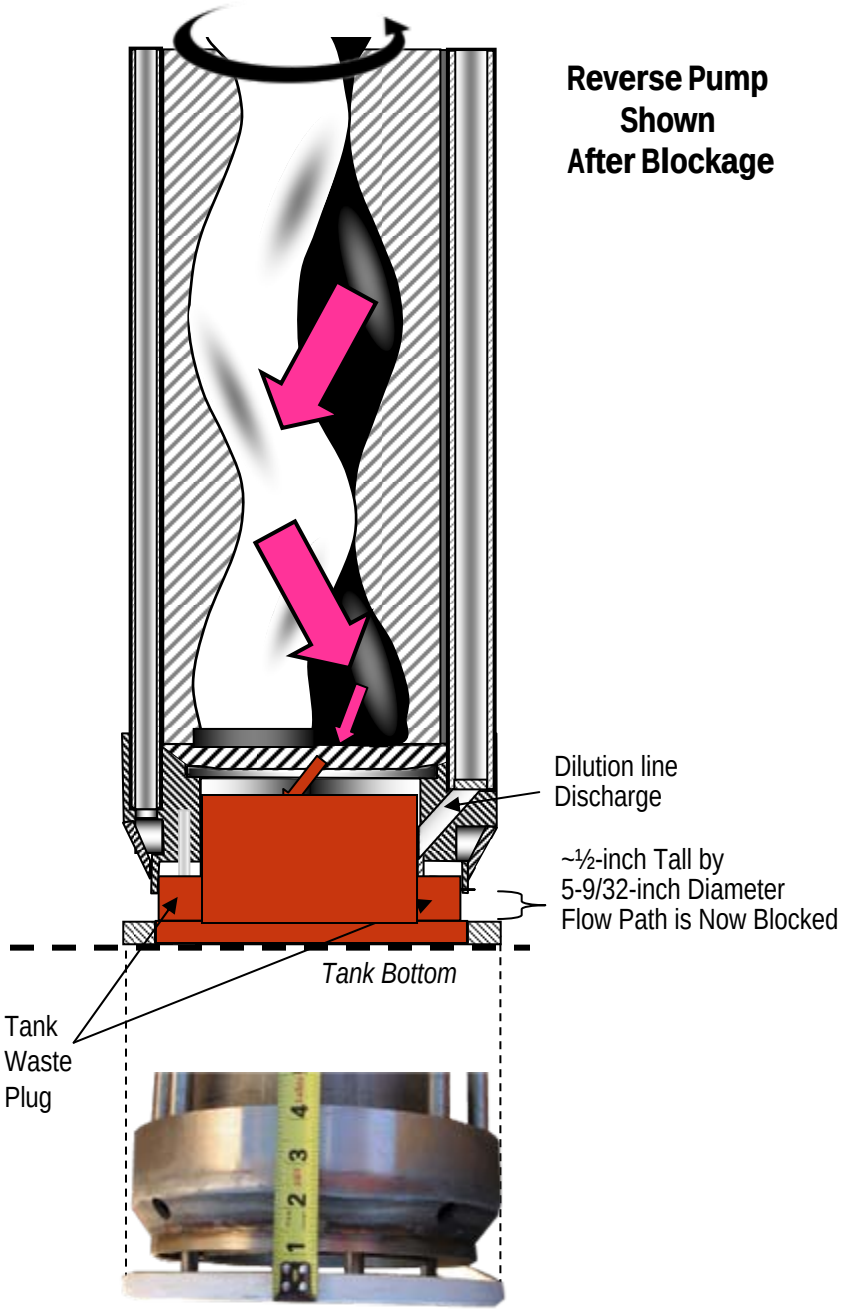


Sparge Ring

Sparge Water/Air Distribution Ports



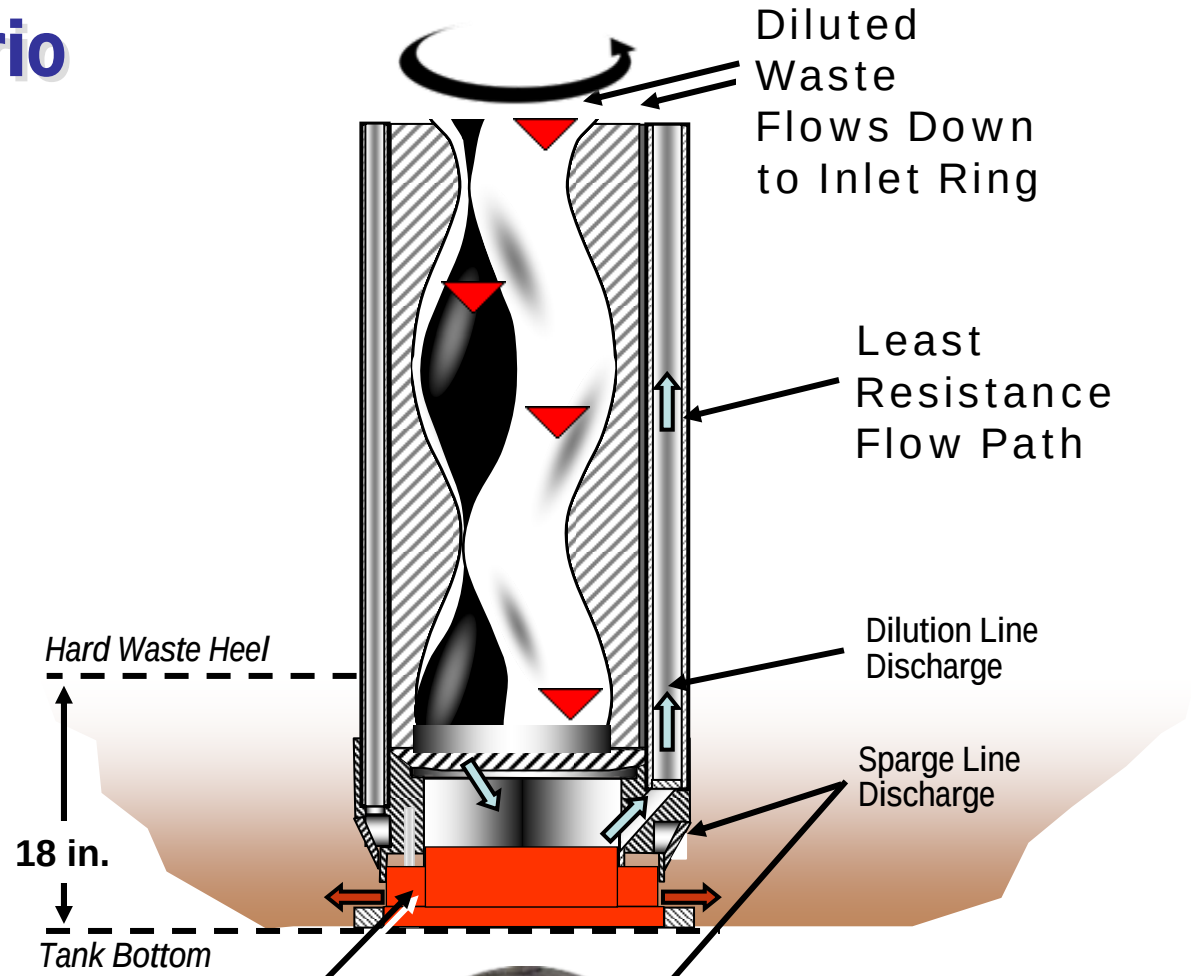
End Photo of Pump Inlet



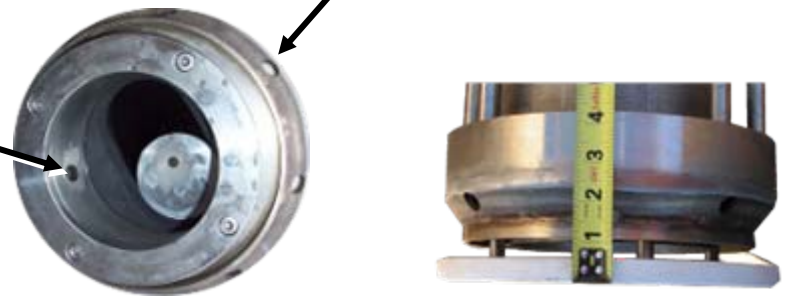
Side-View Photo of Pump Inlet

Confirmed Scenario

With the pump suction plate within ½ - 1” from the tank floor, the dilatant (shear thickening) waste heel was sufficient strong to block the free flow of relatively low density (Specific Gravity of about 1.1) of flushed transfer waste during reverse rotation and subsequently pressurized the suction cavity of the pump to about 400psi. When the sludge level lowered below the Dilution Line discharge the pressure was transmitted up the dilution line, rupturing the above ground hose, and allowing dilute waste to spray and spill to the ground.

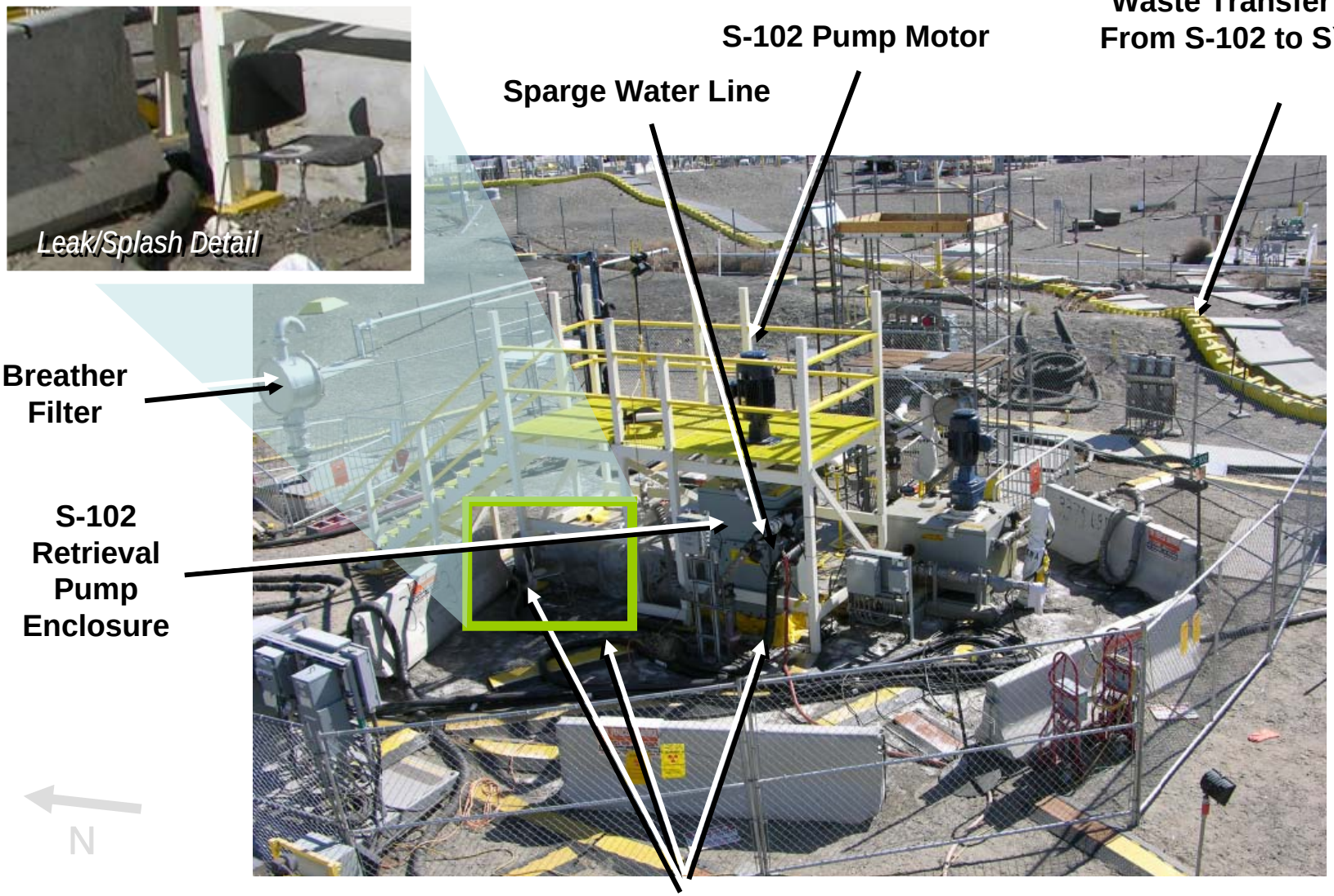


~½-inch Tall by 5-9/32-inch Annular Flow Path is Blocked along with bottom opening (near tank floor) forcing dilute waste up Dilution Line

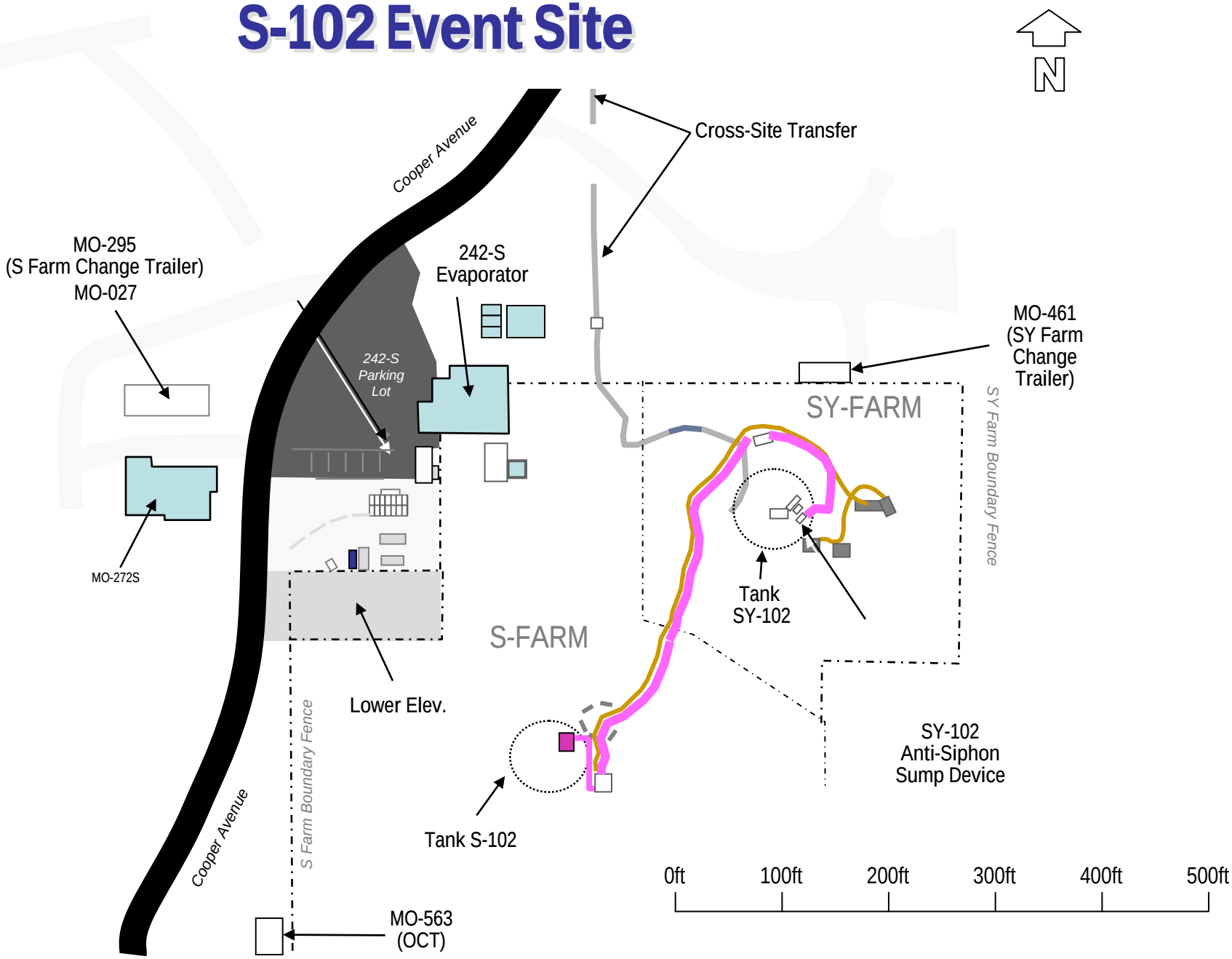


Bottom and Side-View Photo of Pump Inlet

S-102 Event Site Overview



S-102 Event Site



Spill Area

Sludge Waste Removal and Tank Closure

Noel Chapman
F-Tank Farm Closure Engineering
Washington Savannah River Company
Savannah River Site

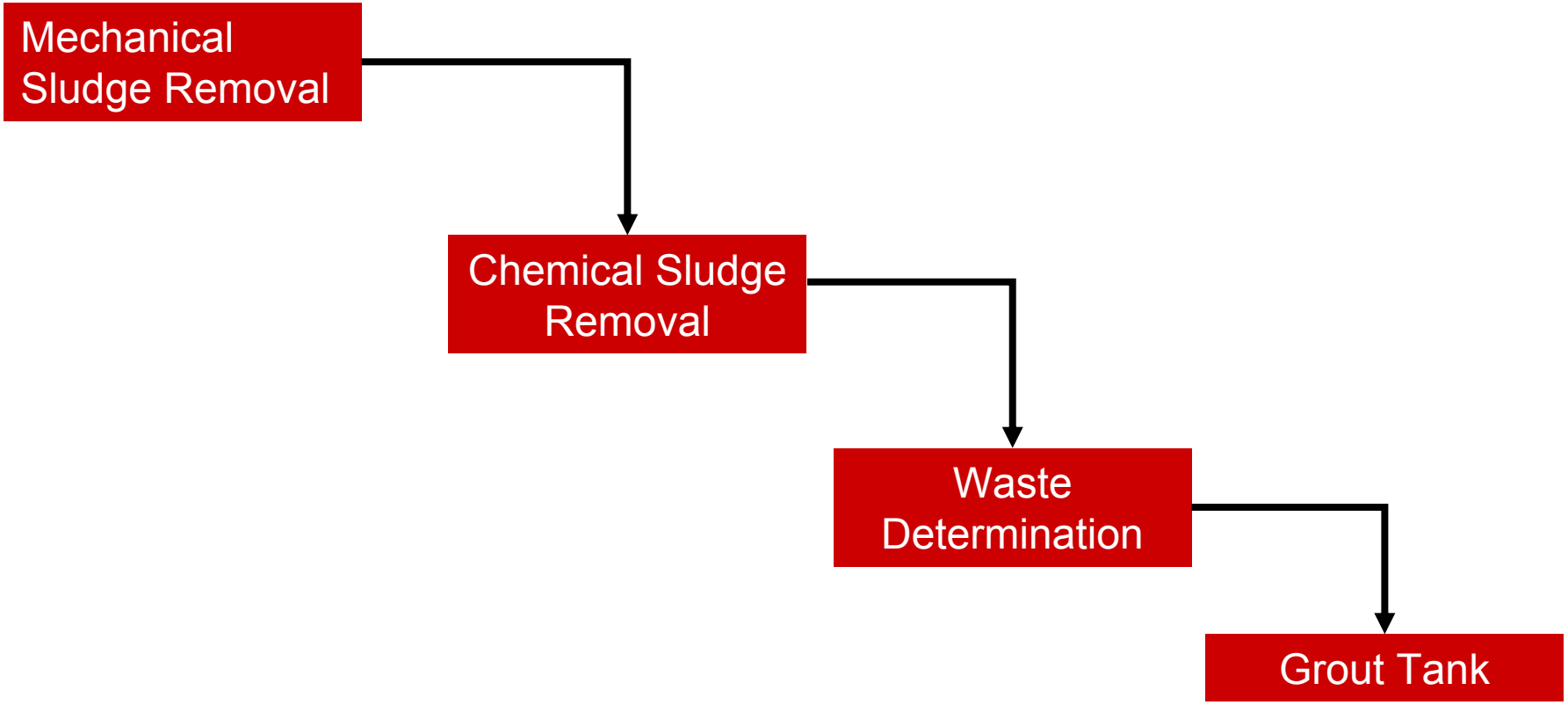
Tank Closure Driver

- Federal Facilities Agreement commitment to close two tanks by September, 2010

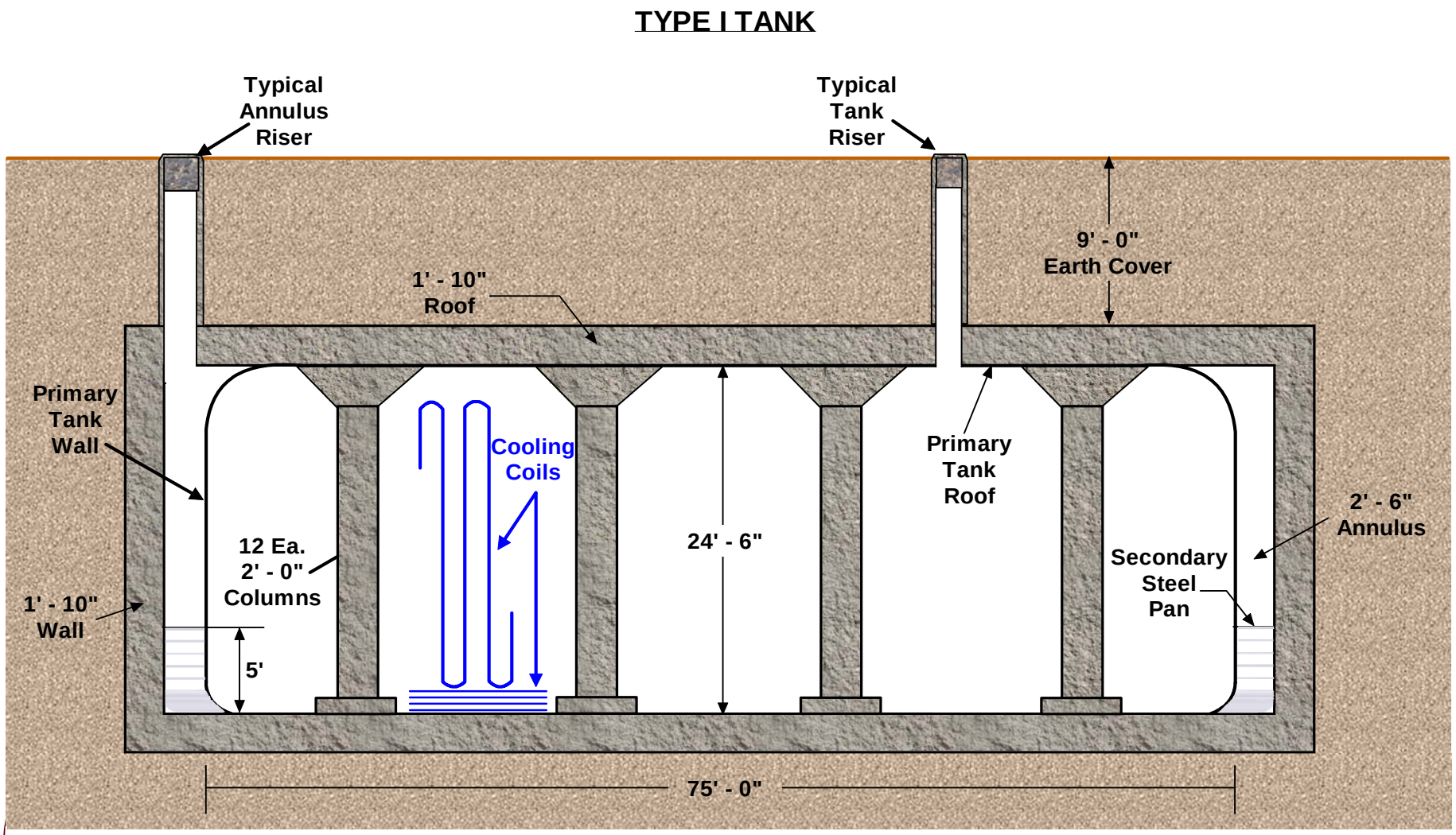
- Tanks selected for closure are Tanks 5 and 6 in F Tank Farm

A.3.58

Tank Closure Process



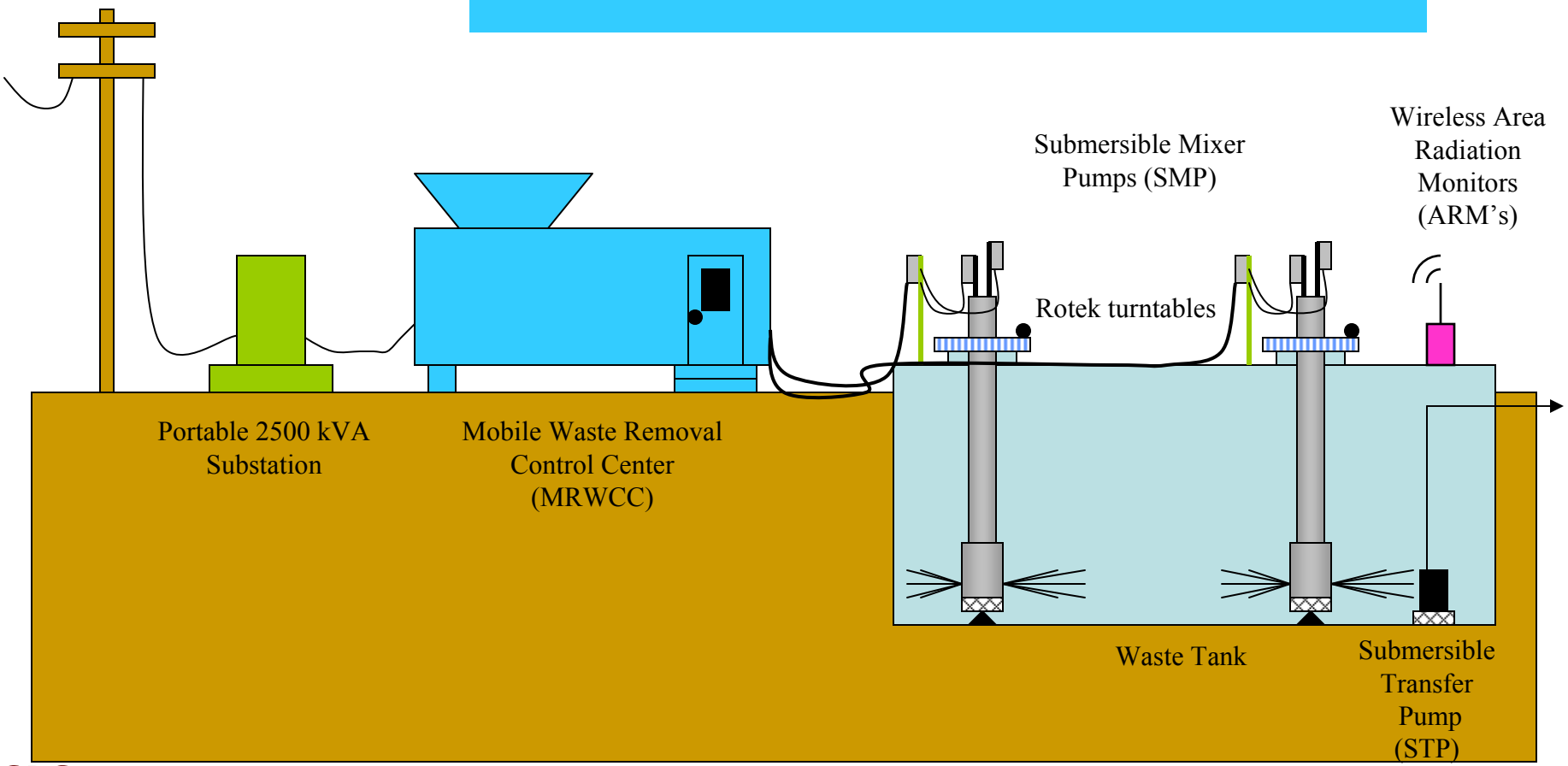
SRS Type I Tank



A.3.60

Mechanical Sludge Removal Utilizing Waste on Wheels (WOW)

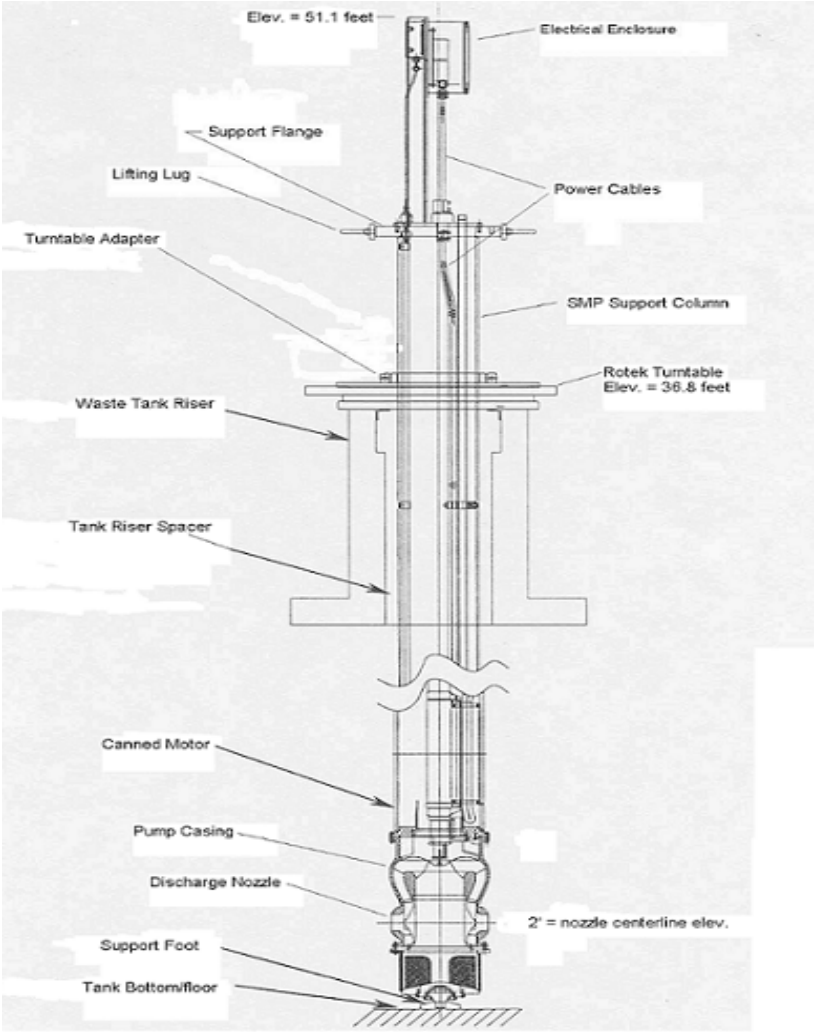
Major WOW Components



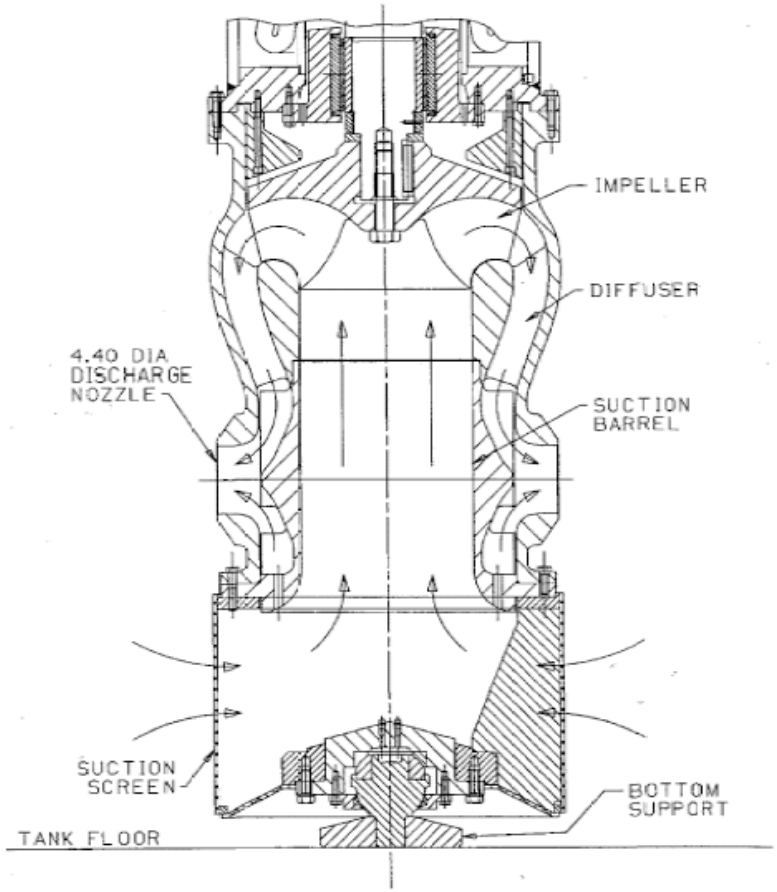
A.3.61

SMP (Submersible Mixer Pump)

- Canned Motor Design
  - Total Flow 7602 gpm
  - Flow Velocity 80.2 ft/s
  - Speed 400 - 1400 rpm
  - Motor 305 Hp
  - Torque 1226 ft-lbs
  - Weight 9600 lbs
- Variable Speed
- Product Cooled
- Floor Supported or Hung
- 51 ft. length
- 18” Pipe Column
- Rotated by Turntable



SMP With Screen and Foot Bearing



A.3.63

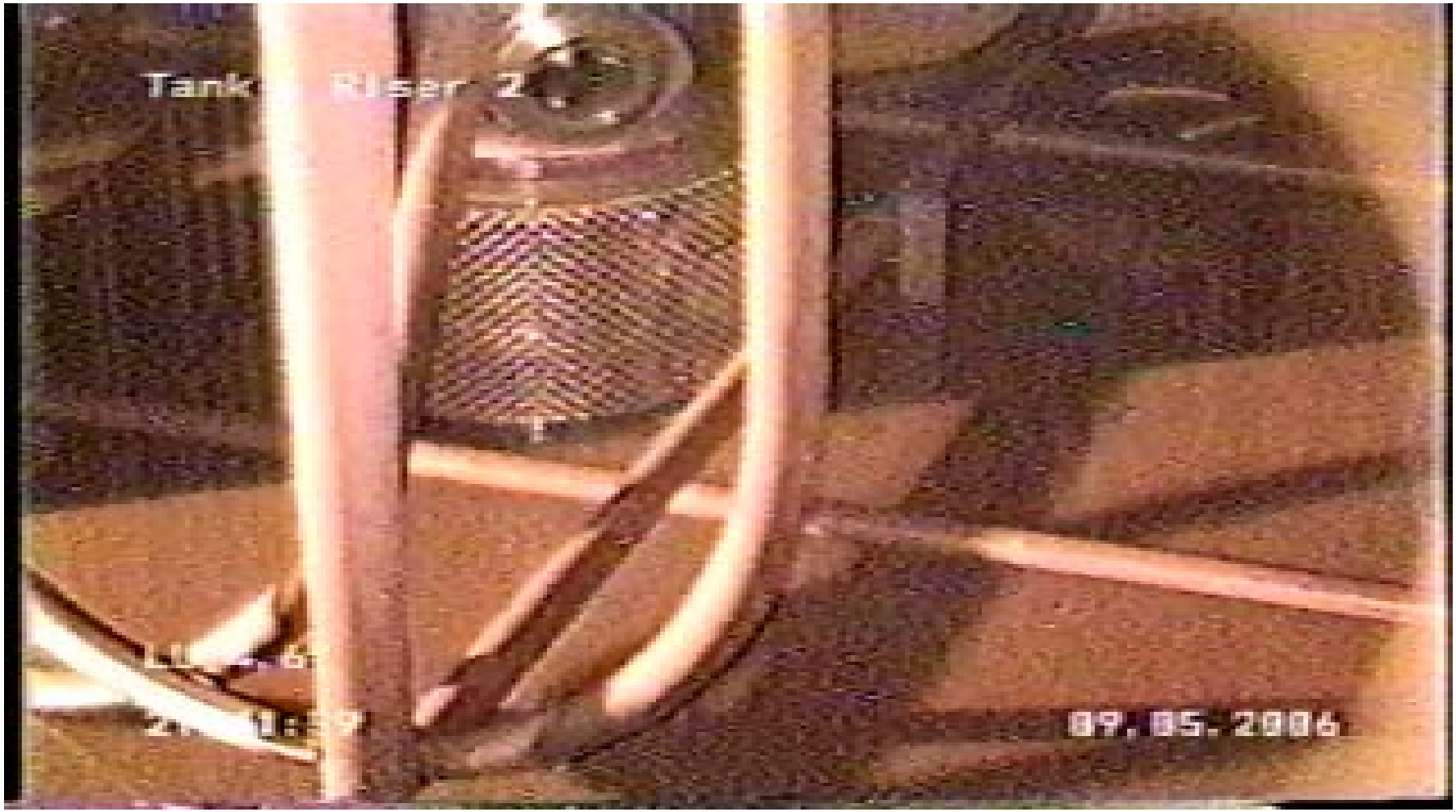
SMP Installation Photos

SMP Installation Photos

Tank 5, Riser 3 SMP Placement

A.3.64

Tank 6 SMP in Riser 3

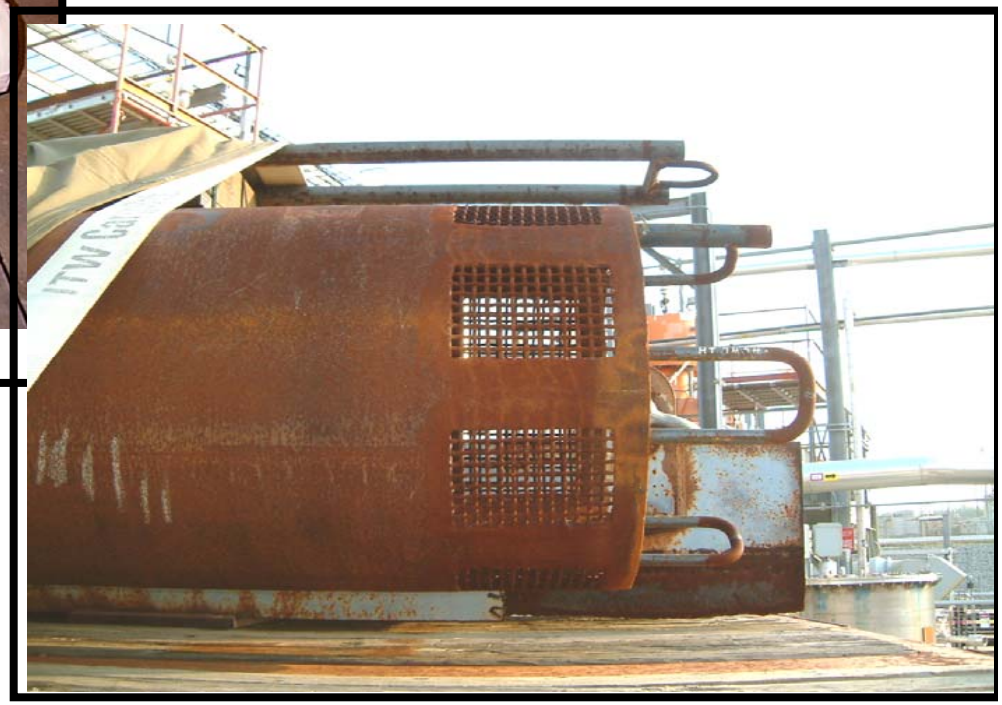


A.3.65

STP/Caisson Details

STP Motor/Pump Assembly

STP Caisson



A.3.66

Typical Mechanical Sludge Removal Process

Start Mixing w/ Liquid level @ 10 Feet (120”)

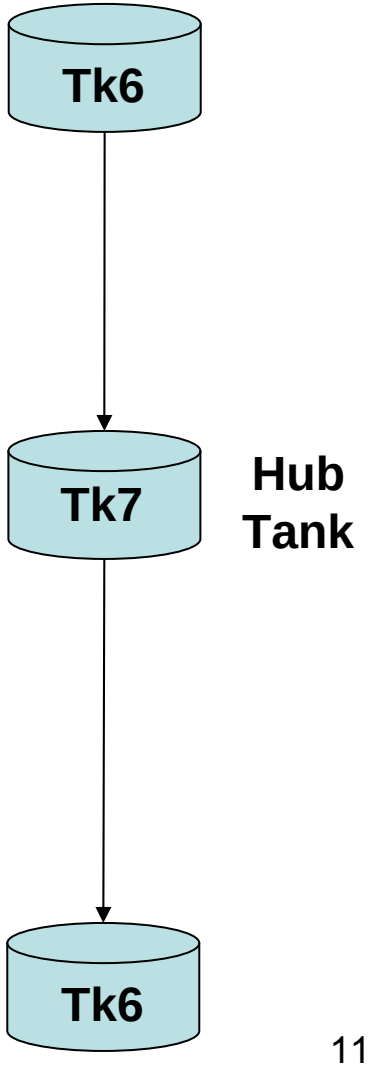
Operate SMPs in Index and Oscillation Mode

Transfer Slurry to Tank 7

- Continue Mixing until LL = ~ 3 Feet.
- Sludge Mapping begins at ~28 inches
- Transfer Ends w/ LL = ~ 3 inches

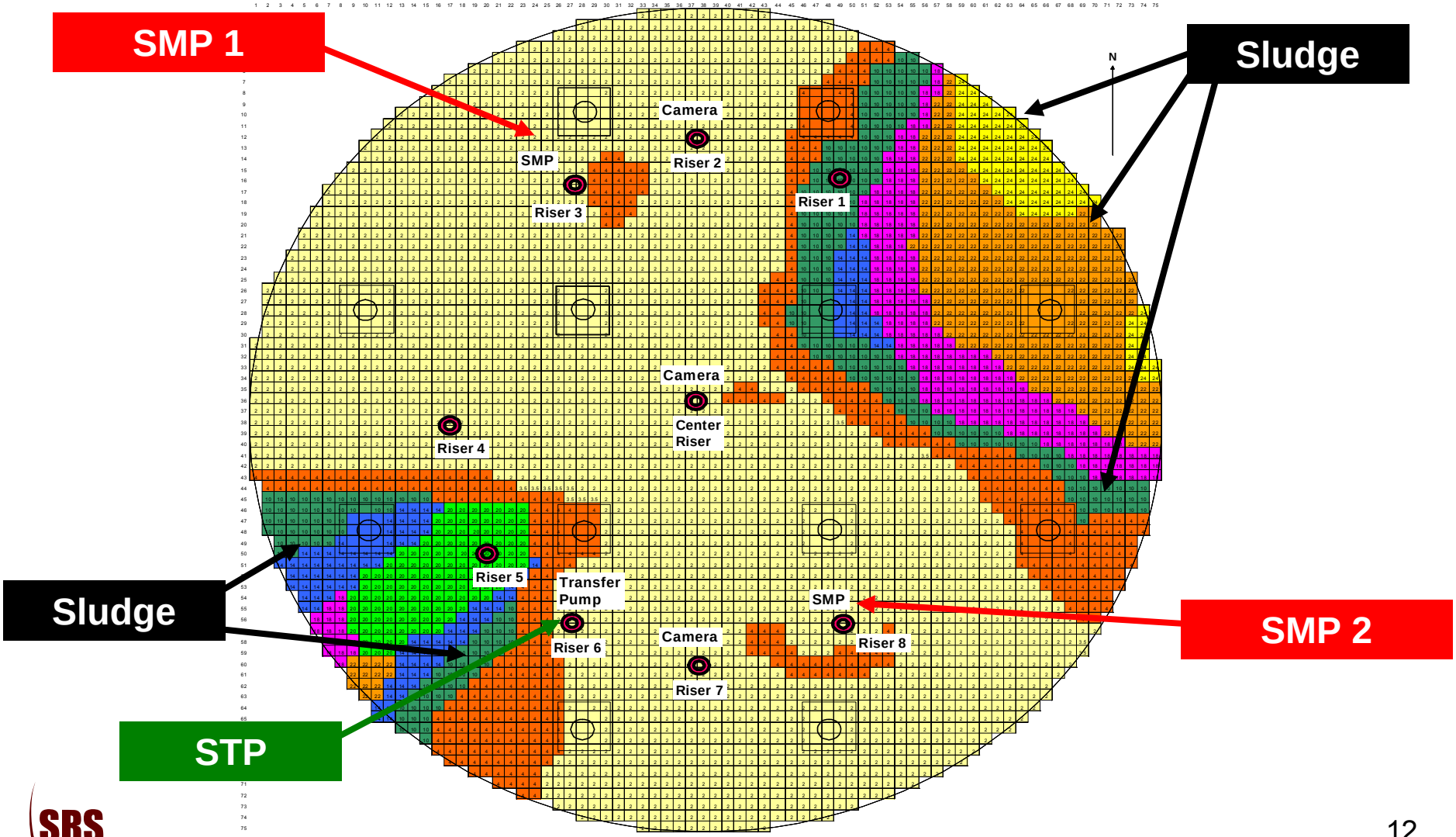
Solids Settle in Tank 7

Transfer Supernate Back to Tank 6



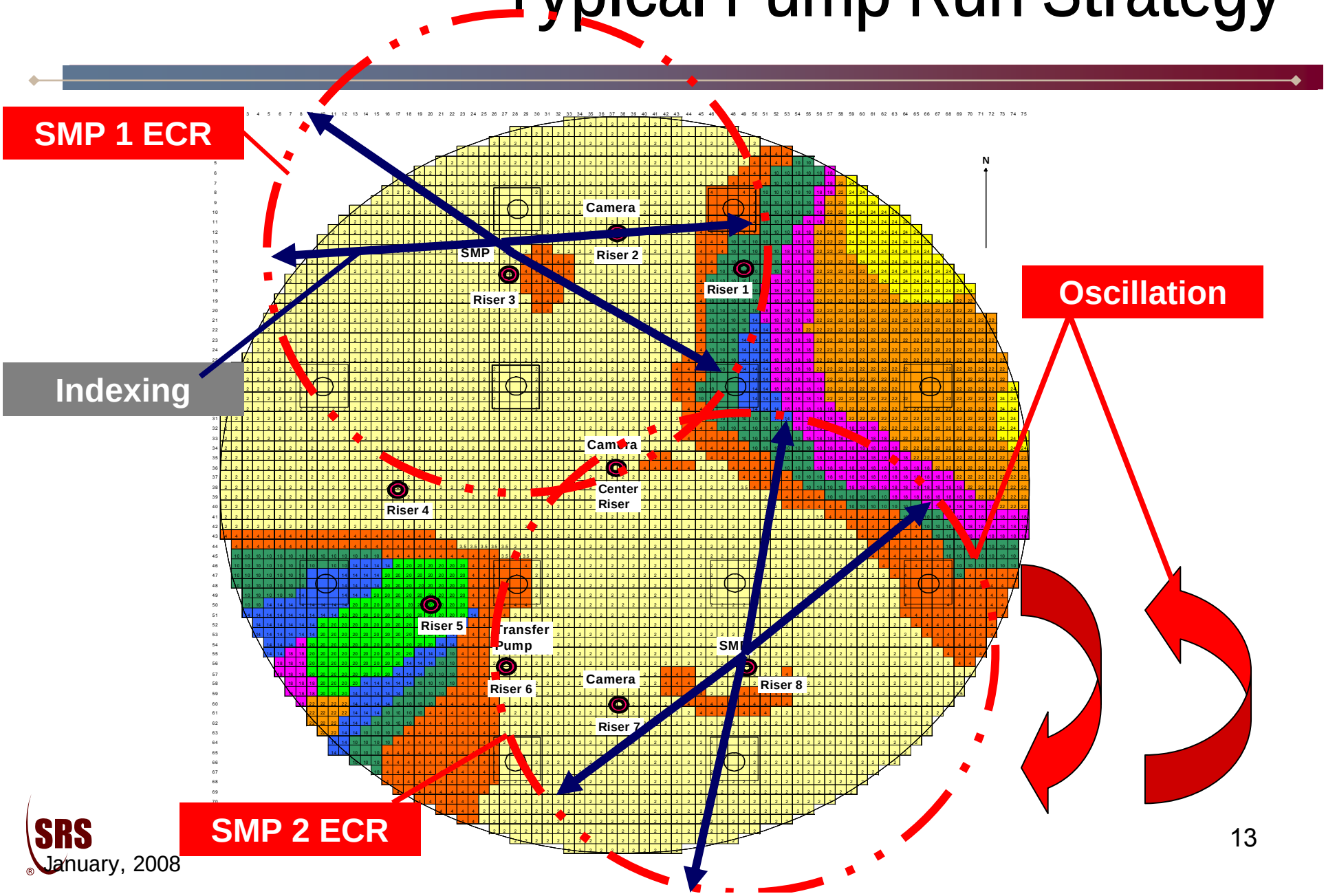
A.3.67

Tank 6 Beginning MSR 7/9/2006 (24932 gals)



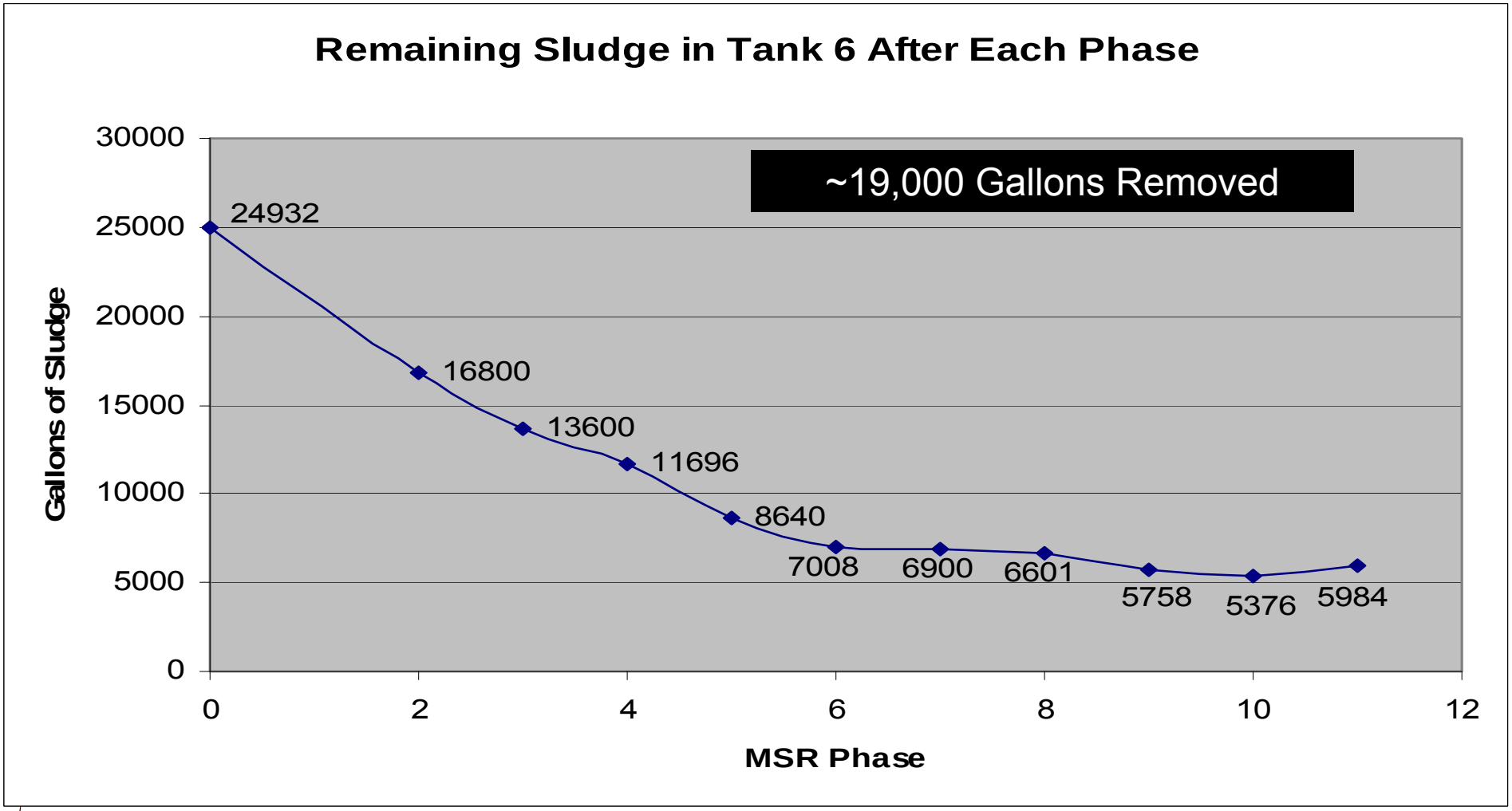
A.3.68

Typical Pump Run Strategy



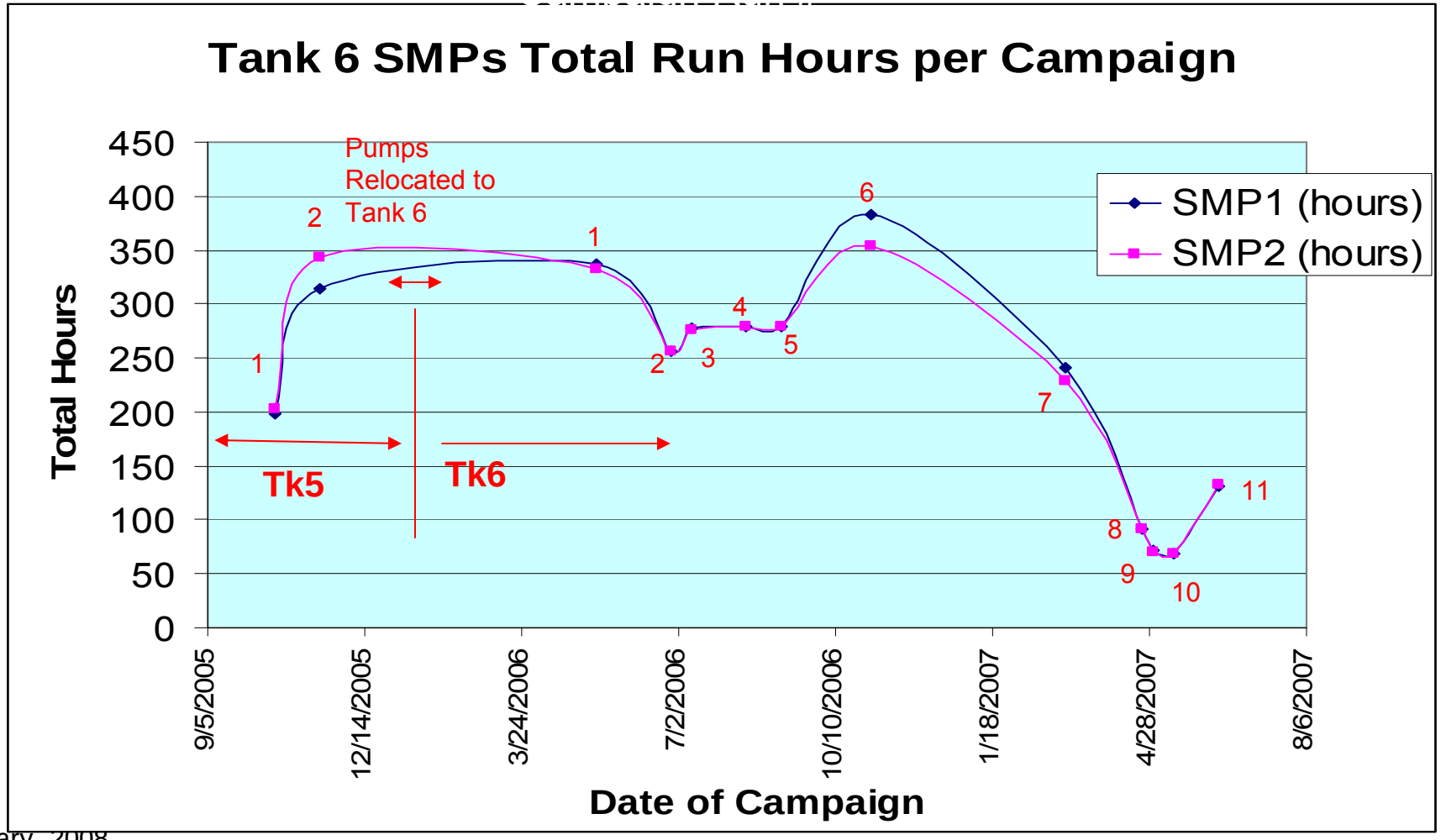
A.3.69

Tank 6 Sludge Volume

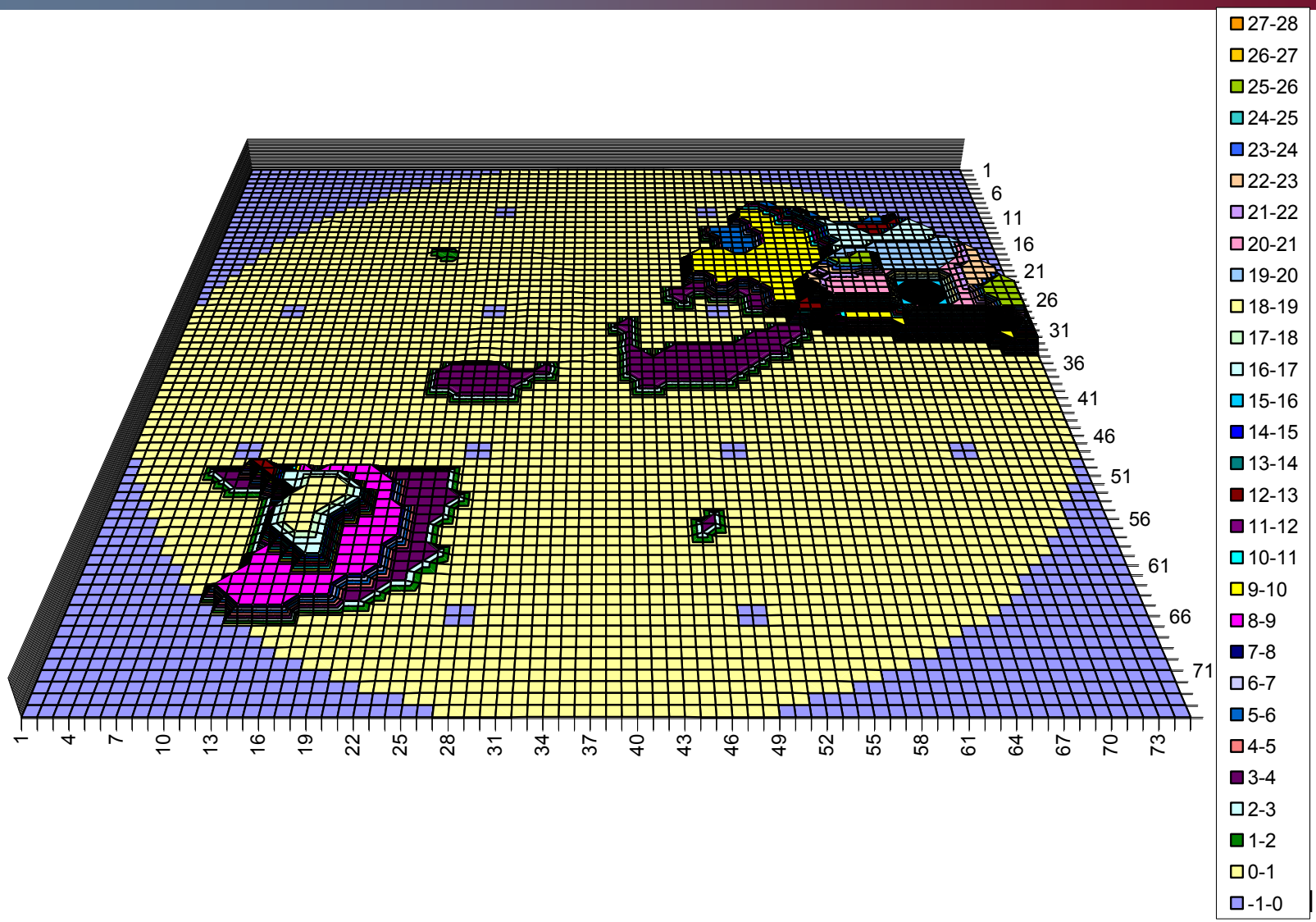


A.3.70

SMP Hours of Operation



Tank 6 Today (5984 Gallons)



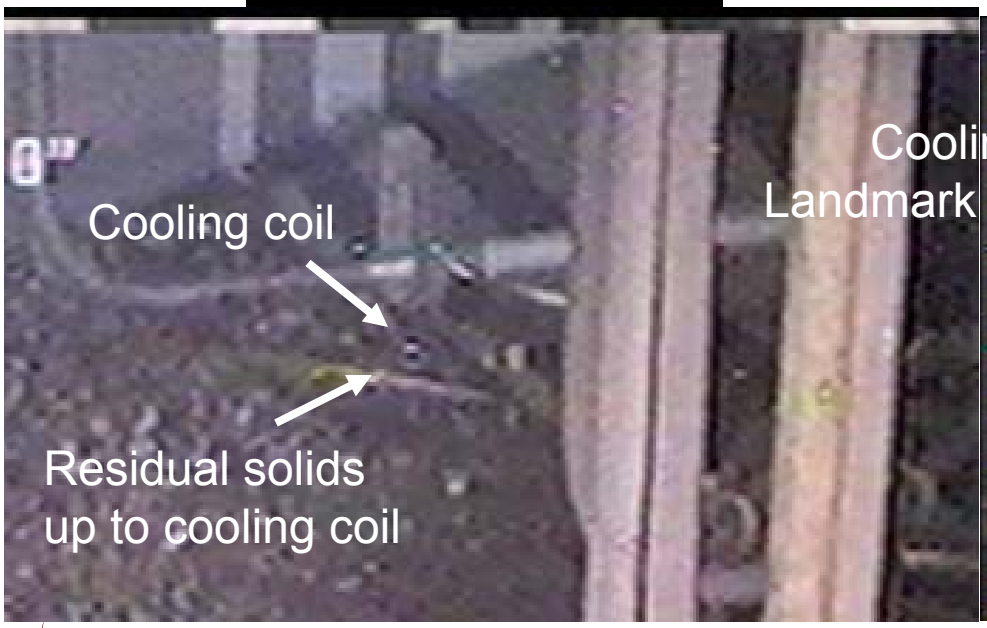
Tank 6 Northeast Mound

After Phase 6 (12/8/06)

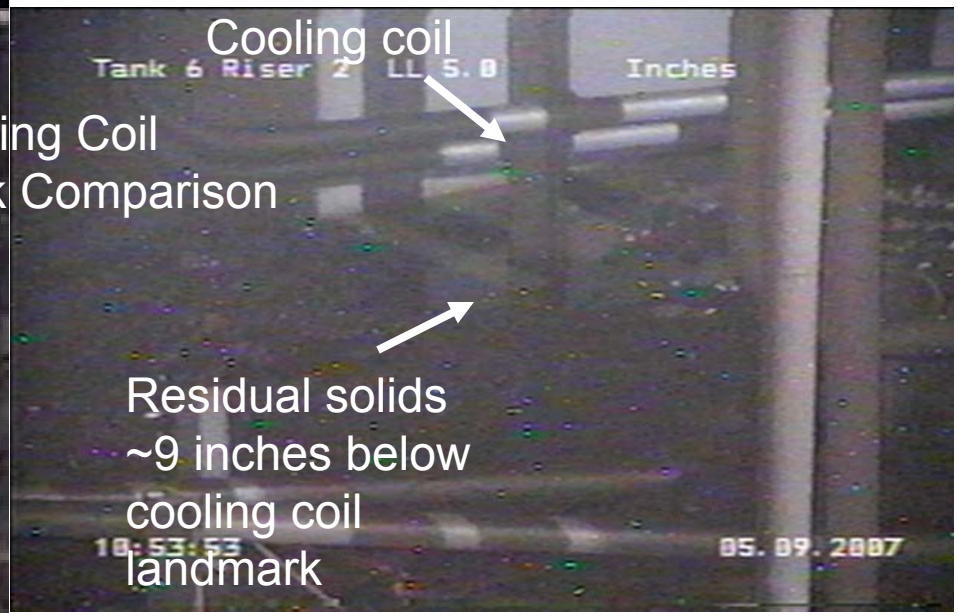
- Residual solids level ~22 inches.
- NE mound volume ~5,200 gallons.

After Phase 9 (5/9/07)

- Residual solids level ~13 inches.
- NE mound volume ~4,000 gallons.



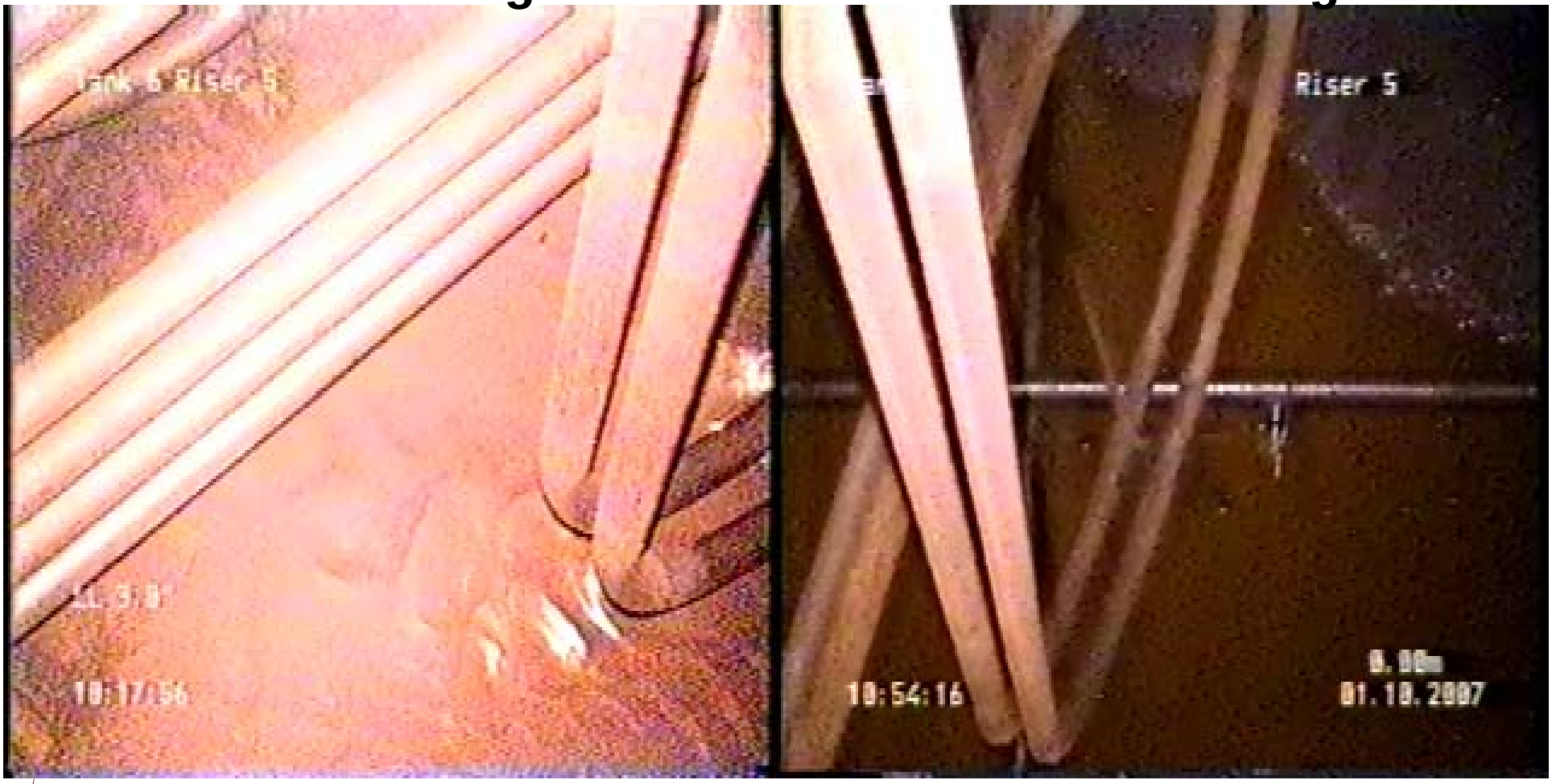
Cooling Coil
Landmark Comparison



A.3.73

Tank 6 Riser 5

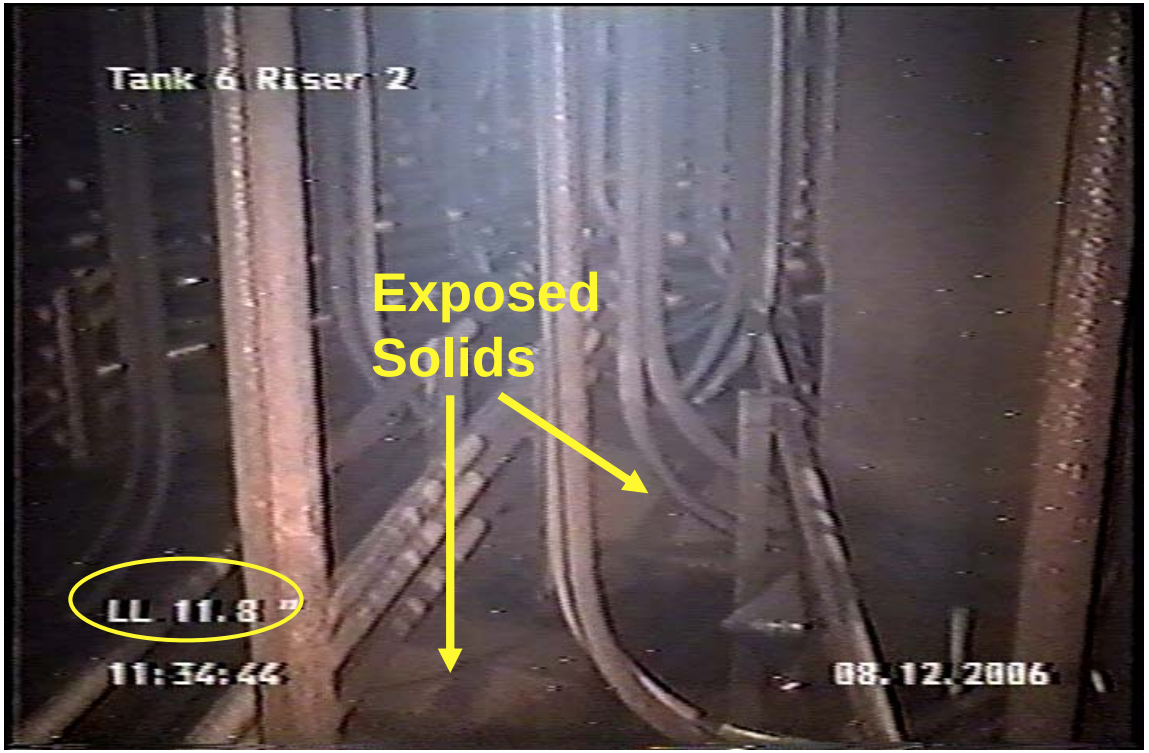
Before Lancing **After Lancing**



A.3.74

Tank 6 Riser 2
Exposed Solids
LL 18.1"
09:48:27
08.12.2006

Tank 6 Riser 2
Exposed Solids
LL 14.7"
10:35:05
08.12.2006


Tank 6 Riser 2
Exposed Solids
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A.3.75

Mechanical Sludge Removal Lessons Learned

- Cooling Coil routing/congestion created areas of tank which hindered slurry suspension
- Interferences reduce SMP Effective Cleaning Radius (ECR) from 53 feet to approximately 29 feet

A.3.76

Chemical Cleaning of FTF Type 1 Tanks

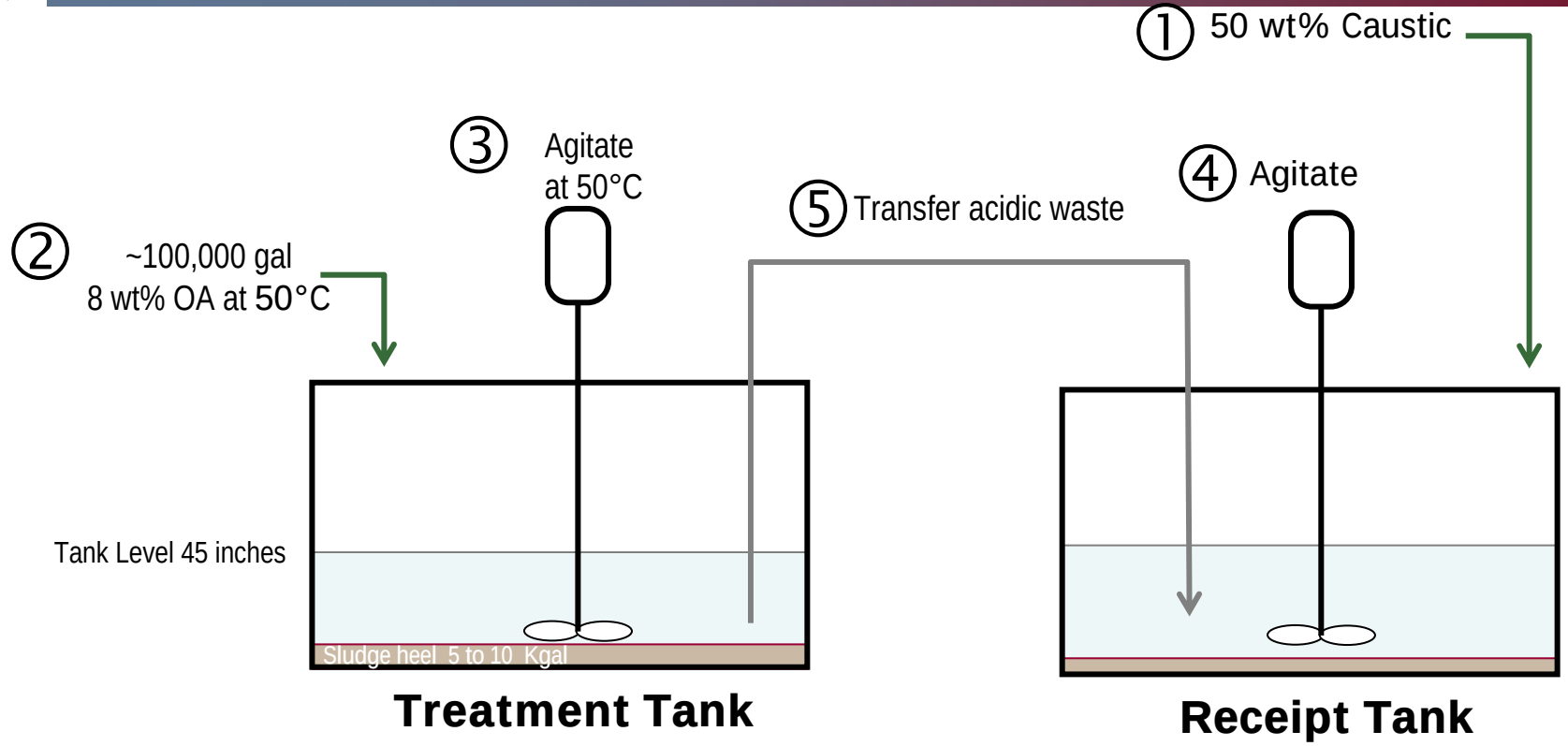
- **SRNL testing on actual waste and simulant**
  - **Tests executed at 25, 50, and 75 degrees C in two modes of operation – mixed and unmixed**
  - **Bounding hydrogen generation rate determined based on corrosion of carbon steel**
  - **Total gas generation rate measured**
  - **Corrosion rate of carbon steel quantified**
  - **Neutralization of acidic waste in receipt tank demonstrated**
  - **Dissolution efficiency demonstrated**

A.3.77

Technical Acceptability Chemical Cleaning

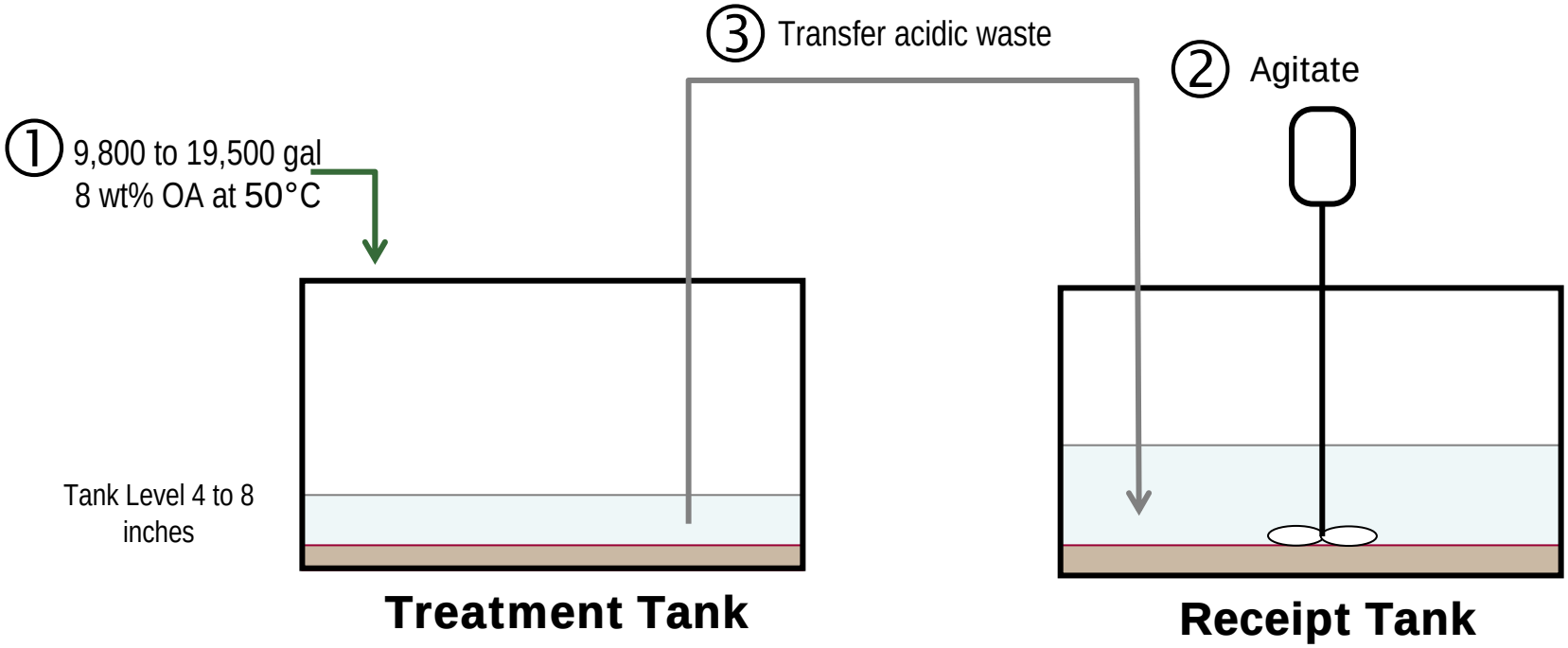
- Dissolution efficiency range from 60 to 99 percent in a first OA strike depending on metal hydroxide.
- H2 generated from both corrosion of carbon steel and radiolysis. Existing tank ventilation system will provide Safety Class function of minimum airflow.
- Tank wall corrosion rates will be on order of 45 to 60 mpy. Corrosion Control Program will define time limitation for OA cleaning to protect waste tank structural integrity.
- Total gas generation rates (CO2 dominates) will require approximately 700 cfm of supplemental exhaust ventilation from the Treatment Tank.
- Treatment Tank and Receipt Tank heating due to chemical reactions manageable with existing cooling systems.
- Materials of Construction in transfer path compatible with OA waste stream.

Planned Process Steps – OA Addition



- Caustic unloaded to Receipt Tank from tankers.
- OA unloaded to Treatment Tank from tankers at 50°C
- SMPs operated in Treatment Tank to aid dissolution and suspension
- Standard Slurry pumps operated in Receipt Tank to enhance neutralization
- Transfer procedures developed per existing Transfer Control Program

Process Steps – Subsequent OA Additions



- OA unloaded to Treatment Tank from tankers at 50°C and allow to soak
- SMPs will not be operated in Treatment Tank
- Heel transferred to Receipt Tank while Receipt Tank is slurried

A.3.80

Status of Chemical Cleaning

- Hazards Analysis and Documented Safety Analysis are approved to allow Chemical Cleaning of FTF Type 1 Tanks
- NCSE is approved and shows criticality is not credible
- Field modifications being implemented to add Oxalic Acid to Tanks 5 and 6
- Chemical cleaning scheduled to begin in April

Questions?

Noel Chapman

LWO F Tank Farm Closure Deputy Project Manager

Building 704-70F, Savannah River Site

Office Phone (803)952-3806

noel.chapman@srs.gov

SRNL Work to Support the Removal of Slurries from Waste Tanks, the Transport of Slurries in Pipelines, and the Separation of Solids from Liquids at SRS

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SAVANNAH RIVER NATIONAL LABORATORY
We Put Science To Work

Michael R. Poirier

January 16, 2008

Introduction

- Savannah River Site (SRS) liquid waste contains slurries composed of solid particles and salt solution
- Effective processing of these slurries important to Tank Farm and DWPF operation
- Slurry processes include mixing, retrieval, transport, and solid-liquid separations
- Talk will discuss SRNL experience in testing and recommending processing methods for these slurries

SRNL

Slurry Mixing (Tank Farm Processes)

- Occurs in waste tanks (~1 million gallons) and process tanks (1,000 – 30,000 gallons)
- Waste tanks generally mixed with slurry pumps (jets), spargers
- Process tanks generally mixed with agitators, spargers

SRNL

Approaches to Recommending Mixing Equipment and Operating Parameters

- Tank Farm Experience
  - Limited data
  - Anecdotal
- SRNL Testing
  - Simulant rather than actual waste
- Computational Fluid Dynamics (CFD)
- Other DOE Site Testing
- Technical Literature

SRNL

SRNL Testing

- Evaluated mixing of Bingham plastic slurries in a 85 ft diameter tank
  - Developed model of effective cleaning radius
- Testing to evaluate resuspension of sludge/MST slurries after sitting for 6 weeks at elevated temperature
  - Able to resuspend high yield strength slurries even though cavern model predicted they would not be resuspended
  - Fraction resuspended was a function of time
  - Yield stress was a function of setting time
- Measure rheology (yield stress) of SRS slurries

SRNL

Waste Retrieval

- Bulk sludge removal
  - Mix sludge with 1 – 4 slurry pumps and transfer to downstream process
- Sludge Heel Removal
  - Remove sludge heels from waste tanks prior to closure
    - Slurry pumps
    - Flygt mixers
    - Chemical Cleaning (oxalic acid dissolution)
    - "New Technologies"
- Salt Removal
  - Add "water", dissolve salt with or without agitation, and transfer to downstream tank

SRNL

Waste Retrieval Testing and Demonstrations

- Flygt mixers to remove heels in sludge tanks
  - Testing performed at 4 scales
  - 1.5 ft, 6 ft, 18 ft, and 85 ft diameter
  - Different scaling for sludge and zeolite
- Waste Tank Salt Removal Demonstration
- Chemical Cleaning Demonstration
  - Laboratory-scale testing of sludge heel dissolution using oxalic acid
  - Actual waste and simulant

SRNL

Slurry Transport

- Need to pump slurries between waste tanks, process tanks, and facilities
- Concentrated sludge slurries
  - 12 – 18 wt % solids
- Dilute slurries
  - < 5 wt % solids

SRNL

Slurry Transport Testing

- 1980s
- Georgia Iron Works (Chris Randall, Dave Lewis)
- TNX (John Fazio)
  - 500 foot, 3 inch stainless steel pipe
- Feed concentrated slurry (Bingham plastic)
- Measured pressure drop as a function of flow rate and rheological properties
  - Compared with Hanks model – good agreement
- Resuspension Tests
  - Allowed to sit for 7 days
  - Flow immediately established when pump started
- Flushing Tests
  - 2 – 3 line volumes needed to flush transfer line

SRNL

Slurry Transport Calculations

- Fast-settling or slow settling
- Fast-settling slurries
  - Use correlations by Durand, Wasp, Turian, Walton, others to calculate minimum transport velocity
- Slow settling slurries
  - If Bingham plastic, use Hanks model
- Calculate needed flow rate, pump discharge pressure by both approaches

SRNL

Solid-Liquid Separation

- Needed for many processes
  - Salt Waste Processing Facility
  - Actinide Removal Process
  - Enhanced Processes for Radionuclide Removal
  - Small Column Ion Exchange
  - Effluent Treatment Facility
  - Sludge Washing
- Impacts process throughput
- Technologies investigated include filtration, centrifugation, flocculation, settling-decanting

SRNL

Solid-Liquid Separation Technology Assessments

- Alternative Filters for ETF (Georgeton and Poirier)
  - Pilot-scale
  - Rotary filter, tubular ultrafilter, and ceramic microfilter performed best
- Solid-Liquid Separation Technologies for DOE Applications (McCabe)
  - Paper study
  - Tubular crossflow filter recommended technology
  - Testing needed
  - Baseline in many SRS processes
- Solid-Liquid Separation Technologies for SRS Salt Processing (Poirier and Fink)
  - Paper study, bench-scale testing, pilot-scale testing
  - Identified rotary filter as plausible technology
- Evaluation of Alternative Filter Membrane media (with INL)
  - Bench-scale Testing
  - Ceramic membranes produced higher flux than stainless steel membranes

SRNL

Filter Testing

- Tubular crossflow filter
  - Bench-scale with simulant
  - Bench-scale with actual waste (SRS and Hanford)
  - Pilot-scale with simulant
  - Obtained design data
  - Evaluated alternative filter media
- Rotary microfilter
  - Bench-scale with actual waste
  - Pilot-scale with simulant
  - Full-scale with simulant
  - Evaluating new technology

SRNL

Lessons Learned

- Pore size dependent on manufacturer
- Filter flux
  - Polymer > ceramic > stainless steel
- Need to test to select best pore size
- Many commercially available flocculants not suitable for high pH, high ionic strength material in SRS Tank Farm
- Centrifuge not effective at separating particles typical of SRS waste
- Flocculation + settling-decanting could remove fraction of the solids, but would need polishing filter

SRNL

SRS savannah river site

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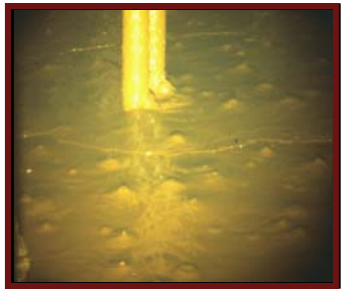
We Put Science To Work

Overview of the Design Basis, Operating Experience, and the Application of Laboratory Data to Support the Transport and Mixing of Sludge Slurries in the Defense Waste Processing Facility



J.M. BRICKER
J.R. NEUVILLE
T.L. FELLINGER
Defense Waste Processing Facility

E.K. HANSEN
Savannah River National Laboratory

January 16, 2008

Agenda

- Overview of operations: Defense Waste Processing Facility (DWPF)
- Savannah River National Laboratory (SRNL) characterization techniques
  - Chemical and physical characterization
  - Recent efforts
- Modeling efforts
- Identification of unresolved issues
- Conclusions

A.3.98

DWPF: Operations Overview

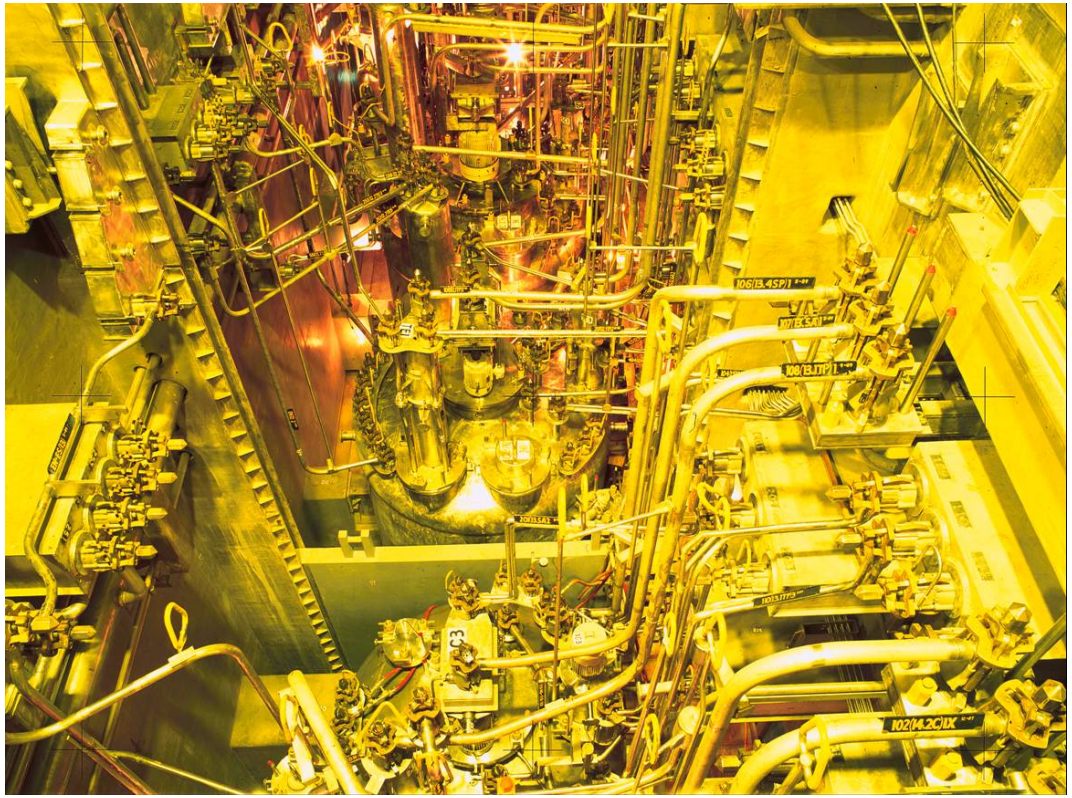
Design purpose is to process and vitrify high level waste for safe, permanent geological storage

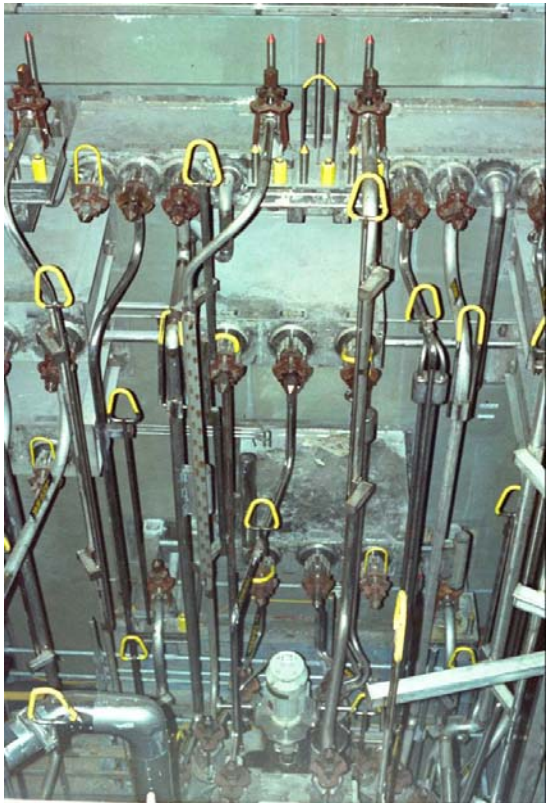
- Receipt and chemical adjustment/concentration
- Frit addition
- Melter feed and canister pouring

A.3.99

DWPF: Transport Issues

Complex canyon configuration complicates transport issues





A.3.100

DWPF: Transport Issues

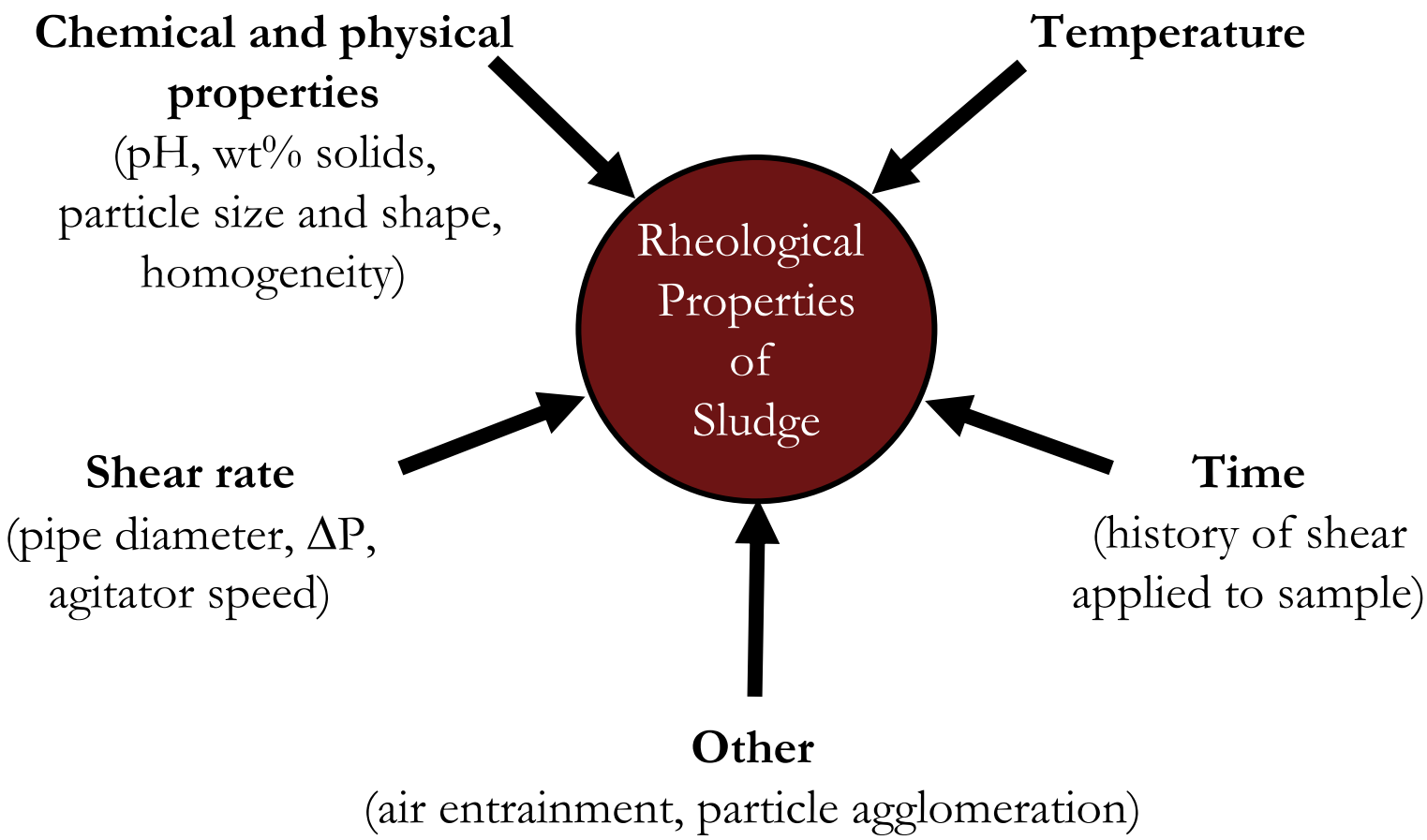
- Chemical and physical properties of sludge differ significantly among batches
  - HM sludge → high Aluminum
  - Purex sludge → high Iron

A.3.101

Sludge Batch	Problem Type	Problem Description
SB1a	Chemical	Inability to destroy nitrite
SB1b	Rheological	Sludge slurry exhibited tacky nature
SB2	Rheological	Sludge exhibited excessive air entrainment/foaminess
SB3	Chemical	Greater quantities of off-gassing as a result of higher anion concentrations
SB4	Chemical/Rheological	Greater quantities of off-gassing as a result of higher anion concentrations; melter feed pump issues

DWPF: Transport Issues

Factors that Influence Sludge Rheology



A.3.102

Tools Available

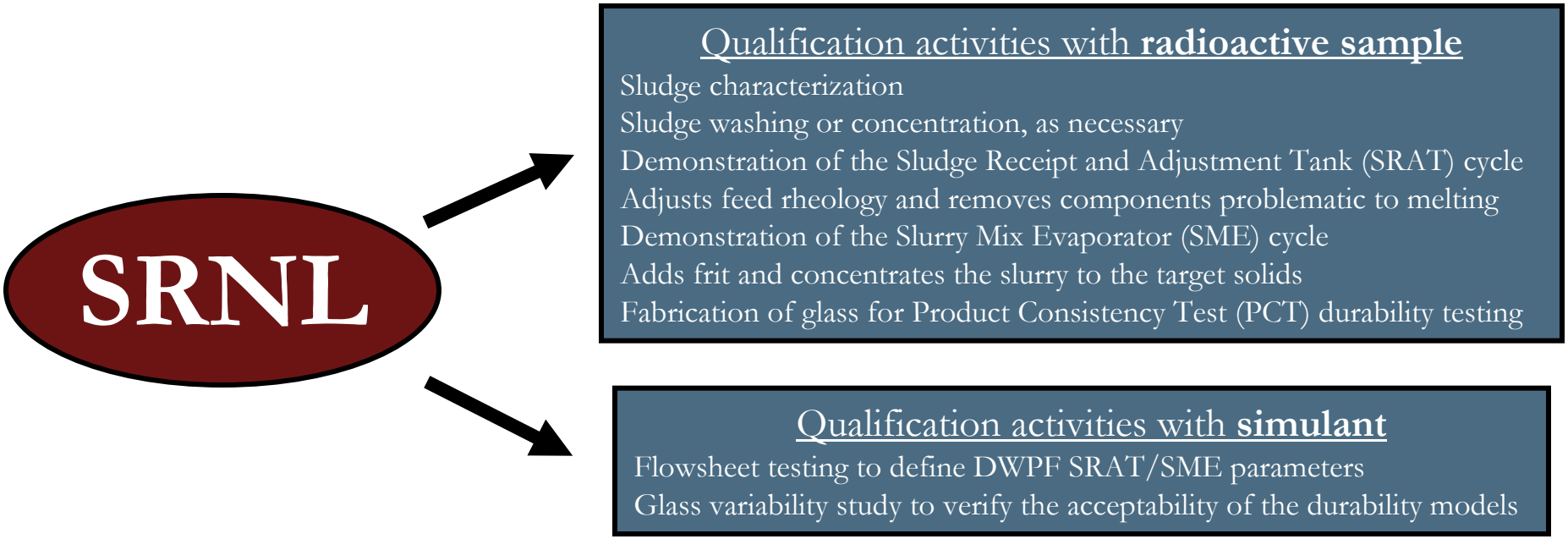
- No ability to evaluate rheology while in canyon
- Issues which may impact DWPF processing are typically identified during sludge batch **qualification**
- SRNL research and DWPF modeling efforts

GOAL: To avoid future process interruptions due to transport issues by having the ability to mitigate problems quickly and effectively, and to improve overall processing at DWPF.

A.3.103

DWPF Qualification

Prior to processing sludge at DWPF, it is well-characterized through SRNL radioactive and nonradioactive testing



A.3.104

SRNL: Analytical Methods

Density

- Plastic pipette tips are sealed with hot glue
- Volume calibrated using DI water (V_i)
- Sample placed into sealed tip using slurry pipette
- Mass of sample recorded (M_i)

- Density calculated: $$\rho_i = \frac{M_i}{V_i}$$

- Three to four measurements are made per sample

A.3.105

SRNL: Analytical Methods

Solids analysis

- Alumina crucibles used to obtain dried mass
  - Crucibles dried at ~115 °C, cooled and weighed
  - Sample transferred using slurry pipette and weighed (M_{total})
  - Sample dried at 115 °C until mass differential is less than 0.010 g (M_{dried})
- Sample placed into furnace to obtain calcined mass ($M_{calcined}$)
  - Furnace ramped to 1100 °C at 5 °C/min
  - Held at 1100 °C for two hours
  - When T<500 °C, crucible removed from furnace and air cooled prior to weighing
- Total/Calcine slurry and soluble solids in the supernate fractions are calculated:

$$f_{total_solids} = f_{ts} = \frac{M_{slurry,dried}}{M_{slurry,total}} \qquad f_{calcined_solids} = f_{cs} = \frac{M_{slurry,calcined}}{M_{slurry,total}} \qquad f_{supernate_solids} = f_{ds} = \frac{M_{supernate,dried}}{M_{supernate,total}}$$

- Insoluble solids fraction calculated using conservation of mass:

$$f_{insoluble_solids} = f_{is} = \frac{f_{ts} - f_{ds}}{1 - f_{ds}}$$

A.3.106

SRNL: Analytical Methods

EXAMPLE: Reduce sludge mass via removal of aluminum

↓

Reduction in number of canisters produced

	Tank 51 Before Al Dissolution (SC3) Products		Tank 51 After Al Dissolution (SC4) Products		
Physical Property	SRAT	SME	SRAT	SME	Units
Wt% Total Solids in Slurry	**21.3**	**49.6**	**22.6**	**48.4**	**%**
Wt% Soluble Solids In Supernate	**11.5**	**14.7**	**14.0**	**17.2**	**%**
Wt% Insoluble Solids in Slurry	**11.1**	**40.8**	**10.0**	**37.6**	**%**
Density of Slurry	**1.22**	**1.44**	**1.17**	**1.46**	**g/ml**
Density of Supernate	**1.09**	**1.12**	**1.10**	**1.13**	**g/ml**

- Total solids analysis are similar for both SRAT/SME products; differences apparent in soluble and insoluble contributions

A.3.107

SRNL: Rheology

Rheological measurements

- Haake RV20/30 rotoviscometer is utilized
  - Haake RV20/30 is functional-verified as operational using a NIST traceable oil standard to within +/- 10% of the standard at 25 ºC whenever sample measurements are made
- Sample placed into transfer cup and then placed into RV20/30 cup
  - Assists in removing entrained air bubbles
- Forward and backward rate sweeps performed on all materials
- Flow curve measurements are performed at 25 ºC
- Newtonian shear rate data is fitted with Bingham Plastic rheological model to determine Bingham Plastic yield stress and plastic viscosity

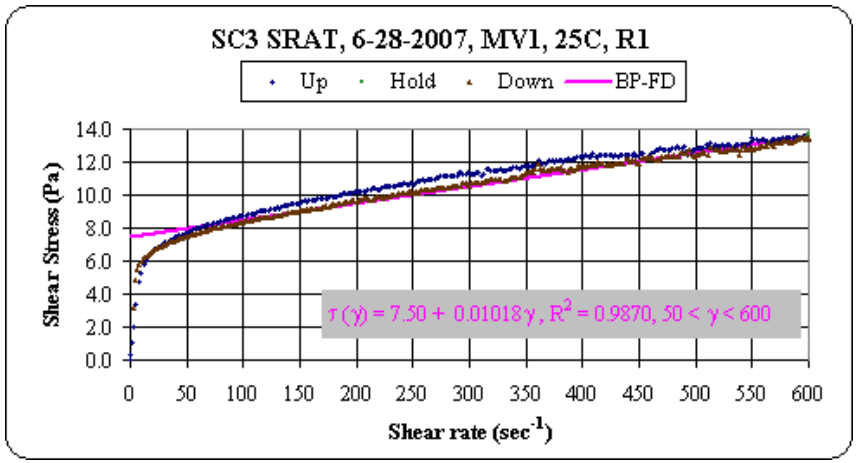
$$\tau = \tau_0 + \eta_{pl}\dot{\gamma}$$

- Currently, simulants are not suited to represent rheological behavior of radioactive waste

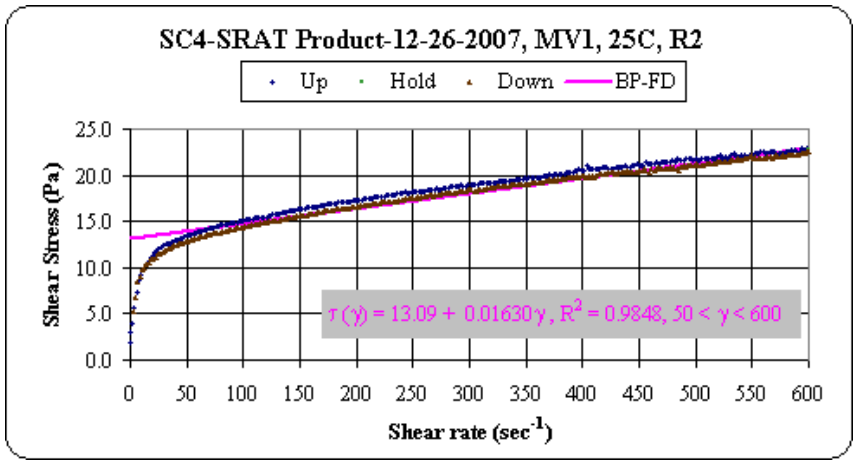
A.3.108

SRNL: Rheology

SRAT Product without LTAD



SRAT Product with LTAD



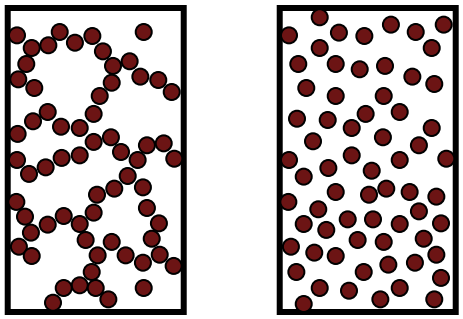
DWPF Design Basis in red

	Tank 51 Before Al Dissolution (SC3) Products		Tank 51 After Al Dissolution (SC4) Products		
Physical Property	SRAT	SME	SRAT	SME	Units
Bingham Plastic Yield Stress	7.2 (1.5-5)	10.9 (2.5-15)	13.3	21.6	Pa
Bingham Plastic Viscosity	10.2 (5-12)	19.8 (10-40)	16.5	29.2	cP

A.3.109

SRNL Work: Rheological Modifiers

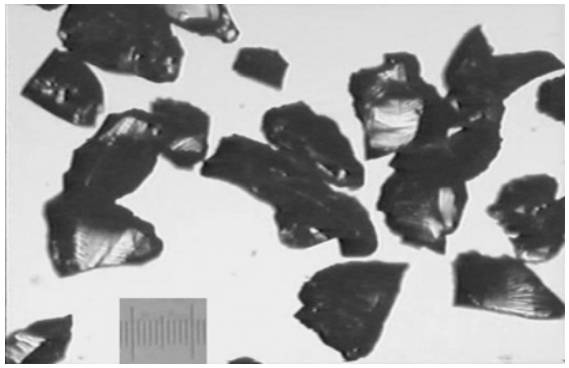
- Lower yield stress and consistency via prevention of particle agglomeration
  - Electrostatic
  - Steric
- Ethylene glycol and polyacrylate based dispersants studied (IIT)
  - Significant reduction in yield stress and moderate reduction of consistency during simulant testing
  - Mechanism based on steric break-down of gel-like structure
- Inherent difficulties
  - Effectiveness highly dependent upon pH
  - Impacts to DWPF unknown

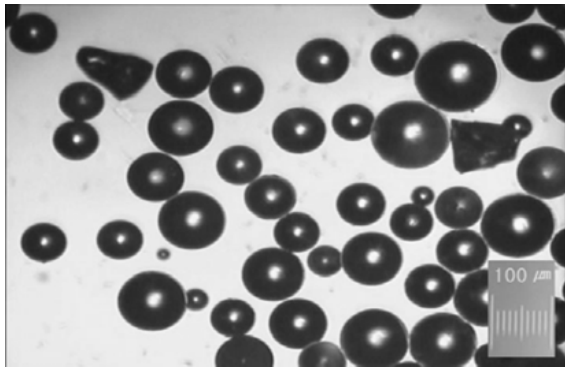


A.3.110

SRNL Work: Frit Morphology

- Transport issues due to high solids loading
- Frit passed through flame and quenched with water
  - 90% morphology alteration rate
  - Nominal size distribution unchanged
- Overall reduction in both yield stress and consistency
  - Higher loadings
  - Improved melt rate via reduction of water content
- Potential negative impacts
  - Settling behavior
  - Glass/waste oxide interaction





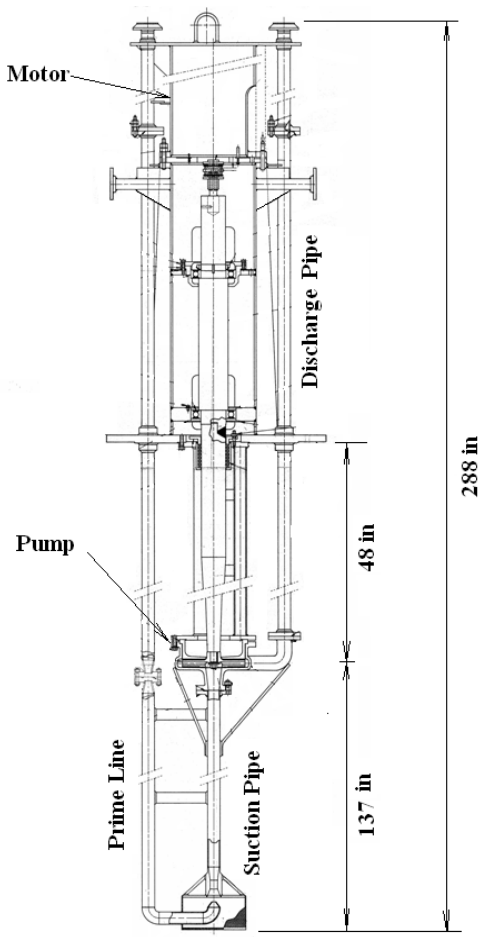
A.3.111

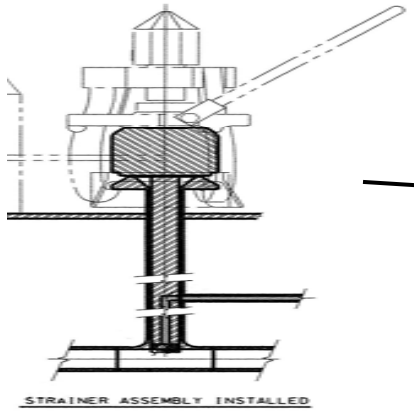
Modeling

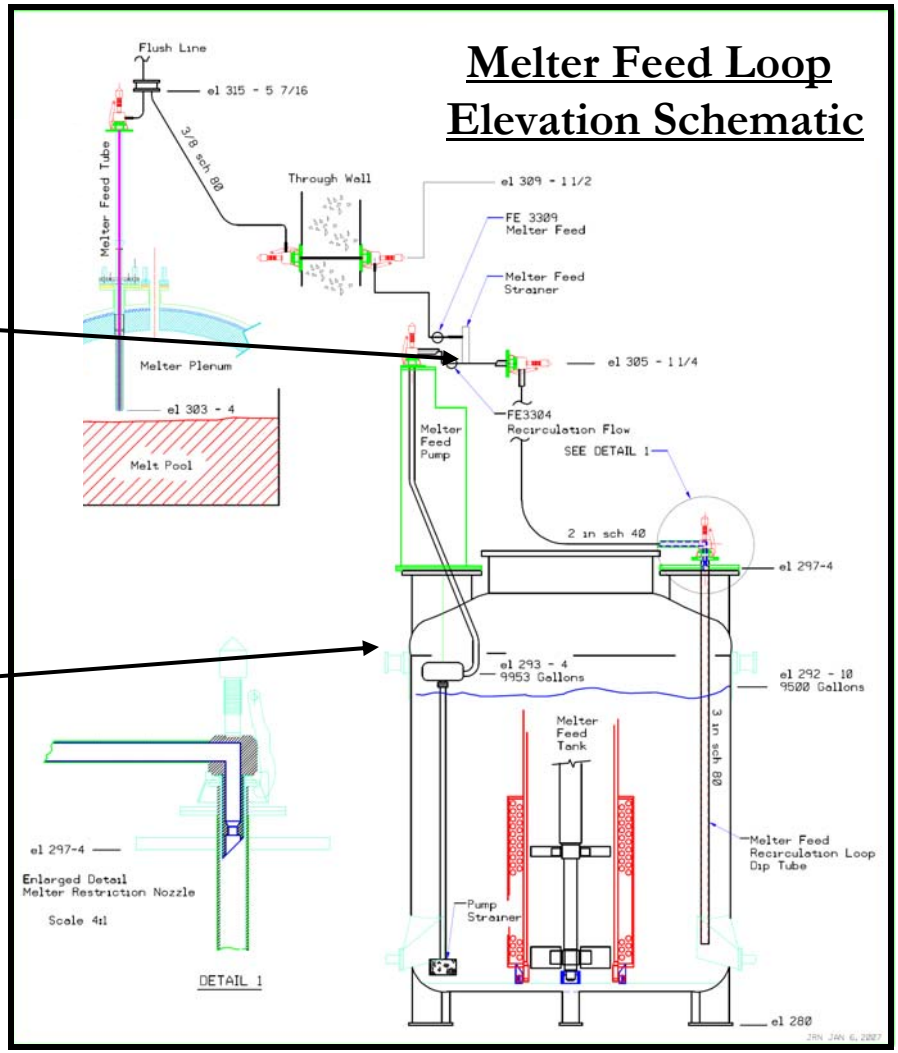
Slurry mixing complicated by geometry of tanks

A.3.112

Modeling







A.3.113

Modeling

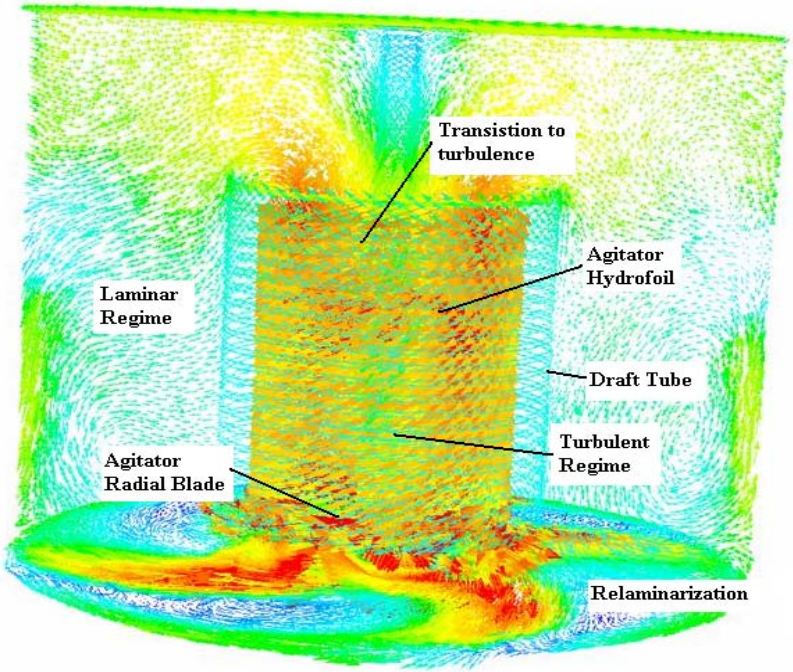
Melter Feed Tank (MFT) mixing process is being evaluated* by flow models built with Fluent Software

***This work is being performed by John Neuville and Professor Jamil Kahn from the University of South Carolina.**

- Tank modeled has a helical cooling coil in the center of the tank, and a centrally located agitator that has two impellers; an axial blade is positioned above a radial blade located at the bottom of the tank.

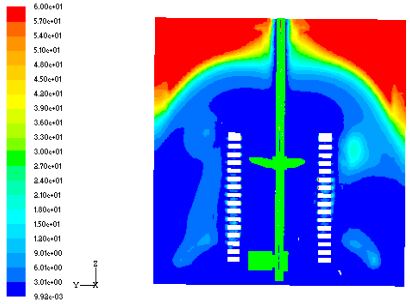
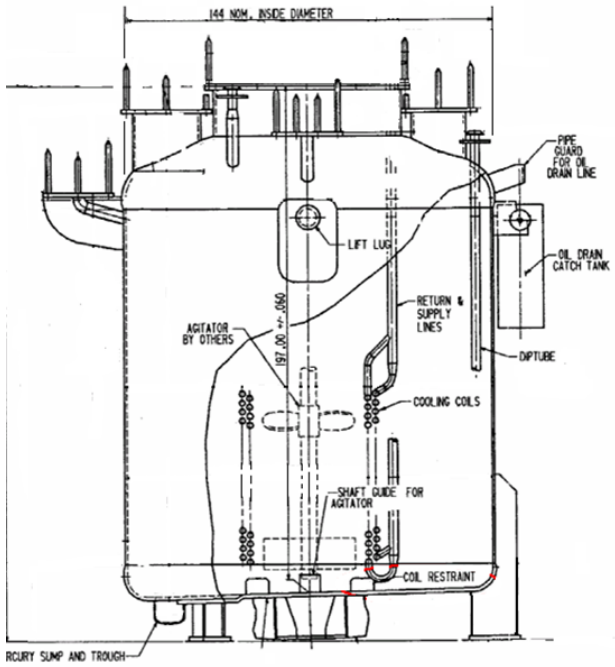
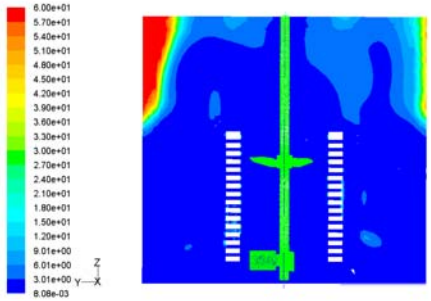
- A mixture in a stirred tank that exhibits a Yield Stress behavior can create various regions in the tank where the flow field is stagnant, laminar, or turbulent.

- Historically tank mixing performance has been evaluated by developing small scale models. The data collected by these models is then scaled-up by the use of dimensional analysis and similarity principals.



A.3.114

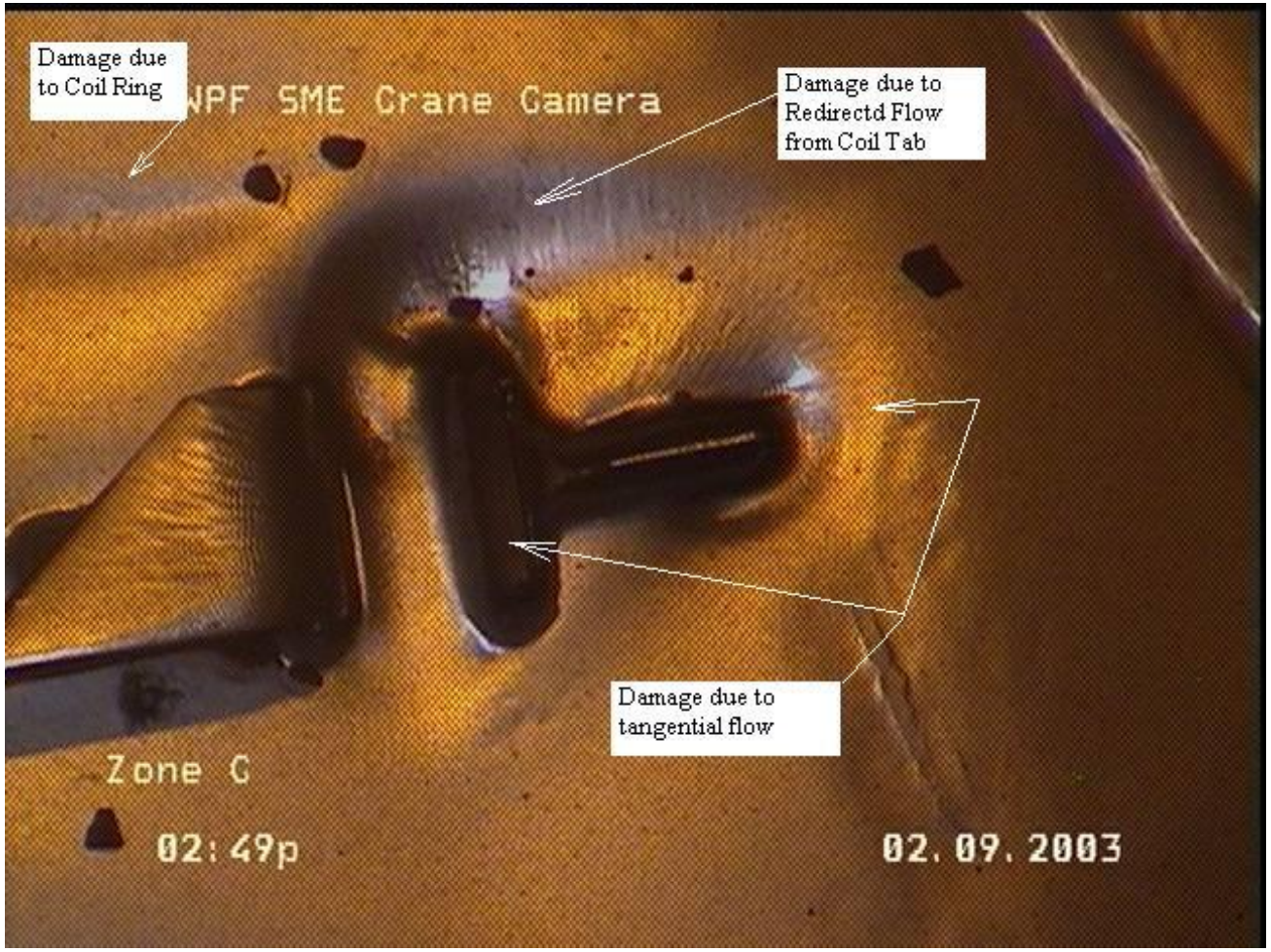
Modeling

CFD Scale Model Results- Stagnation Regions are Shown in Red	
Scale Model Result 40 Gallon Tank Herschel Bulkley; 5.0 Pa, 1.057 Pa*sec, n = 0.4133, N = 250 rpm	DWPF Melter Feed Tank 9500 Gallon Tank
	
Scale Model Result 40 Gallon Tank Herschel Bulkley; 5.0 Pa, 1.057 Pa*sec, n = 0.4133, N = 350 rpm	
	

A.3.115

Modeling

Tank Erosion to Coil Guides



A.3.116

Identification of Data Gaps

- Limited data on radioactive samples
  - Effort to create rheology database
  - Development of new rheological tests

- Need for simulant more representative of physical behavior of real waste

- Constitutive relationship describing slurry flow behavior

A.3.117

Conclusions

- Transport issues inherent in DWPF process are critical to operability of facility

- Tools to help mitigate rheological issues available
  - SRNL characterization abilities
  - Supplemental SRNL research
  - Modeling efforts

- Limited database
  - Appropriate constitutive relationships
  - Data on radioactive samples, simulants

A.3.118

Acknowledgements

D. WASAN and **A. NIKOLOV**

Department of Chemical and Environmental Engineering

Illinois Institute of Technology

Chicago, IL 60616

J. KAHN

Department of Mechanical Engineering

University of South Carolina

Columbia, SC 29208

A.3.119

Supplemental Slides

SRNL Capabilities

- Sample Prep
- Density
- Solids analysis
  - Total solids in slurry
  - Soluble solids in the supernate
  - Insoluble solids in slurry
  - Calcine solids in slurry
- Rheology
  - Flow curves
- SRNL ongoing efforts

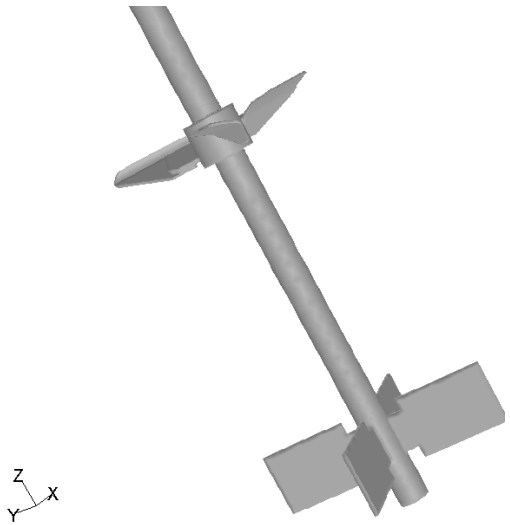
Low Temperature Aluminum Dissolution (LTAD) Example

SRNL: Analytical Methods

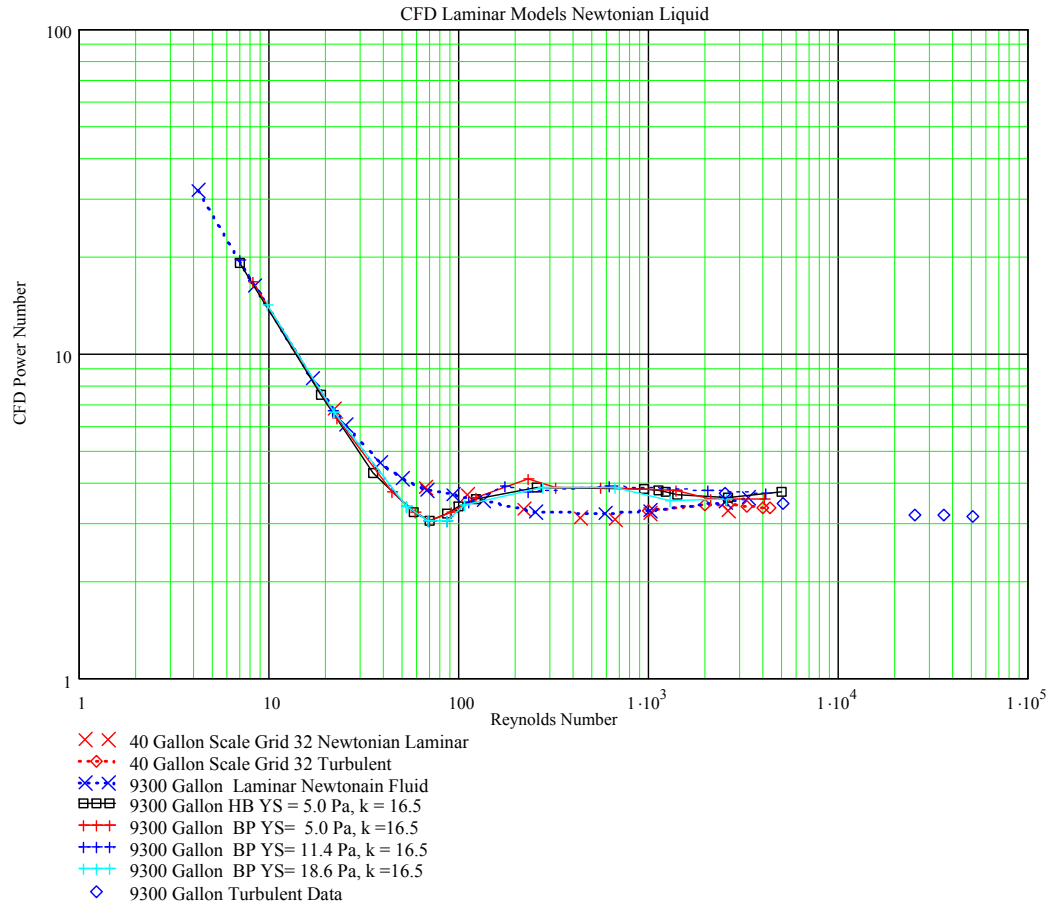
- Sample homogenized via shaking bottle. Rapid shaking can cause air entrainment.
- Sub samples are either transferred using slurry pipette or poured
- Sample reuse typically does not occur
- Supernate sample typically obtained by filtering a homogenized sample through a 0.45 micron filter

Modeling

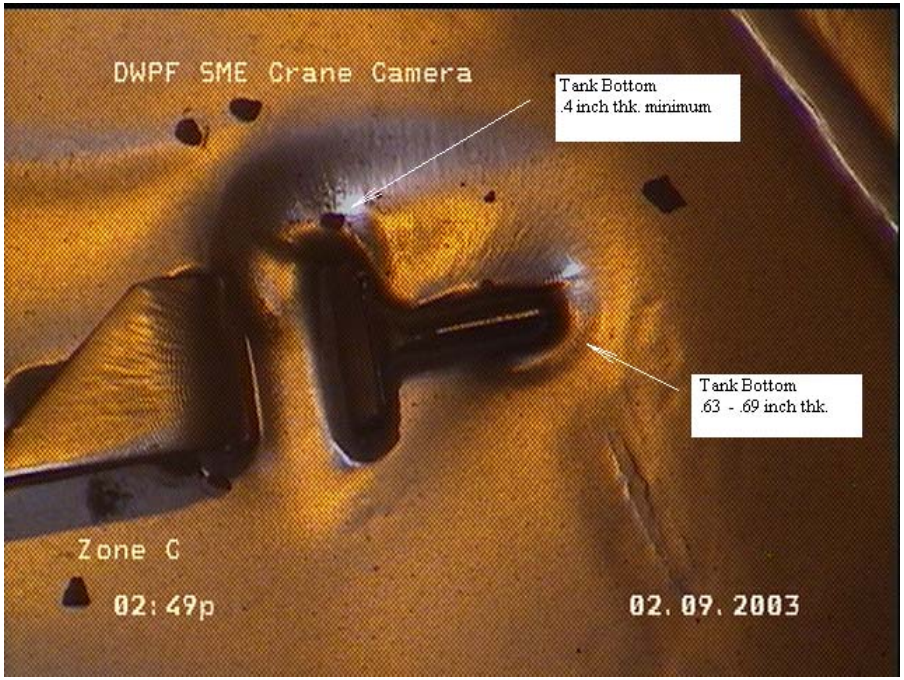
MFT Full Scale CFD Mixing Model

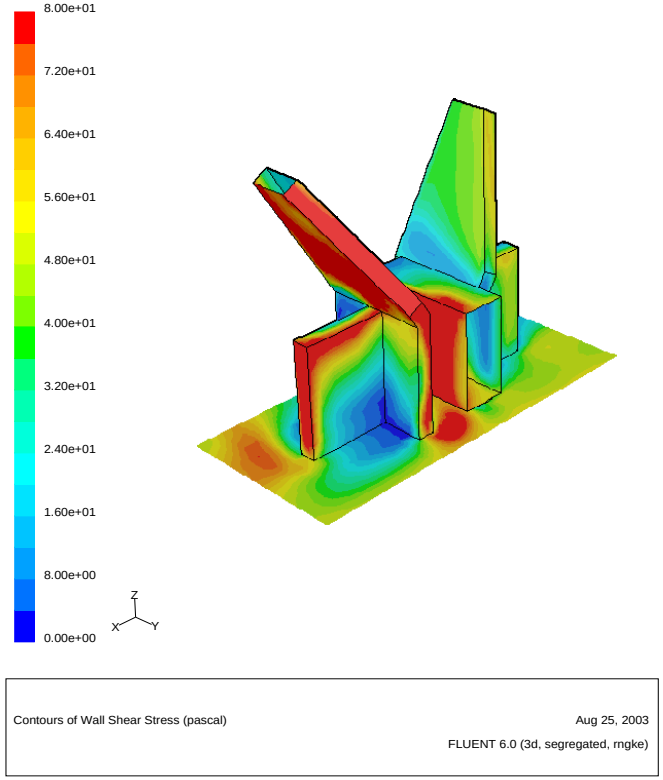


Otto-Metzner Laminar Curve
"Np = 136.8/Re"
Proportionality Constant for
Power Curves k =16.5



Modeling

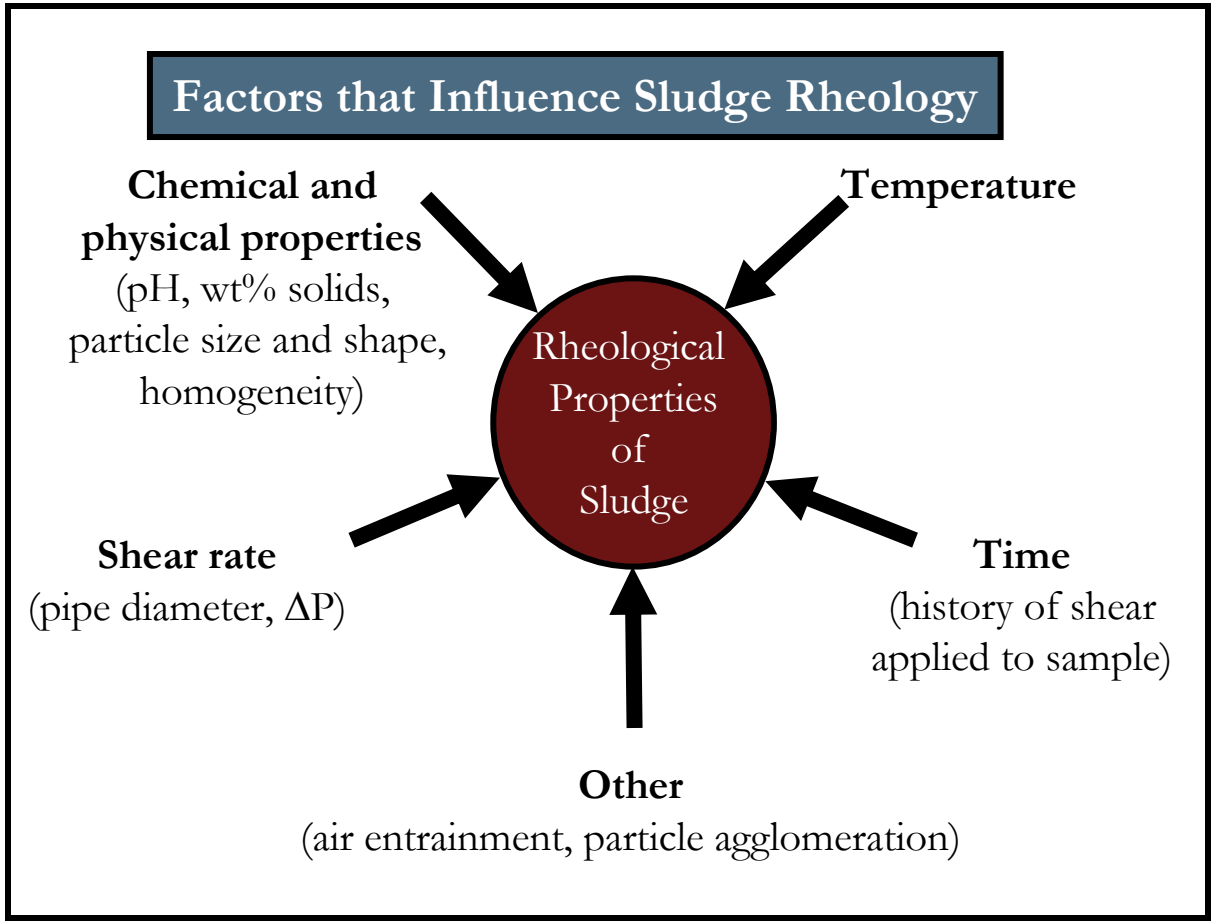




- Calculated Wall Shear Stress Patterns agree with observed wear patterns.
- The relative magnitude of shear stress compare well with depth of erosion.

Si Y. Lee, Richard A. Dimenna, John R. Neuville, Glenn A. Taylor, EROSION MODELING ANALYSIS FOR DWPF SME TANK; WSRC-TR-2003-00534

Summary



Tools Available

SRNL analytical
CFD modeling
Rheology modifiers
Frit bead testing

Tank Waste Retrieval Operations at Oak Ridge National Laboratory

Ben Lewis - Nuclear Science and Technology Division

ORNL Tank Waste Retrieval

- Purpose: Consolidation of waste from the active and inactive storage tanks to a single active waste storage tank system
  - Inactive Tanks
    - 12 Gunite and Associated Tanks (GAAT)
    - 5 Old Hydrofracture Facility Tanks
    - Multiple Federal Facilities Agreement Tanks
  - Active Tanks
    - 5 Bethel Valley Evaporator Service Tanks (BVESTs)
    - 8 Melton Valley Storage Tanks (MVSTs)
    - 6 Melton Valley Capacity Increase Tanks

Waste Consolidation Tanks

A.3.127

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Typical Sludge Characteristics

	Range	Average	Wt %
Density (g/mL)	1.169 – 1.675	1.343	
Water (wt%)	52.9 – 72.7	59.1	59.1
pH	8.7 – 12.0	10.1	
TOC (mg/kg)	2,300 – 13,400	7613	0.76
Ca (mg/kg)	23,400 – 73,700	49,825	4.98
Na (mg/kg)	23,700 – 48,000	39,225	3.92
U (mg/kg)	18,000 – 41,900	29,100	2.91
NO_3 (mg/kg)	73,500 – 233,000	135,400	13.54
^{137}Cs (Ci/gal)	0.0639 – 0.235	0.0989	
^{90}Sr (Ci/gal)	0.0988 – 0.51	0.272	
G Beta (Ci/gal)	0.507 – 1.27	0.731	
G Alpha (Ci/gal)	0.00668 – 0.027	0.0136	

A.3.128

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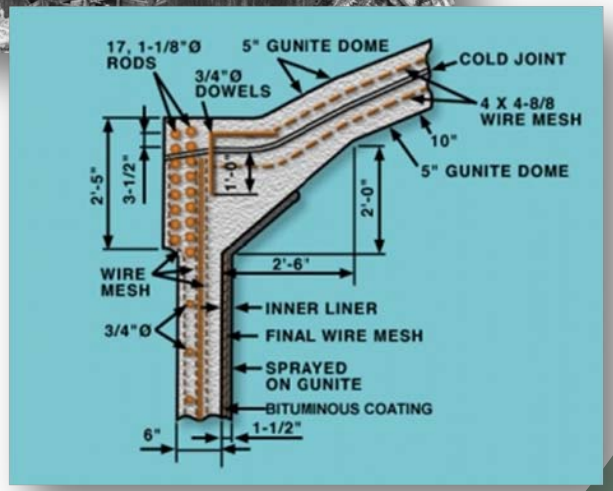
Waste Transfer Line

- **2 in. diameter SCH 40 Stainless steel pipe**
  - **Double contained within 3 in. diam. SCH 40 pipe**
- **Multiple elevation changes (~51 ft) between Bethel Valley Evaporator Service Tanks and Melton Valley Storage Tanks**
- **Over 1 mile long**
- **Moyno progressive cavity transfer pump or Discflo centrifugal transfer pump**
- **Waste Acceptance Criteria**
  - **suspended solids <5 wt %**
  - **maximum particle of 100 μm**

A.3.129

Gunite Tanks History

- In 1943 twelve underground storage tanks were constructed of ”gunite" - a sand and Portland cement mixture sprayed over a wire mesh and reinforcing rod frame.
- The tanks were used to store wastes from “pilot scale” separation operations and research missions.
- The tanks were removed from service in the early 1970’s.
- Most (~90%) of the accumulated sludge and liquid waste was removed during an 18 month campaign from 1982 through 1984.



A.3.130

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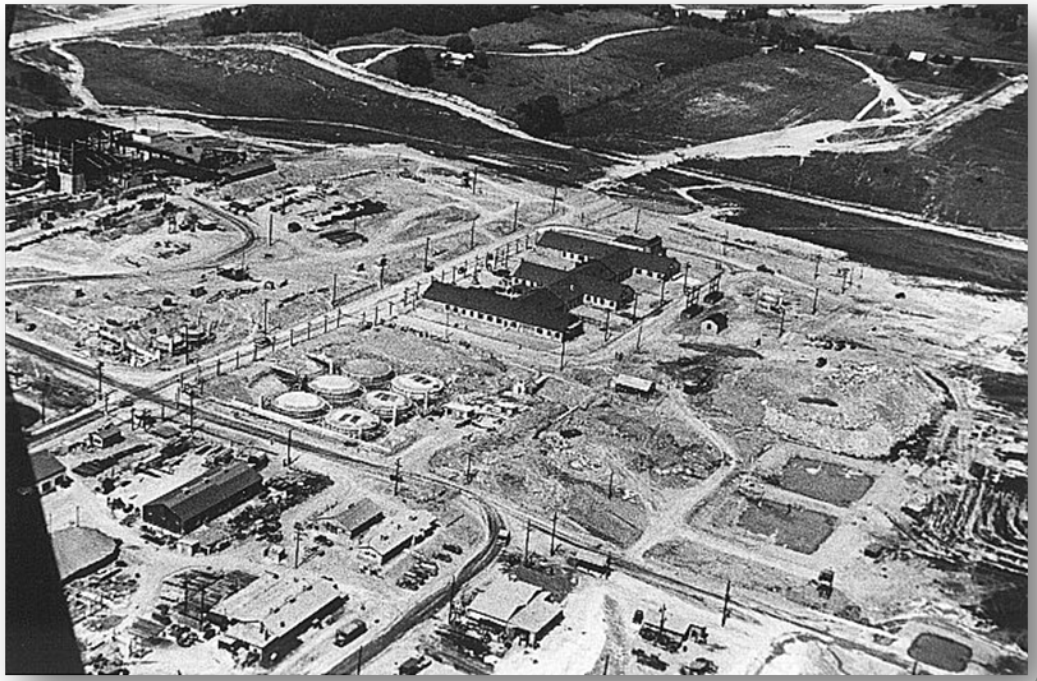
Gunite Tanks Project Goal

- Remove the remaining transuranic sludge (~94,000 gal) and supernatant waste from the 55-year old gunite tanks located in the main plant area of Oak Ridge National Laboratory
- Consolidate the waste in the permitted Melton Valley Storage Tanks
- Address final closure



ORNL during construction - 1943

A.3.131

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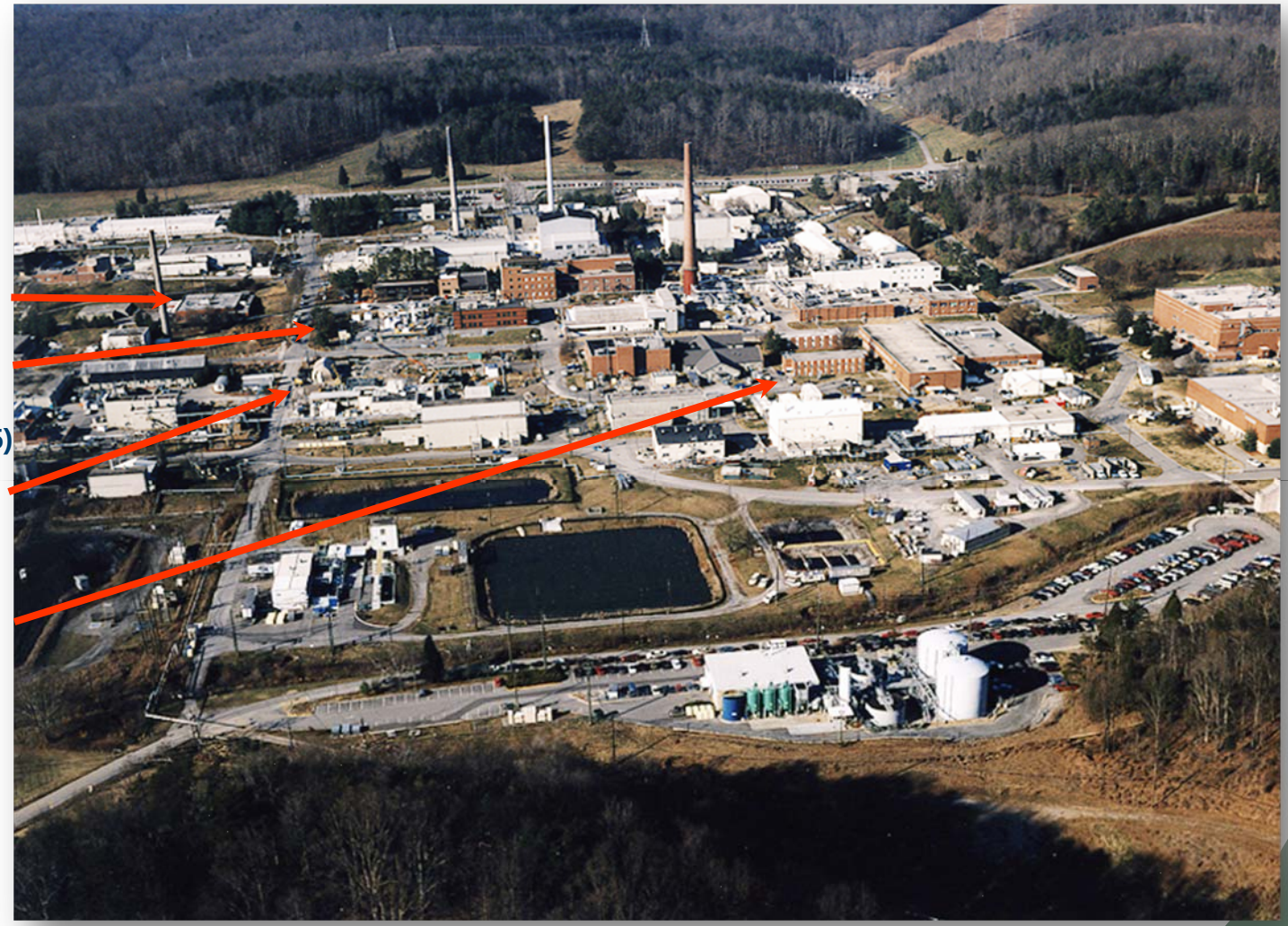
The Gunite Tanks are Located in Central ORNL



A.3.132

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Gunite Tanks Status

- January 2001 – Completed waste removal operations in the nine largest gunite tanks
  - Removed 439,000 gallons of waste (sludge and supernate) containing 82,000 curies
  - Sludge successfully transferred to the Melton Valley Storage Tanks
  - Completed waste retrieval operations ~5.5 years ahead of the original baseline schedule
    - Savings of over $120 Million
- Site demobilization completed
  - Secondary waste has been containerized for disposal and equipment either reused or disposed
  - Tanks have been filled with grout
  - Site is now a parking lot

A.3.133

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The GAAT Remediation Deployed Approximately 40 Technologies*

Sampling, Characterization, and Modification

- Floating boom In tank Camera & Sampling Device
- Ponar Sampling Tool
- Sludge Mapping Tool
- Topographical Mapping System
- Large Diameter Coring Saw for Tank
  - Riser Installation
- Remote Video Cameras & Lighting
  - Multiplexed Pan & Tilt Controller for multiple Cameras
- Gunite Isotope Mapping Tool
- Characterization End-Effector
- Feeler Gauge
- Hydraulic Shears
- Pipe Cutting Saw
- Pipe Plugging Tool
- Wall Coring Tool
- Wall Scraping Tool

Waste Mixing

- Flygt Mixers
- PulsAir Mixers
- Russian Pulsating Mixer Pump

Sludge Heel Retrieval and Wall Cleaning

- Modified Light Duty Utility Arm
- Houdini I Remotely Operated Vehicle Houdini II Remotely Operated Vehicle
- Decontamination Spray Ring
- Waste Dislodging & Conveyance System
  - Confined Sluicing End-Effector
  - Hose Management Arm
  - Axial Flow Jet Pump
  - Flow Monitor & Sampling Device
- Gunite Scarifying End-Effector
- High Pressure Pump for Wall Scarifying
- Gripper End-Effector Hydraulic Pump
- Linear Scarifying End-Effector

Waste Conditioning and Transfer

- In-line Sampler
- Waste Removal & Transfer System
- Sludge Conditioning System
  - Primary Conditioning System Module
    - In-Line Sampler
    - Size Classifier
  - Disc Flow Pump
  - Solids Monitoring Test Loop
    - Particle Size Analyzer
    - Ultrasonic Suspended Solids Monitor
    - Coriolis Density Meter

* Refer to Lewis, B.E, et al., *The Gunite and Associated Tanks Remediation Project Tank Waste Retrieval Performance and Lessons Learned*, ORNL/TM-2001/142/V1, Sept. 2003, for additional information.

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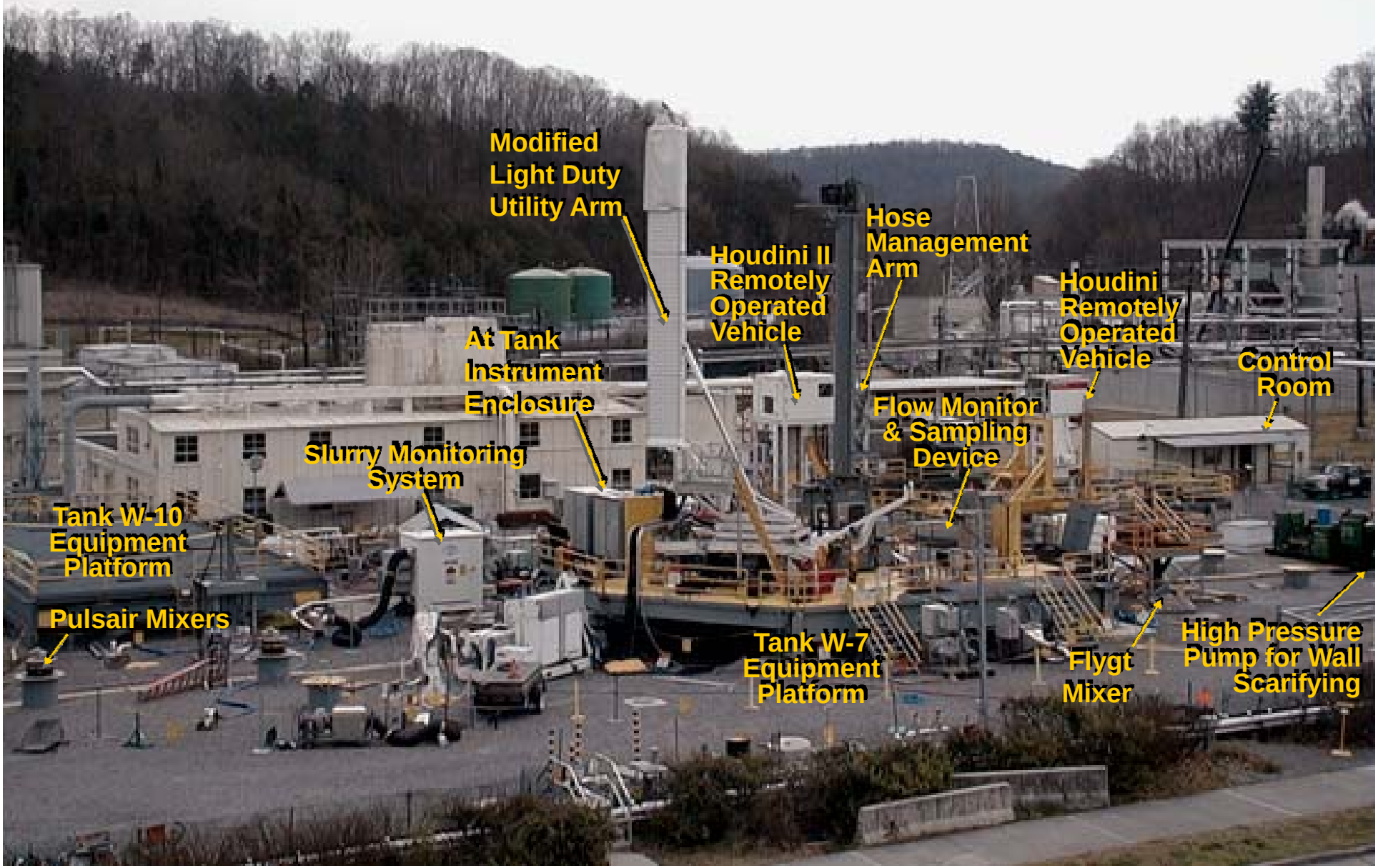
The Gunite Tanks Remediation Project
South Tank Farm Operations



Key Systems

- **Remote camera and lighting** – Served as the eyes of the equipment operators
- **MLDUA** – 8 degree-of-freedom robotic arm used to deploy tank characterization equipment, tank modification tools, and waste retrieval and wall-cleaning end-effectors
  - Gripper end-effector
  - Two cameras
  - 15-ft reach and 200-lb payload capacity
  - Operated remotely or via preprogrammed sequences
- **Houdini ROV** – 1000-lb tethered collapsible vehicle with a 4 x 5 ft expanded footprint that provided versatility during in-tank operations to deploy various tools and end-effectors
  - Track driven via hydraulic motors
  - 6 degree-of-freedom robotic arm and gripper end-effector with a payload capacity of 240 lb
  - On-board cameras
  - Plow blade for breaking up and pushing sludge
- **Waste Dislodging and Conveyance System** – Provided the capability to dislodge and retrieve waste, manage the in-tank hoses and lines, and deploy various tooling
  - Confined Sluicing End-Effector with rotating cutting jets
  - Jet pump vacuum source
  - Hose Management Arm

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Heel Retrieval - Dewatering

The Confined Sluicing End-Effector (CSEE) was used in conjunction with the MLDUA and HMA to remove liquid waste in preparation for sludge mining

A.3.137

Heel Retrieval - Sludge Mining

High-pressure water (10 ksi) and rotating (0–500 rpm) cutting jets were used to dislodge the sludge

The jet pump removed sludge through a Flow Monitor and Sampling Device to a waste consolidation tank via a 2-in.-diam hose connected to the Hose Management Arm

A.3.138

Heel Retrieval - Sludge Mining

The Houdini was used to plow sludge toward the CSEE to improve sludge-mining operations

Typically, less than 1 in. of sludge remained in the tanks after sludge mining

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Wall Cleaning

The Gunite-Scarifying End-Effector (GSEE) was used in conjunction with the MLDUA to remove contamination from the tank walls

Water pressure up to 22,000 psi was used with the GSEE

A.3.140

Waste Mixing

- Pulsair Mixers
  - These devices used a 13 pulse-plate mixing system to mix the waste and keep the solids suspended in the consolidation tank
- Flygt Mixers
  - Two 15-hp mixers were used to mix and suspend sludge for transfer out of tanks W-5 and W-9

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Waste Mixing

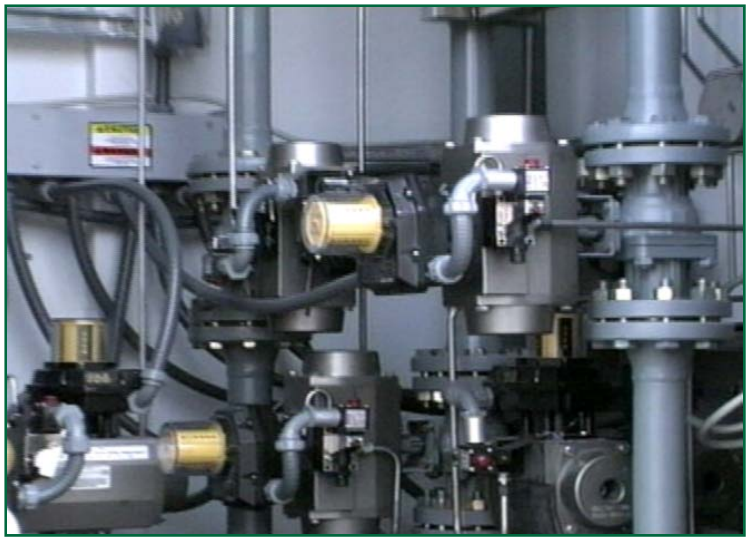
- Russian Pulsating Mixer Pump
  - *Mobilized waste and kept sludge suspended in tank TH-4*

  - *82.5% of sludge volume and initial radioactivity were removed*

A.3.142

Waste Conditioning and Transfer

- Primary Conditioning Module
  - Particle size classification to meet Waste Acceptance Criteria
- Slurry Monitoring Module
  - Monitored waste characteristics to ensure uniform consistency
- Discflo Pump
  - Provided consistent pressure and flow during waste transfers



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Old Hydrofracture Facility

- Five horizontal cylindrical tanks, 8- to 10.5-ft diam and 23 to 44 ft long, constructed of carbon steel
- Initially contained about 53,000 gallons of radioactive waste from hydrofracture operations
- Submersible pumps were used in conjunction with a Bore-hole miner with an articulated extendable nozzle to mobilize and retrieve >98% of the wastes from these tanks
- Completed closure of the tanks in FY 2000 by grouting in place

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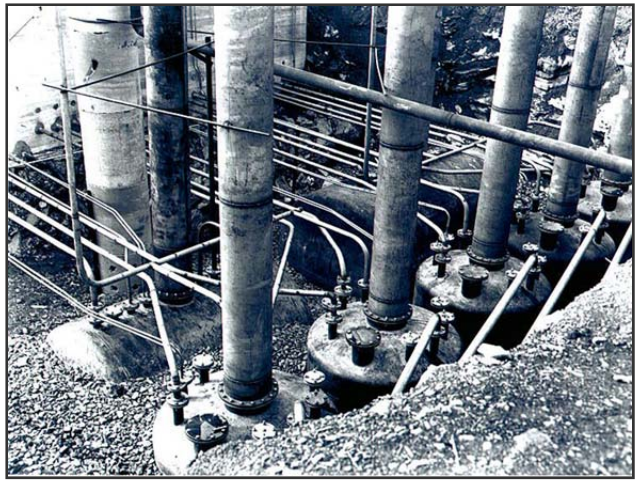
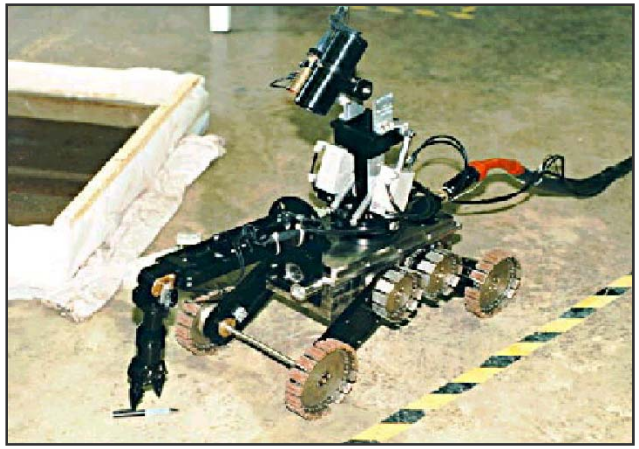
FFA Tanks

- Many of the FFA (Federal Facilities Agreement) tanks were emptied and then filled with grout
- The Scarab III ROV was deployed in tank T-14 for sludge sampling
  - Small mobile platform
  - Lightweight manipulator
  - On-board cameras and lights
- A mobile AEA power fluidic pump and a hurricane nozzle retrieval system were deployed in FFA tank 3003A



Numerous inactive Federal Facility Agreement tanks



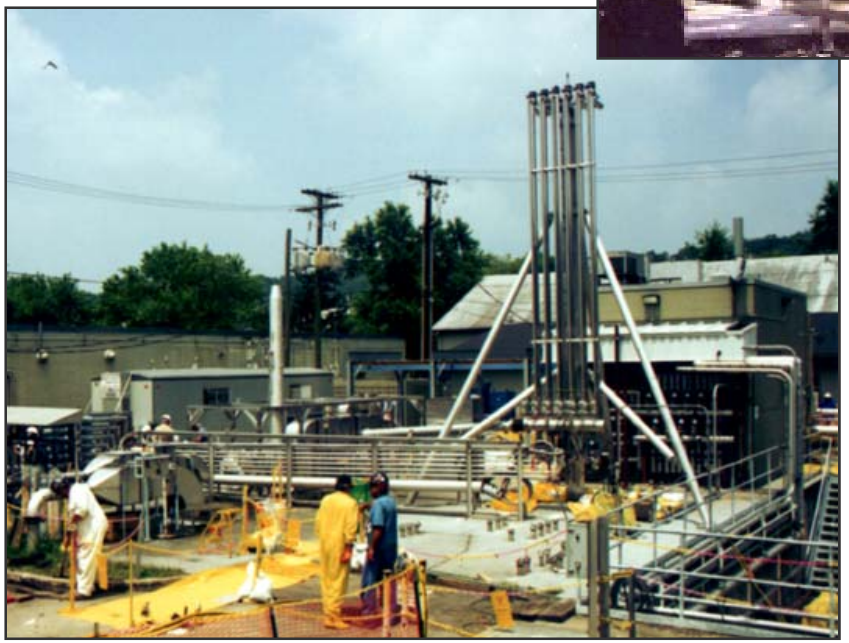
Scarab III Remotely Operated Vehicle

Mobile AEA power fluidic pump at tank 3003A

A.3.145

Active Low-Level-Waste Tanks

- AEA pulse-jet fluidic mixer systems were deployed at the BVESTs to mobilize ~40,000 gallon (>97%) of sludge for transfer to the MVSTs
  - Five 50,000 gal horizontal stainless steel tanks
- Existing piping and in-tank nozzles were used with the AEA system in three of the BVESTs
- An AEA mixing system was also included in the design of the Melton Valley Capacity Increase Tanks
- Principle of operation:
  - Vacuum used to pull slurry into pulse chamber
  - Pressurized air used to expel slurry from pulse chamber to mobilize sludge



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Advice

- **Equipment**
  - Consider ergonomics for repetitive manual operations
  - Use high quality components and parts
  - Understand the nature of prototypic systems
    - Expect some valve and line failures in initial prototypes
  - Ensure fastener integrity and robustness
  - Maintenance issues are key to success. Design for:
    - High reliability
    - Ease of maintenance
    - Replacement vs. repair
    - Maintenance friendly containment
  - Practice preventative maintenance and inspections to identify problems and extend operating life
  - Understand equipment limitations
- **Environment**
  - Know the environment but expect something worse – Waste material variability will likely be greater than initially expected
  - Work schedules during hot operations should be structured around weather considerations

A.3.147

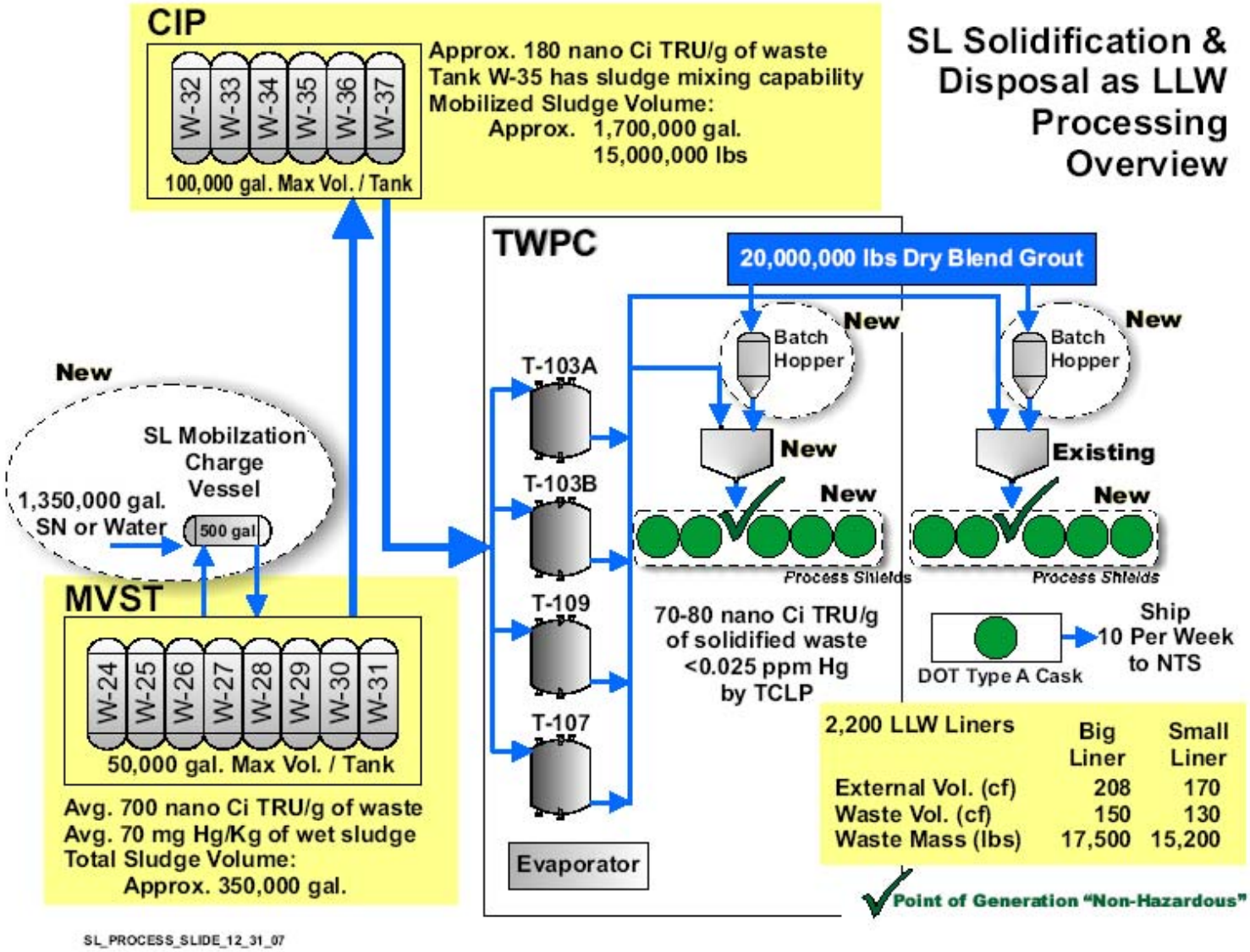
Advice (cont.)

- **Operators**
  - Cross training on various systems may be required to optimize staff utilization
- **Ensure that system components are thoroughly tested under a broad range of conditions**
  - If possible cold test everything
- **Control system**
  - Control system interfaces should be designed with consideration for the talents, abilities, and background of the personnel who will be operating the equipment
  - User-friendly straightforward equipment interfaces should be used
  - Operator input should be used during the design to ensure ownership and acceptance of the equipment in the field

ORNL MVST Sludge (SL) Solidification Feasibility Study Overview

Presented at Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop, January 14-18, 2008

ORNL TRU Waste Processing Center



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Sampling/Analytical Cost

- Lowest sampling/analytical cost
- NTS has agreed to the “big batch” concept (i.e., 80,000 gallon batches in CIP tank W-35)
- Number of batches reduced from ~200 to ~20
- Minimizes the number of samples per batch
  - NTS minimum 4 per EPA SW-846
  - WIPP minimum 10 per waste stream/batch
- LLW Characterization/Analytical costs < ½ the cost of RH-TRU costs for WIPP even if WIPP can accept the “big batch” concept
- Potential for greater savings using modeling to confirm earlier sampling (i.e., gross alpha to TRU correlation)

Sampling/Analytical Cost

- MVST Sludge is well characterized
- Newer 2001 Keller Report on sample results correlates well with Bayne statistical analysis of historical data
- Poor correlation between total Cs-137 (primary gamma isotope) and total alpha, poor application for Dose to Curie (DTC)
- Excellent correlation between total TRU isotopes and Gross alpha (R^2 = 0.9261)
- Gross alpha analysis can be performed at ORNL (< $1,000/sample, <1 day TAT)

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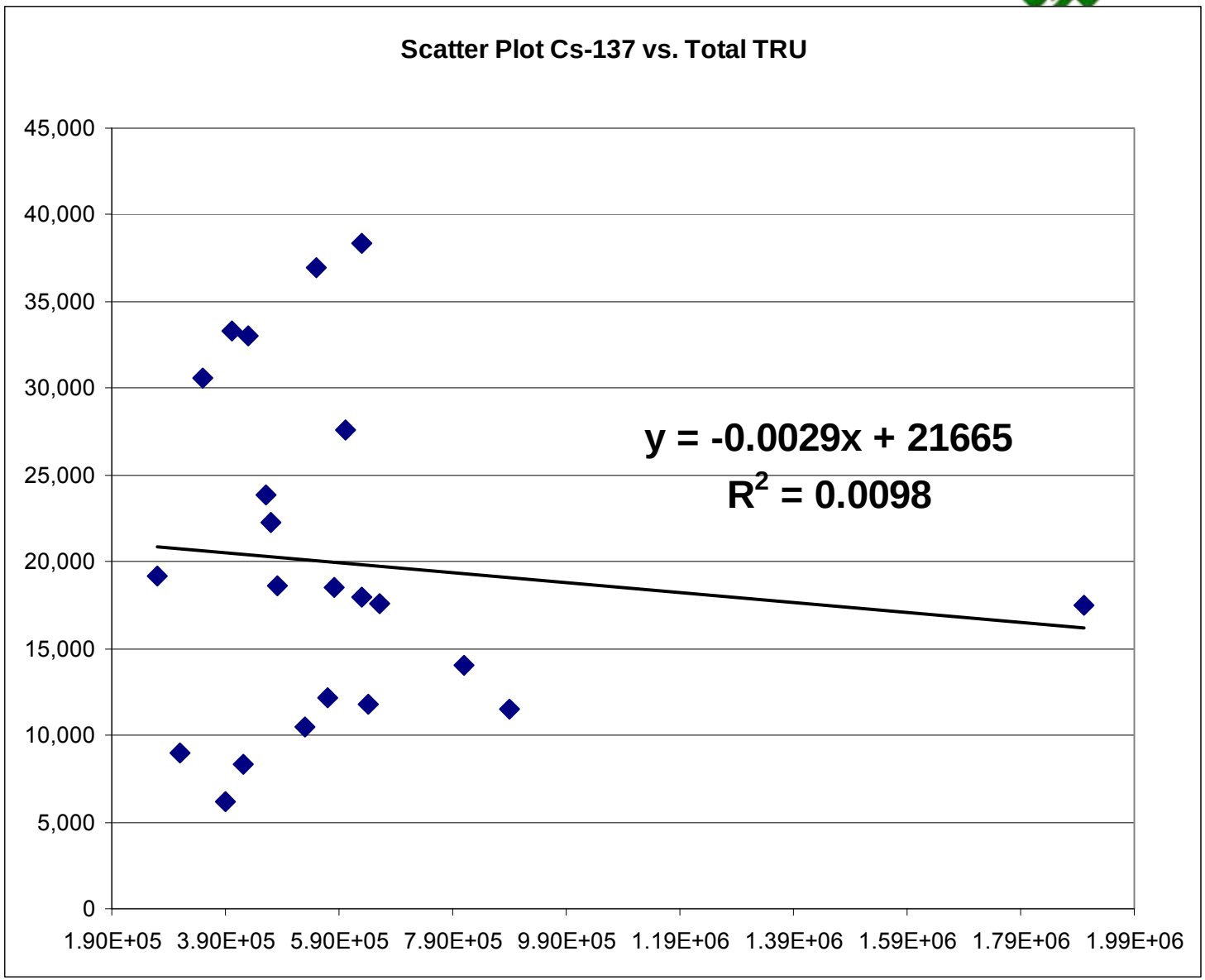

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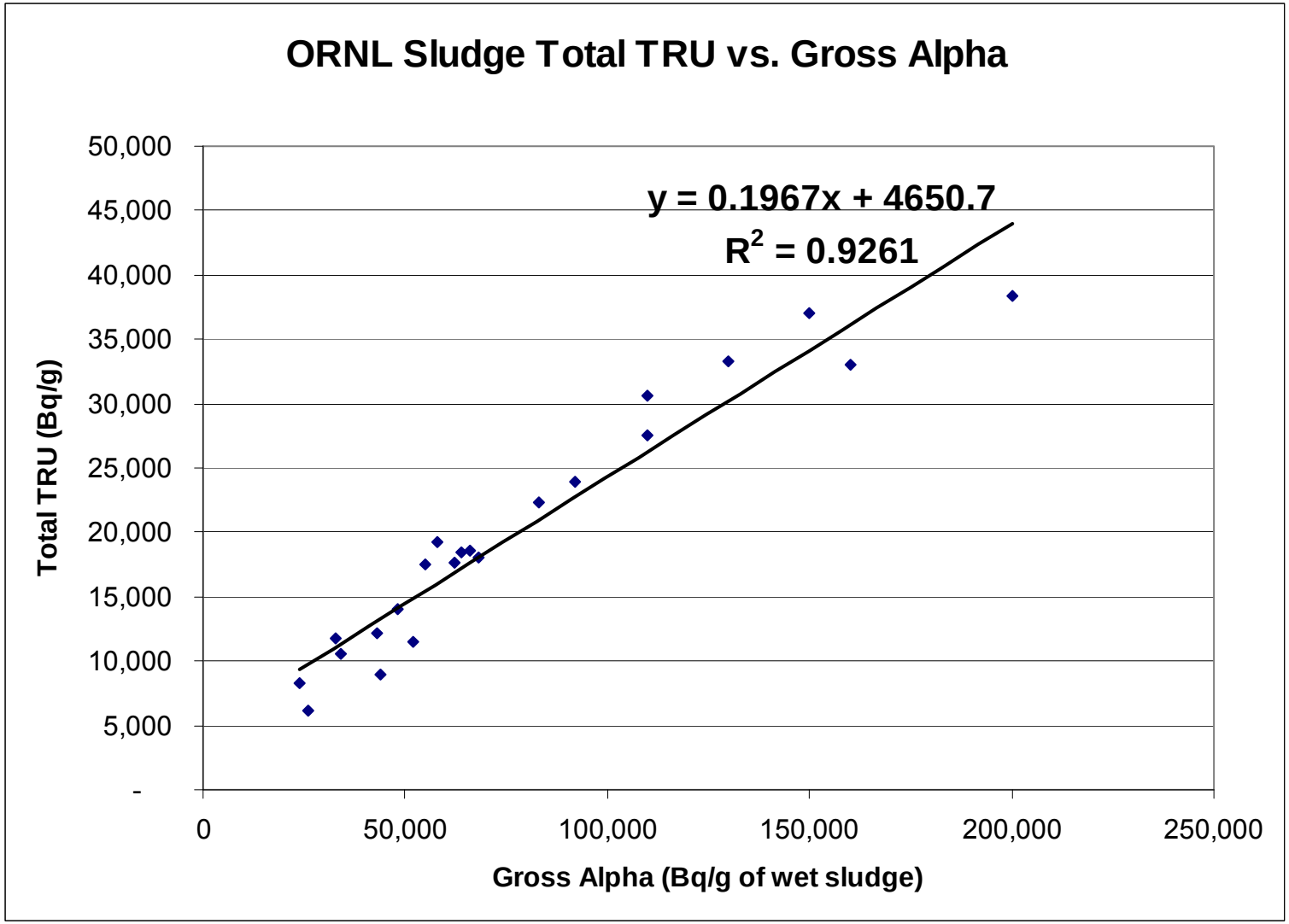

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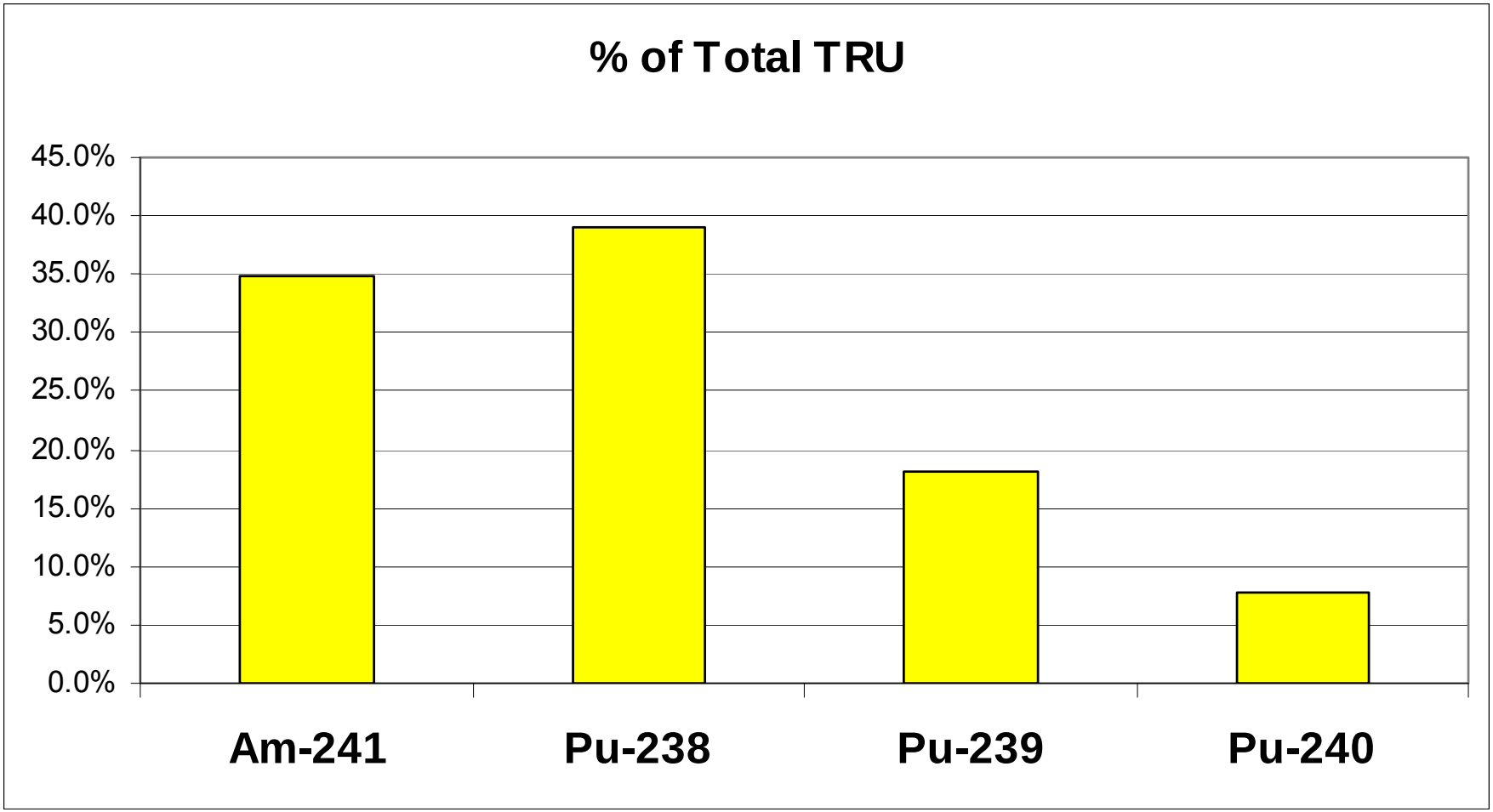

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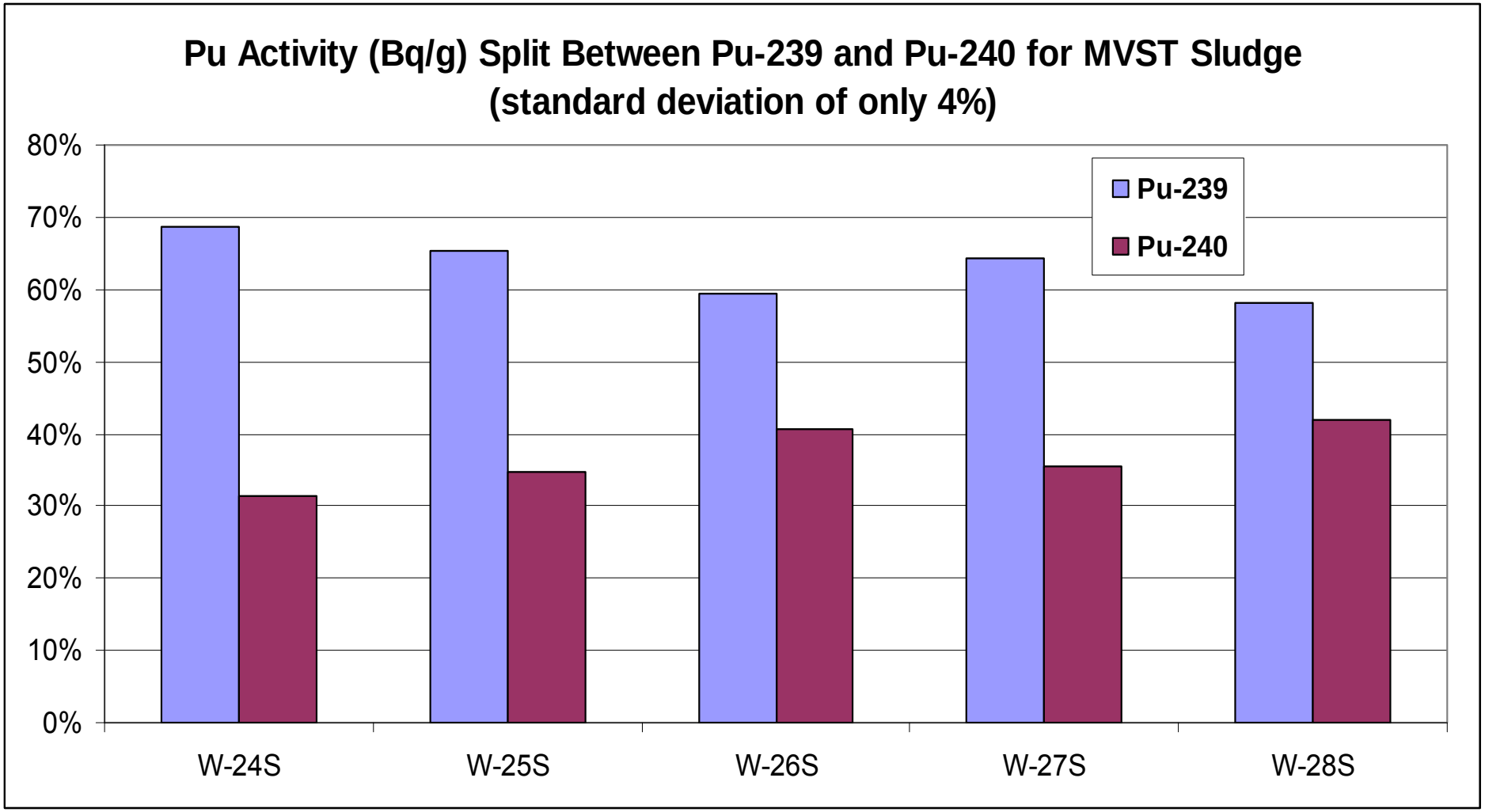

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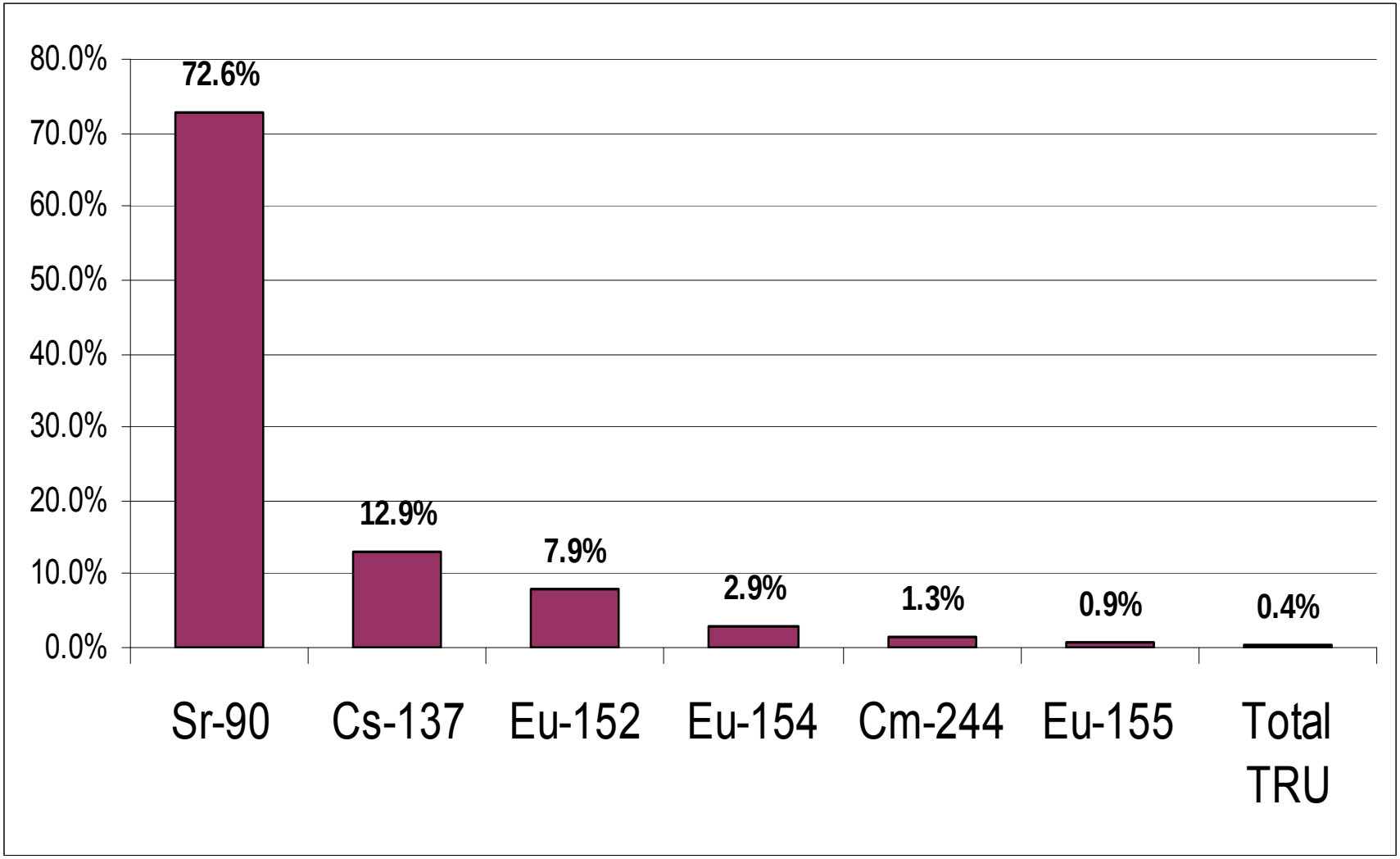

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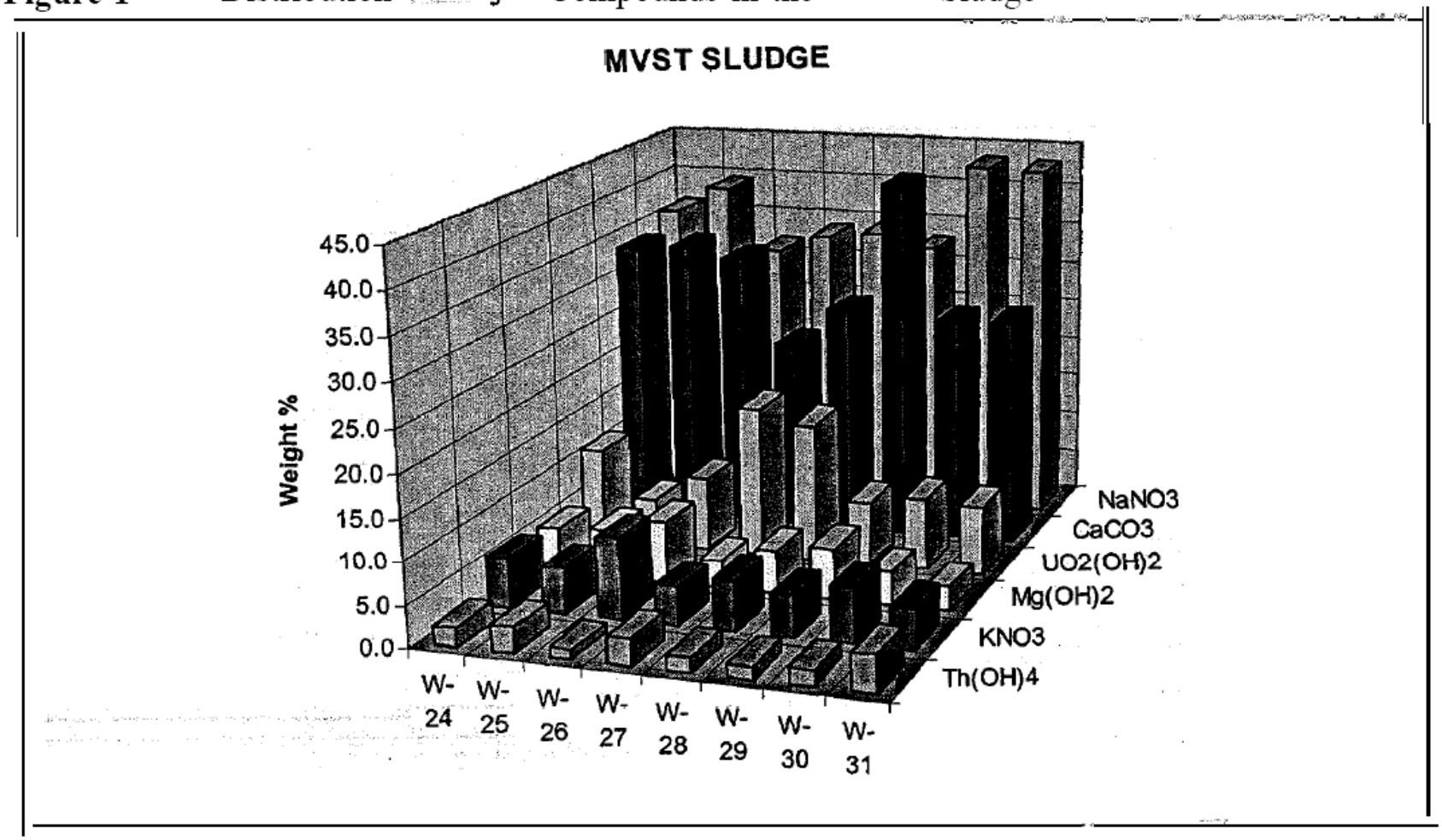
MVST Sludge-Witches Brew

Figure 1 Distribution of Major Compounds in the MVST Sludge



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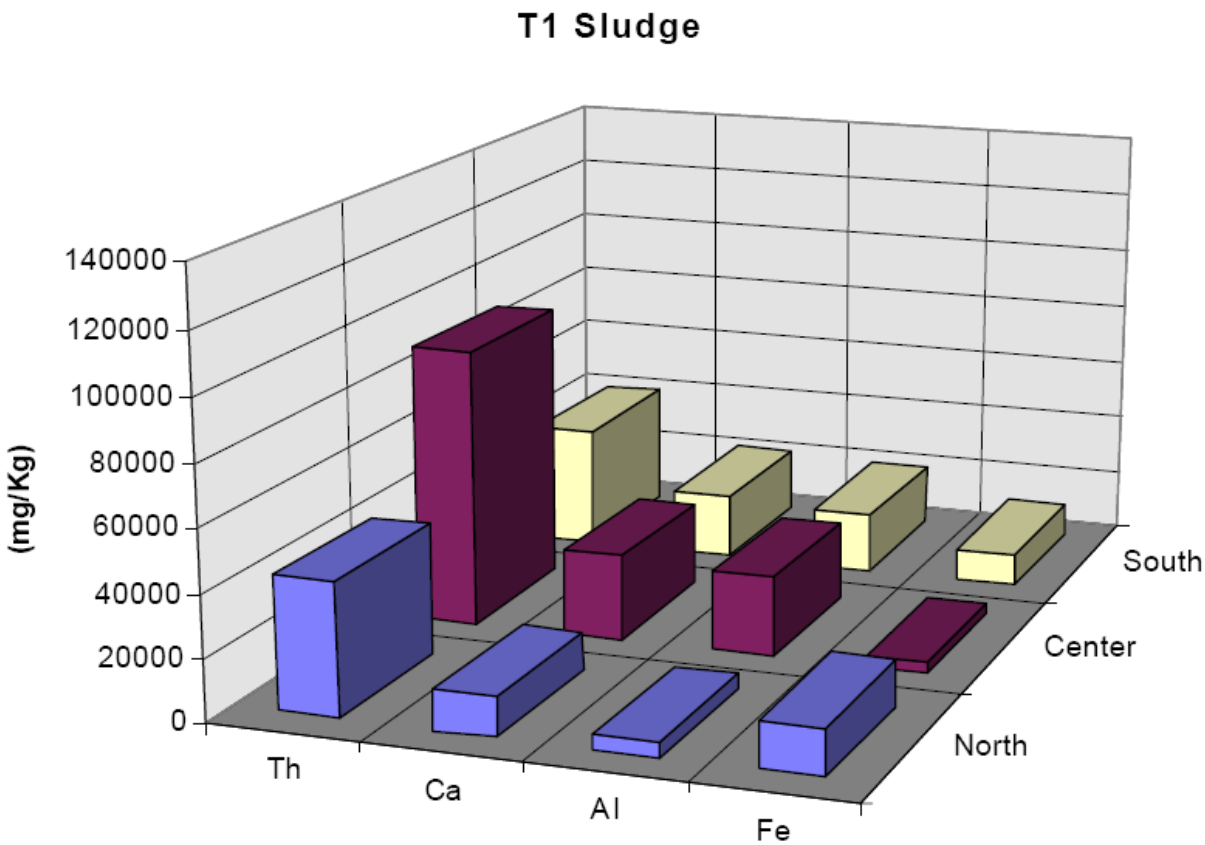
Sludge Major Metal Variation within a Tank

Figure 25 Lateral Distribution of Major Metals in T1 Sludge



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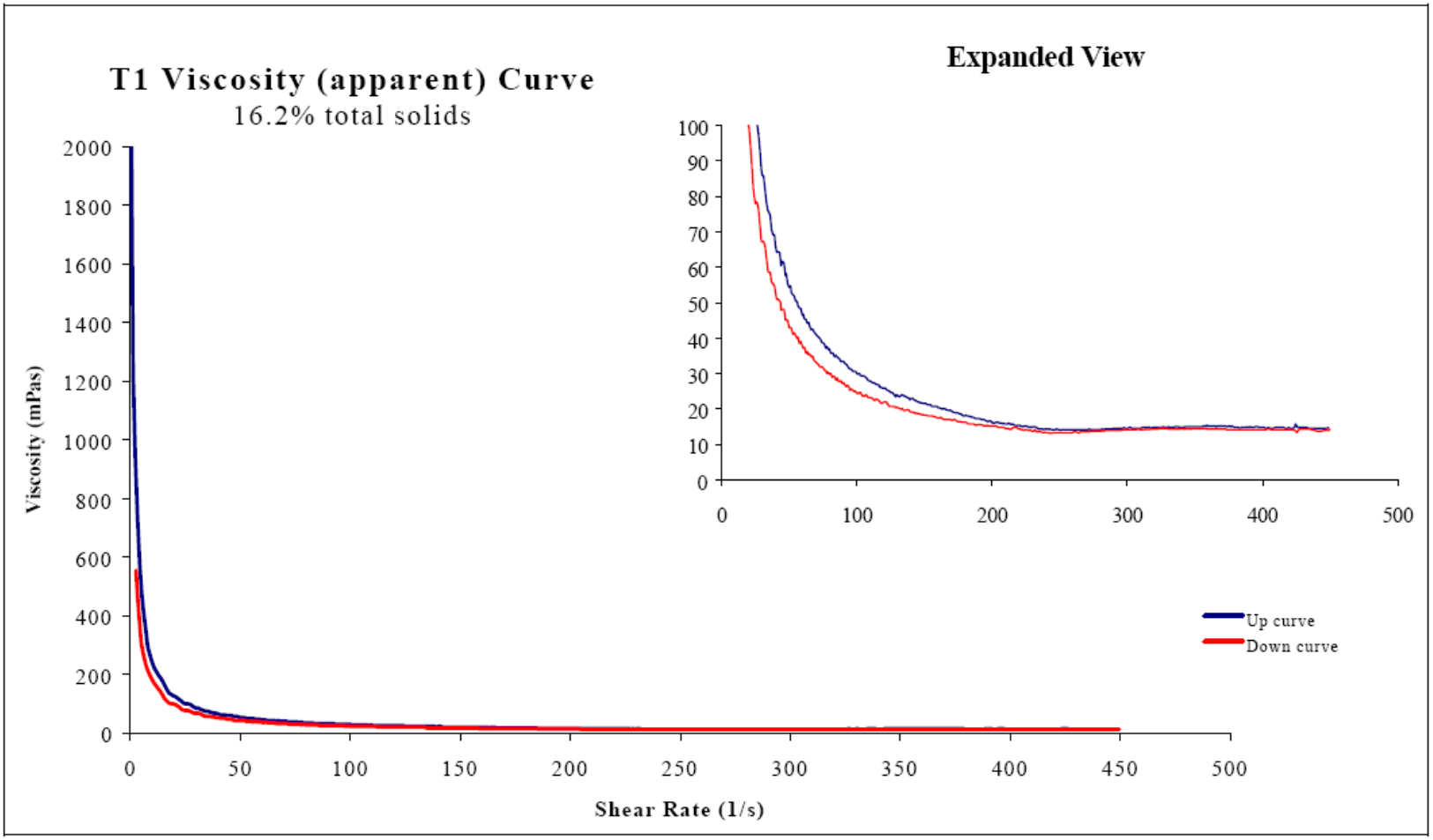
OHF Sludge-1:1 Dilution w/ SN

Figure 15 Viscosity vs. Shear Rate for T1 Sludge Composite



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Waste Package/Transportation $

- LLW Waste Package cost lower than 72-B
  - LLW liners (~$7k each)
  - 72-B canisters (~$15k each)
- Transportation costs to NTS are less than half the cost of 72-B cask shipping costs to WIPP
  - Type A cask to NTS (~$15k/trip)
  - 72-B shipping cask to WIPP (~$26k/trip)
- Total cost for LLW waste packages and transport is less than RH cost even if twice the LLW packages and shipments are required

D&D Costs

- Lowest D&D Cost option
- SN system (already contaminated) is modified for SL solidification
- SL systems and areas are not contaminated
- SL equipment can be used as spares for the SN systems

RH Debris Synergies-DOE

- Lowest risk of RH-Debris shipment disruption
- Enables 72-B cask fleet to meet DOE complex needs
- Eliminates handling/disposal of 1,600 RH Canisters at WIPP
- RH Debris operations throughput enhancements (e.g., additional shifts) could be achieved now that RH-Debris operations are not constrained by RH TRU SL
- TWPC better suited to handle additional RH Debris from other sites if DOE elects to centralize RH certification

Process Risk

- Lowest process risk
- Solidification is a mature and established technology
- Uses existing reliable SN equipment, with minor modifications, and additional simple poweder handling equipment & shielding
- Some risk that conversion of the Jaygo SN Dryer to a batch mixer may not provide adequate mixing
  - Bench scale testing on actual sludge and pilot scale mixing tests with surrogate needed to confirm that mixing is adequate
  - SN Dryer has a heavy duty shaft, ribbon style mixing blades, thick vessel wall, heavy duty pillar block bearings, custom live-loaded packing glands on shaft seals along with oversized gearbox/motor
  - SN Dryer is very similar to the Jaygo mixers used at Fernald for Silos 1 & 2 solidification campaign

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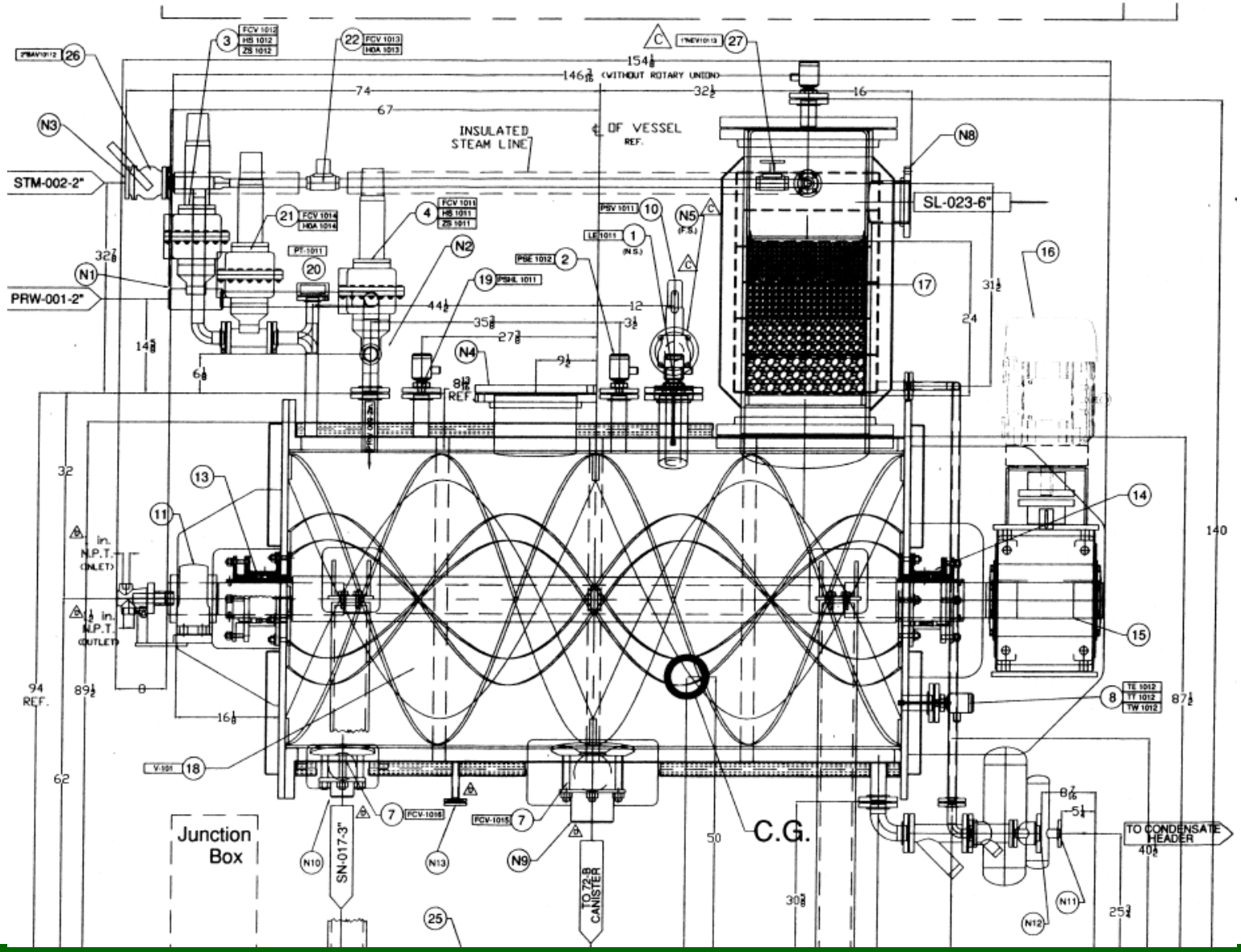

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Process Risk: W-23

Recently provided isotopic estimates of W-23 SL

- Preliminary isotopic estimates of "New Generation" SL in W-23 containing dissolved sources are much higher than MVST mean values for Pu-241(non-TRU) and Am-241(TRU)
- Sludge mass in W-23 is lower than other tanks
- Total TRU curies in W-23 only 40% above average TRU curies per tank
- TRU level increasing each year of delay
  - ***Pu-241 decay to Am-241 (t1/2 = 14 years)***
- *May require processing, packaging, certification and disposal as RH-TRU at WIPP*
- *Mitigating risk by collecting and analyzing samples to ensure that the solidified waste will remain LLW*

A.3.166

Process Risk (W-23 as RH-TRU)

- Solidification is adaptable to fill small liners (i.e., to fit in a 72-B canister) to allow disposal at WIPP
- 200 RH Monolith canisters from W-23 if shorter liners are filled to minimize modifications
- Minimal benefit would be achieved by direct loading of 72-B canisters since WIPP WAC Rev. 6 gross canister weight limit has been reduced from 8,000 lbs to only 4,240 lbs. and FGE limitations
- Direct loading of canisters would be weight limited
  - ***Only reduces the number of W-23 canisters to 171***

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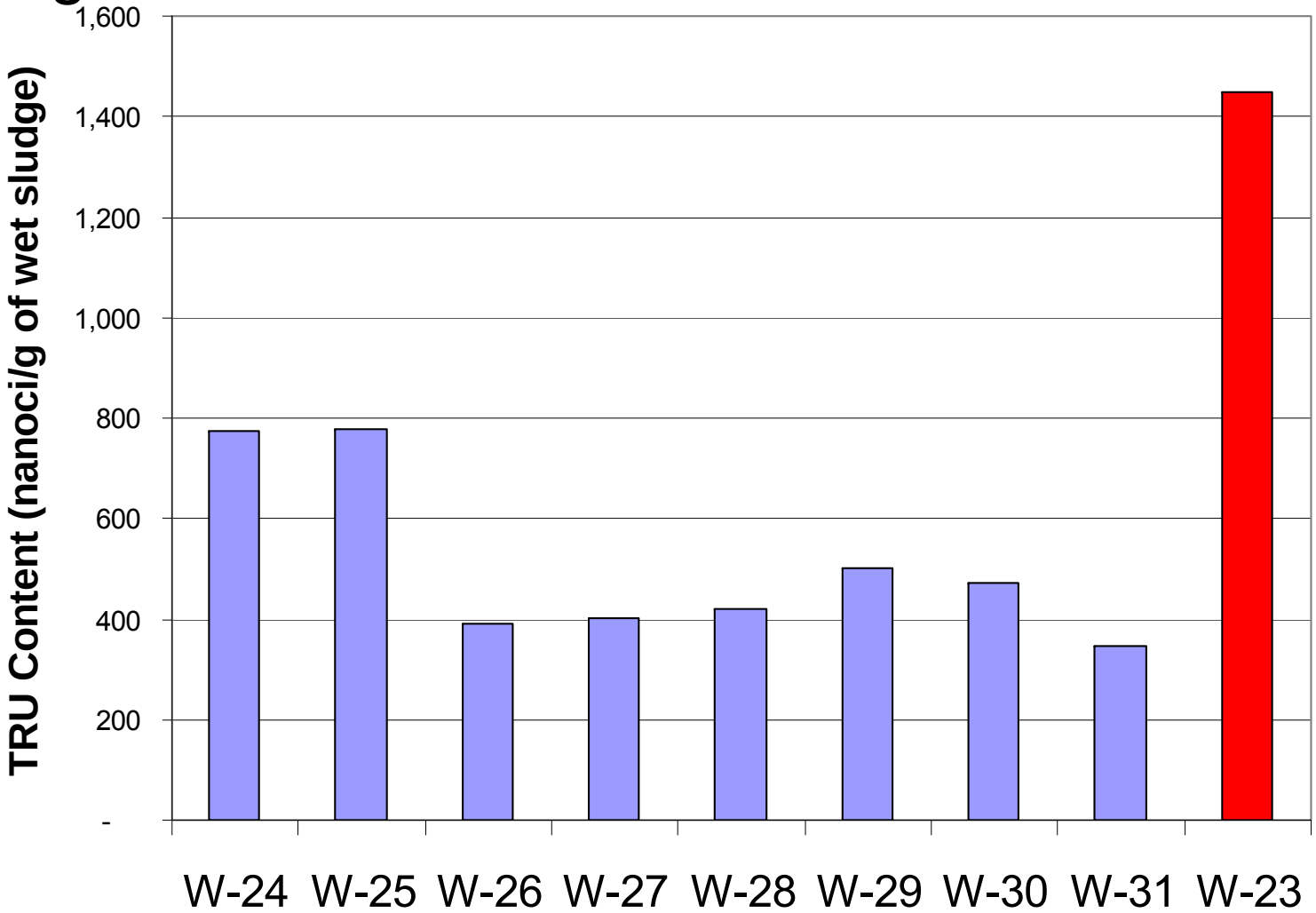
Avg. TRU Concentration in MVST/BVST Tanks



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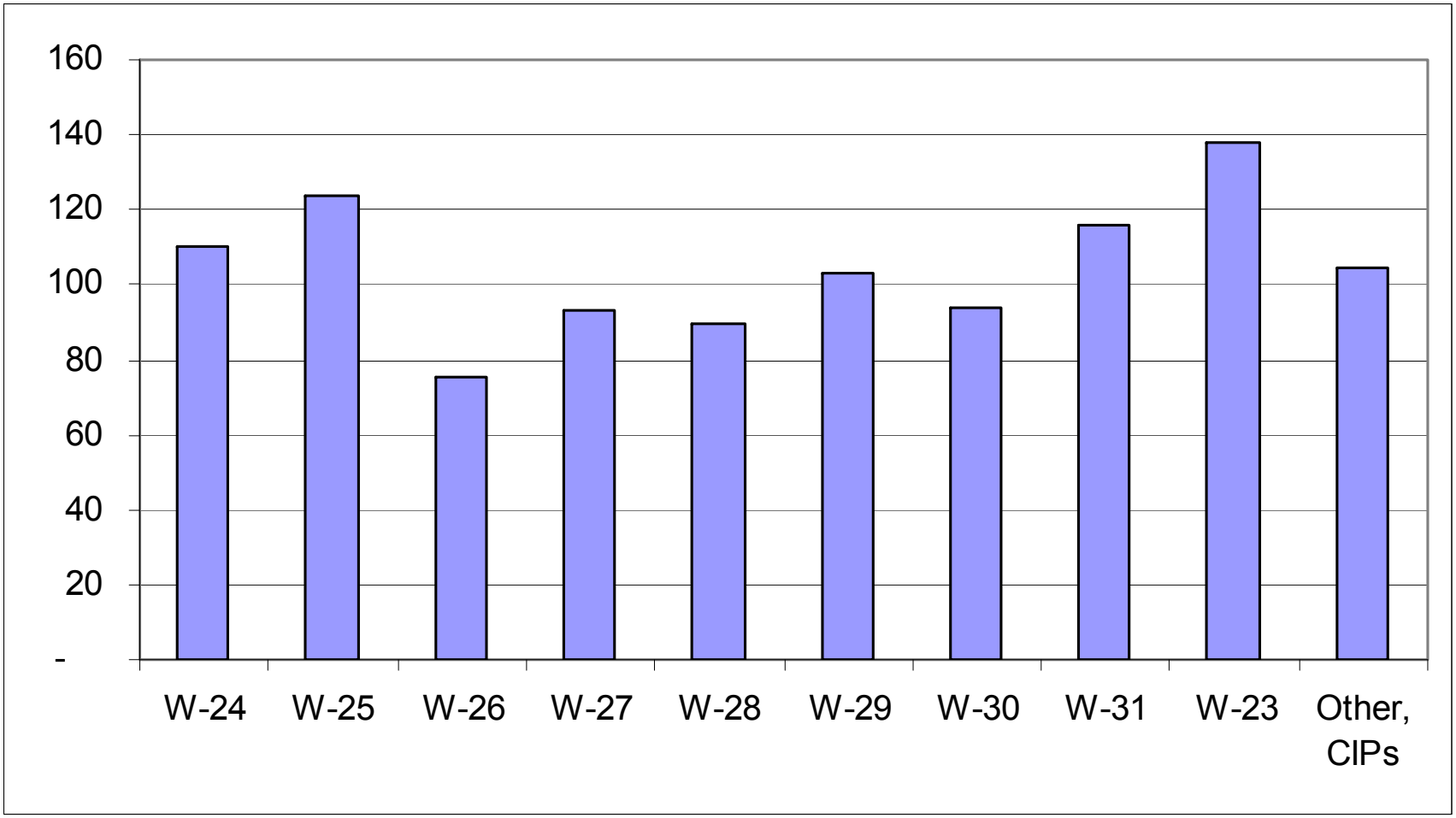
TRU Curies in MVST/BVST Tanks



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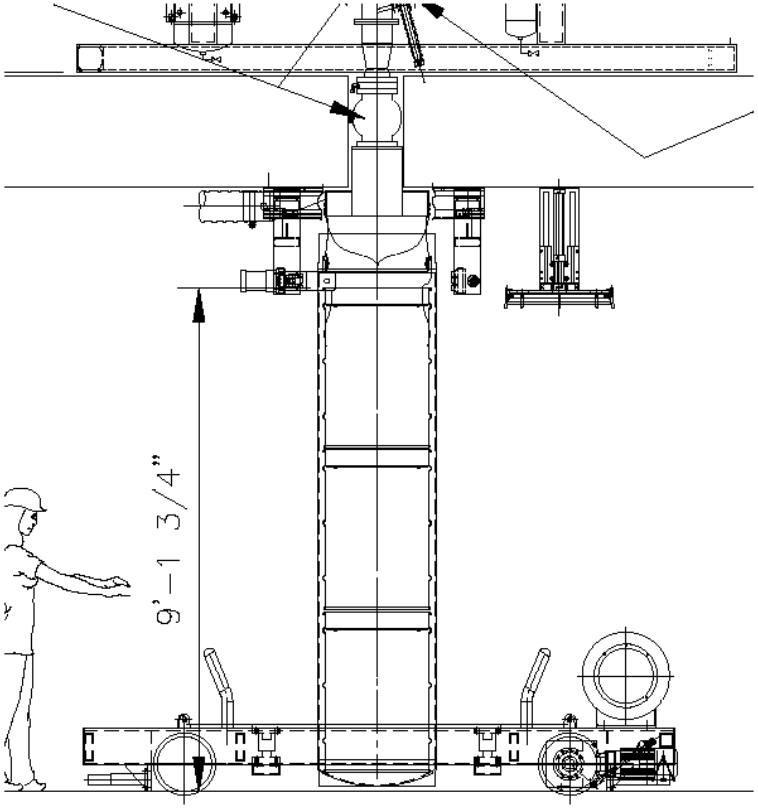
72-B Canister Liner Filling in LLW Liner Filling Station



A.3.170

Transportation Risk

- Modest increase in transport risk (e.g., potentially 50% increase in the number of cask shipments)
- However, robust, solidified, monolith provides added defense in depth against release from a transportation cask breach incident
- DOT Type A shipping casks
  - Readily available from commercial company
  - Large number of Type A casks eliminates the 72-B Cask bottleneck
  - May have to use a smaller liner at max shipping rate to allow usage of smaller Type A casks
  - Cost to build additional Type A casks (~$300k) is much lower than 72-B casks (~$1-2M), if additional casks required
- Initial transportation analysis indicates that the solidified SL meets the standard DOT Type A fissile exemption and no special permit is needed

ALARA/Criticality

- Lowest dose rates (3R/hr) on final form due to self shielding from added mass (grout/water)
- Negligible incremental airborne contamination risk vs. SN, lower airborne risk than dewatering
- Criticality remains incredible (lowest K_{eff} due to addition of boron and solidification additives)
- Simple, low risk process based on proven SN systems and extensive DOE solidification experience
- Lowest projected collective worker radiation exposure and lowest potential for internal exposure/uptake

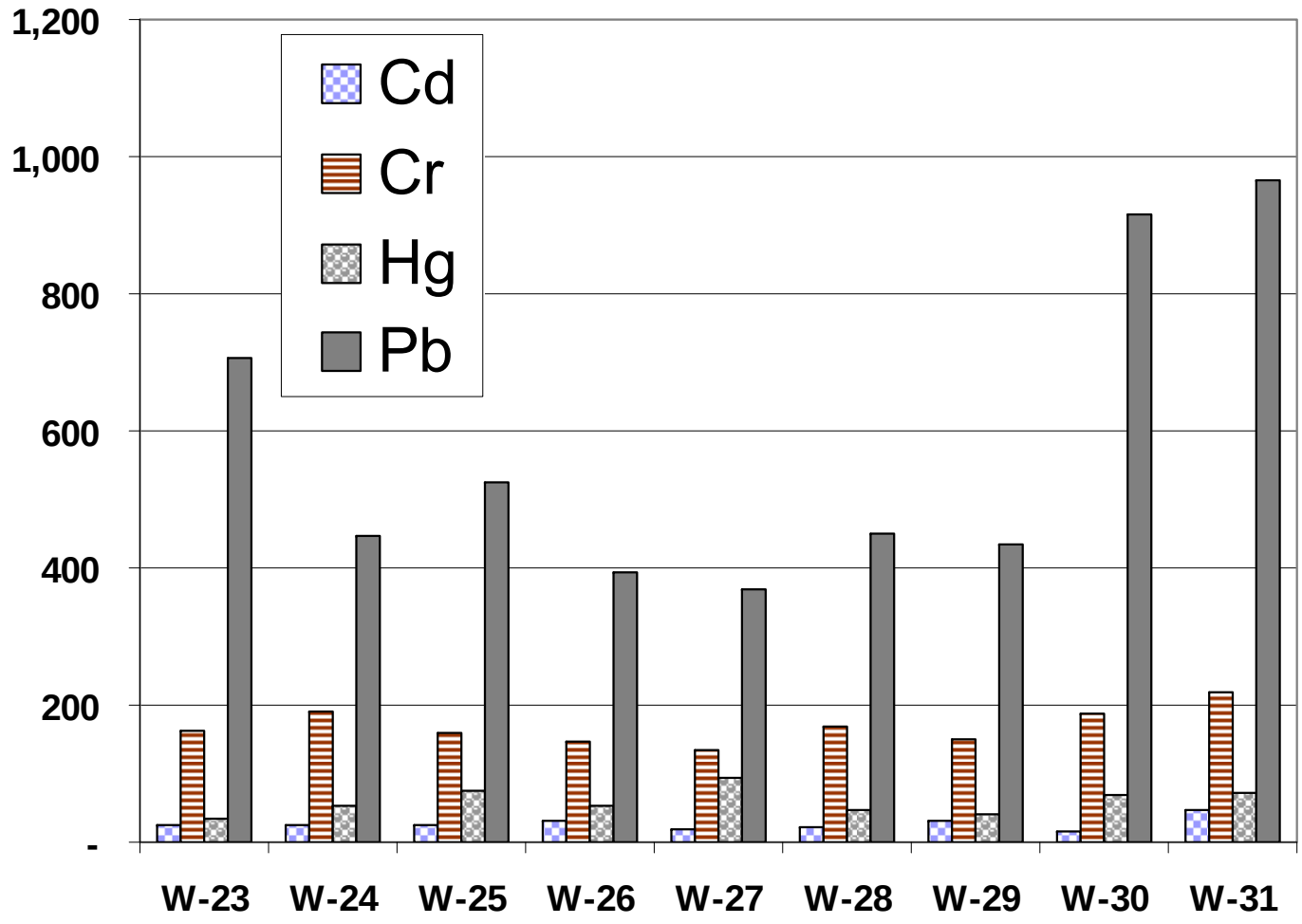
Training/Procedure Requirements

- Simple nature of the solidification process
- Use of existing SN equipment and most of the SN operations personnel are still at the TWPC
- Greatly reduced procedure and training needs
- Numerous TWPC Personnel have first hand experience solidifying this or very similar waste

Final Waste Form Attributes

- Superior final waste form
- Low dose rate (3 R/hr on contact)
- Robust, solid monolith that is no longer characteristic mixed waste
- Characteristic of toxicity eliminated by stabilizing RCRA metals (i.e., Cr, Hg, Pb,) to pass the TCLP
- Waste at “Point of Generation” is non-hazardous
- Simple verification of solid waste form (e.g., temperature profile exotherm, remote visual inspection via camera, or penetrometer)

Avg. Top 4 RCRA Metals per Tank (mg/Kg)



A.3.175

RCRA Perspective - Treatment vs. Dilution

- Not diluting the waste to avoid treatment
- RCRA metals stabilized via solidification/stabilization
- Water added to facilitate treatment (i.e., transfer, aggregation, homogenization, sampling, solidification)
- Self shielding from the water and cement reduces radiation levels and personnel exposure to radiation
- SL is fissile but over moderated, the addition of more water and the solidification agents reduces keff
- Hanford determined that adding water to mobilize and aggregate sludges was not impermissible dilution
- EPA acknowledges that dilution that is a necessary part of the treatment process, which otherwise destroys, removes, or immobilizes the hazardous constituents, is normally permissible

RCRA Perspective – TWPC part of WWTU

- Prior correspondence with TDEC confirms that the TWPC (Transuranic Waste Remediation Facility at that time) is part of the ORNL Liquid Low Level Waste System
  - RCRA Permit-by-Rule (PBR) Wastewater Treatment Unit Exclusion
  - DOE is exempt from RCRA Permitting
  - PBR notifications for changes or additions to the WWTU no longer required
  - The TWPC units are referenced in the ORNL NPDES permit application
  - As long as waste at the Point of Generation (POG) is non-hazardous, the UTS requirements are not applicable and the resulting LLW does not require disposal as mixed waste at NT
    - Note: NTS's permit for mixed waste disposal expires in 2010. Mixed LLW-GTCC generated after 2010 has no current disposition pathway (i.e. orphan waste)
    - Solidified sludge at POG must remain non-hazardous LLW

Grout Recipe

- Refine recipe from bench scale testing on actual MVST SL samples
- Dry blend similar to SRS Saltstone
  - 47% Fly Ash (Class F)
  - 47% Blast Furnace Slag (Grade 120)
  - 5% Portland Cement or MaG-Ox (MgO)
  - <1% fumed silica (powder flow agent)
- Stabilization additives (e.g., FeS for Hg)
- ORNL SN Monoliths used a blend of Portland cement, blast furnace slag, flyash, and fumed silica

Typical Grout Ingredients

Table 1: Slag Activity Index Requirements of ASTM C989

Chemical Constituents (as Oxides)	Type I Cement	Type C Fly Ash	Type F Fly Ash	GGBFS
SiO_2	21.1	33.5	43.4	40.0
Al_2O_3	4.6	22.9	18.5	13.5
CaO	65.1	27.4	4.3	39.2
MgO	4.5	4.6	0.9	3.6
Fe_2O_3	2.0	6.1	29.9	1.8
SO_3	2.8	2.8	1.2	0.2
L.O.I.	1.4	1.2	1.2	0.0

A.3.179

Benefits of Blast Furnace Slag

- Reduced setting rate/extended work time
- Lower heat of hydration
- Lower permeability and ionic diffusion rates
- Increased salt stability and metals stabilization
- Allows use of standard Type I or II Portland Cement, rather than Type IV
- Iron Sulfide (FeS) content stabilizes metals, especially mercury with low risk of overtreating (high excesses of soluble sulfides can form mercury polysulfides which are more soluble)

Benefits of Flyash

- Spherical particle shape and fine size
- Improves fluidity of grout
- Lower heat of hydration, reduces cure temperature
- Reacts with and consumes excess caustic
- Benefits to final monolith properties
  - Increased resistance to alkali-silica reactivity
  - Higher ultimate strength
  - Reduced permeability (lower leaching of metals)
  - Ion-selective material for Sr-90 Stabilization

ORNL TRU Waste Processing Center

CHARACTERISTICS

Size and Shape. Fly ash is typically finer than portland cement and lime. Fly ash consists of silt-sized particles which are generally spherical, typically ranging in size between 10 and 100 micron (Figure 1-2). These small glass spheres improve the fluidity and workability of fresh concrete. Fineness is one of the important properties contributing to the pozzolanic reactivity of fly ash.

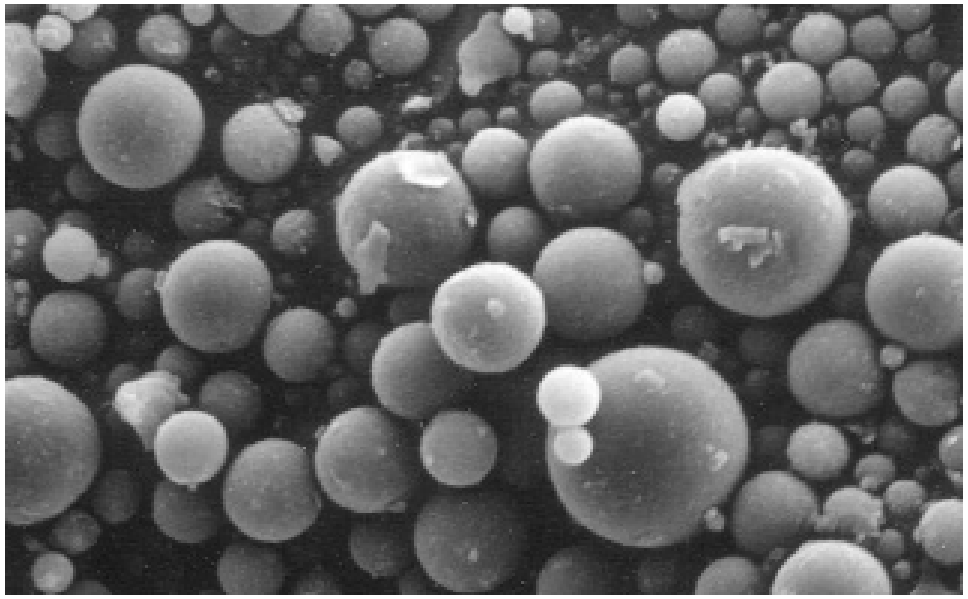


Figure 1-2. Fly ash particles at 2,000x magnification.

A.3.182

Bleed Water Control

- Bleed water (free water above grout) can occur during curing at high waste loadings
- Several options exist to control bleed water
  - Increasing dry blend to SL ratio (reduce waste loading)
  - Additives: bentonite clay, sodium silicates, polymers, methylcellulose, fumed silica
- Refine/adapt SRS “zero-bleed” grout recipes
- Multiple recipes with high dry blend to SL ratios will be developed to handle variations in SL composition and temperature to ensure no free liquid in the monolith
- Process can add anhydrous sodium metasilicate into the grout or as a monolith “cap” via Metso addition system
- Pre-load LLW liners with absorbent such as NoChar Acid Bond and Metso (Metso preload utilized during SN)

Process Control Approach

- Each “big batch/CIP tank” will be characterized for total alpha prior to solidification to ensure that the resulting monoliths will be LLW
- Confirmatory bench scale solidification testing for each big batch to confirm recipe will produce a monolith with no free liquid
- Mass ratio control via SL metering and dry blend charging weigh hopper
- Batch mixing provides high confidence level in batch composition control and LLW liner filling
- Visual observation (camera) of mixing and LLW filling to ensure grout consistency

SL Mobilization

Base Case is Pulse Fluidic Mobilization using AEAT, (now NuVision Engineering or NUVE)

SL Mobilization Alternatives

- Two Alternatives to NUVE Pulse Fluidic SL mobilization were evaluated
  - Mechanical mobilization using a remote manipulator sluicing wand
  - Chemical mobilization (in-tank dissolution of the SL using concentrated nitric acid)

BVST Tanks Project ORNL

Tank W-23 Prior to Commencement of Pulse Jet Mixing Operations

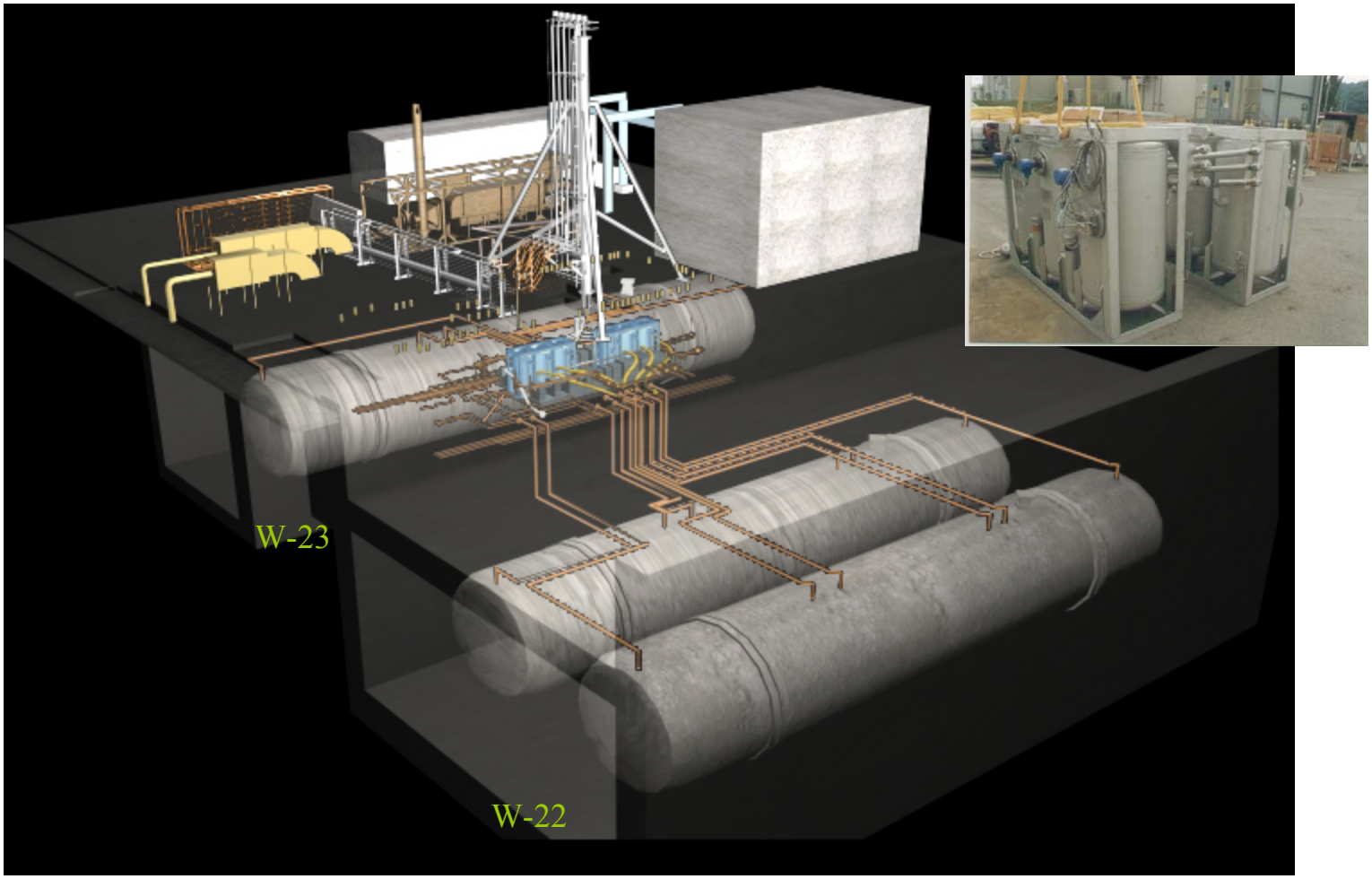
Tank W-23 After Completion of Pulse Jet Mixing Operations & Waste Retrieval

A.3.187

ORNL TRU Waste Processing Center

NuVision Tank Mobilization Systems

BVEST
W-Tank
System



A.3.188

Mechanical Mobilization Alternative

- Mechanical mobilization using a remote manipulator sluicing wand
  - Lessons learned from ORNL Gunite Tanks cite maintainability/reliability issues (e.g., wrist)
  - Requires installation of manhole tank riser
  - Custom designed system, high upfront design $$
  - Would likely require two systems to allow feeding and blending of two MVSTs
  - Challenging tank geometry and tank centerline nozzle obstructions interfere with manipulator arm travel path
  - Unique/complex system requires specialized personnel to operate and maintain
- Evaluating more versatile/robust design from SA Robotics to mitigate traditional risks

Chemical Mobilization Alternative

- Alternative to mechanical mobilization
- Chemical mobilization (in-tank dissolution of the SL using concentrated nitric acid)
- Dissolved SL transferred to CIP tanks for blending, homogenization and sampling
- Solution neutralized prior to solidification with little or no increase in disposal volume vs. mechanical mobilization

Chemical Mobilization Advantages

- Eliminates the cost/time to design, build, operate and D&D the NUVE system, ~$5M savings
- Eliminates worker dose for installing and disconnecting ~56 hoses in high rad areas
- Accelerates earliest possible SL start date by 1 yr
- Produces a solution which is easier to blend and obtain representative samples. Minimizes analytical costs.
- Compatible with boric acid (possible criticality control)
- TWPC personnel already have experience using nitric acid for SN Decon

Chemical Mobilization Disadvantages

- Cost to design, build, operate and remove an acid system
- Chemical costs (nitric acid and caustic)
- Additional worker hazard related to handling strong acid
- Reduces MVST pump stator life, purchase of spare pump required, pump replacement may be required
- Will only remove soluble SL, insoluble content (silica, alumina, rust, grit…) will remain as a “heel” in the tanks
- Monitoring of offgas humidity, acid fume concentration needed to preserve function of ventilation system/HEPAs

Chemical Mobilization System

- Covered/bermed tanker unloading area
- Covered/bermed bulk acid storage tank
- Acid metering/injection system
- Sump pump/sump tank, acid resistant coating over the floor/berm walls
- Metal frame enclosure, unfiltered ventilation, unheated, non-sprinkled

Slurry Transfer at the Idaho National Laboratory

Rick Demmer

Slurry Retrieval, Pipeline Transport, Plugging and Mixing Workshop

January 16, 2008

Idaho National Laboratory

INTEC/INL/ICPTank Farm Contacts

Co-authors of presentation

- Rick Demmer, 208-533-4277
- Frank Ward, 208-526-3010
- Mike Swenson, 208-526-3576

A.3.195

Idaho National Laboratory

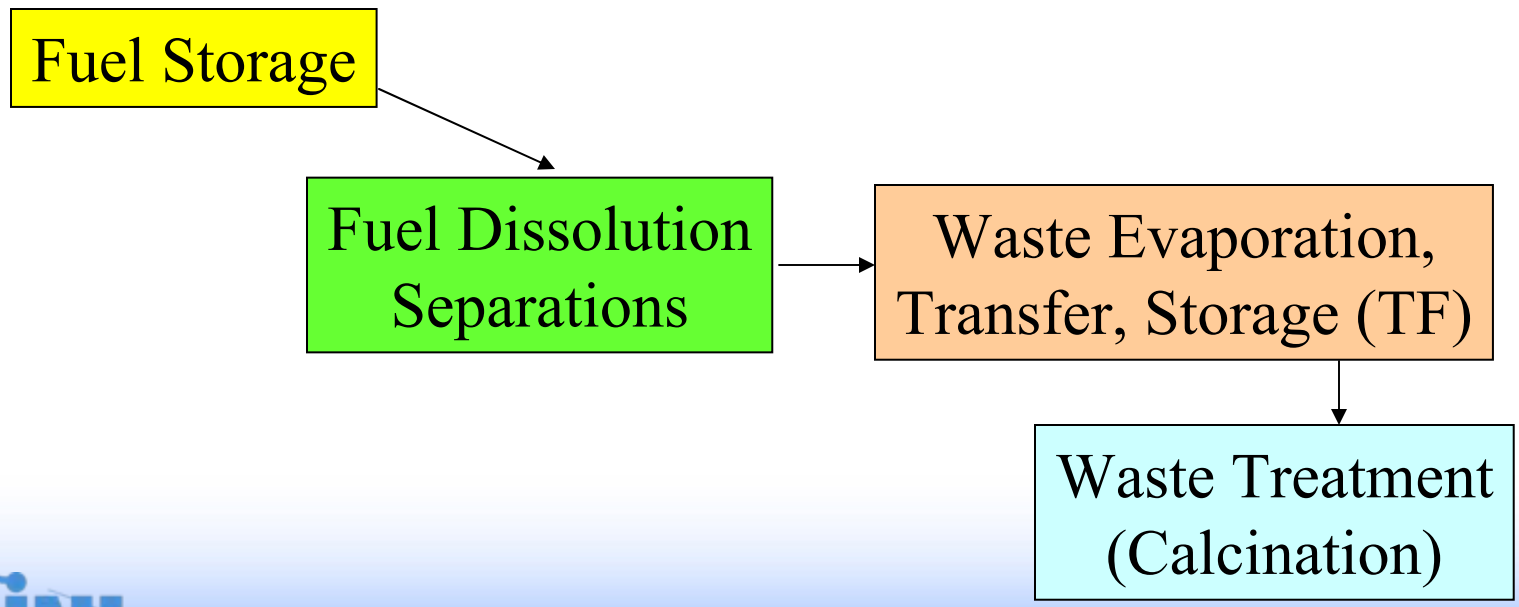
What is the INL?

AKA: INEL, INEEL

Idaho National Laboratory

Systems at the INL (ICP) Tank Farm

- INTEC was an integrated system (microcosm of problems)
  - Fuel Storage Basins
  - Fuel Dissolution and Separations
  - Waste Evaporation Transfer and Storage
  - Waste Treatment (Calcination)



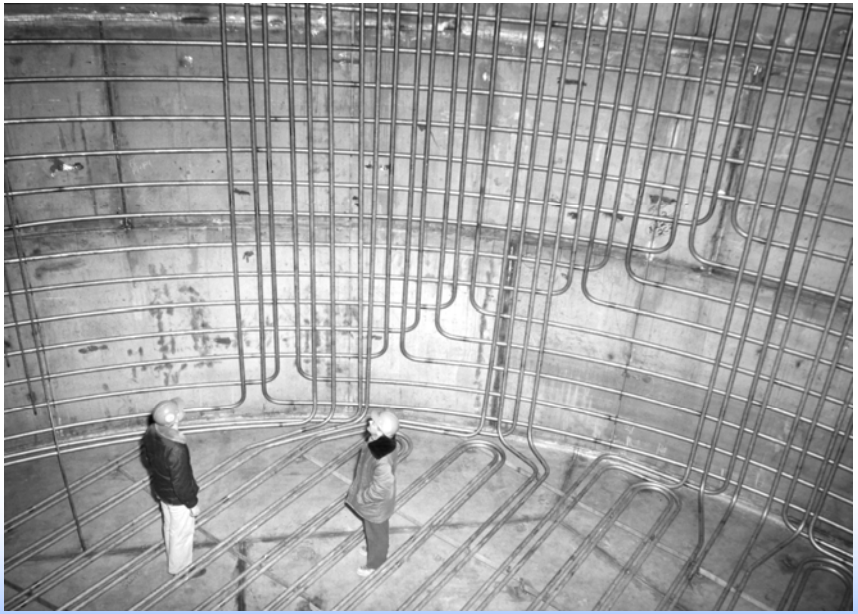
Idaho National Laboratory

Common Issues for Reprocessing/TF

- Undissolvable solids (UDS)
  - Nuclear processes create undissolvable materials
  - Nuclear Fuels have complex chemistry/metalurgy
- Continuous (no flange) piping
- Heavy duty construction (Schedule 40-160 all welded)
- Modifications to systems and dusty operations cause plugs
- Miles of “spaghetti” piping in processes
- Treatment and storage of evaporated (high density) solutions
- Chemical compatibility/instability

INL Tank Farm Differences

- Tanks (ancillary equipment) constructed of 304 SS
- Tanks were small (300,000 gal. nominal)
- Tank Farm is small (11 tanks, 8 empty now)
- Solutions not neutralized, stored as acidic
- Huge effort made **not** to precipitate solids
- Calcination chosen for treatment (some solids compatibility)
- Tanks obstruction free



A.3.200

Idaho National Laboratory

Liquid Sampling Analysis

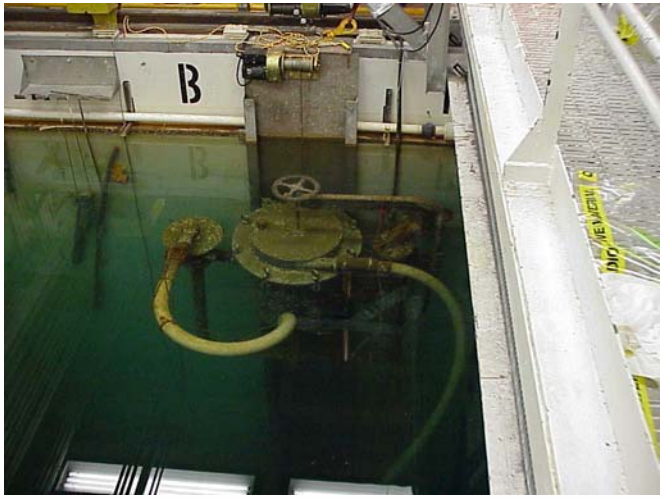
Analyte	WM-180	WM-189
	(mol/L)	(mol/L)
Acid	1.01	2.86
Sodium	2.06	2.04
Aluminum	0.663	0.711
Potassium	0.196	0.225
Sulfur	0.07	0.086
Calcium	0.047	0.073
Iron	0.022	0.027
Nitrate	5.01	6.52
Sulfate	0.05	0.19
Solids Quantity	3%	1%

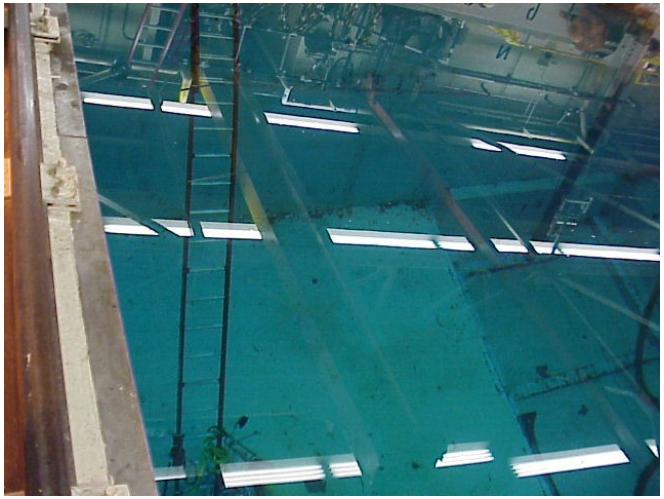
Slurry Properties

Analyte	WM-180	WM-189	Units
Intersitital Water	80	70	%
Particle Size (med)	10	30	um
Particle Dist	2-70	2-200	um
Settling rate	Slow (0.5)	Fast (5)	m/hr
S Viscosity	2.2	1.9	cP
NaNO3	62	29	%
Al(NO3)	17	27	%
K3H6Al5(PO4)8	-	13	%
KNO3	7	-	%
FePO4	-	5	%
Al2O3	5	-	%
Zr(HPO3)2	3	-	%
SiO2	-	5	%
Sr(89+90)	1.00E-04	6.00E-05	Ci/g
Cs-137	1.40E-04	2.60E-04	Ci/g
Pu-238	2.20E-06	8.70E-05	Ci/g

Spent Fuel Pools?

- Though we don't deal with them much, SFPs may be a major source of solids
  - Resins and filters from basin cleanup systems
  - Fuel material CRUD
  - Blown in dirt, debris and algae



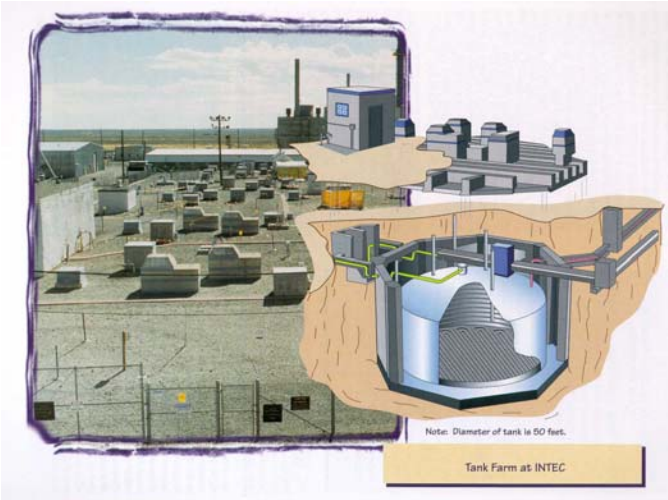


Dissolution/Separation Plugs

- Dissolution of total fuel rods, some undissolvable solids
  - Fissium type solids
  - Centrifugation for solids removal
  - Some use of filtration

INL Plugs in Tank Farm

- INL has few TF plugs
  - Acidic waste solutions (not neutralized)
  - Stainless Steel (304L) construction
  - Small amount of UDS (WM-183)
  - Alternative, redundant, backup systems
- Plugs are generally in instrument probes

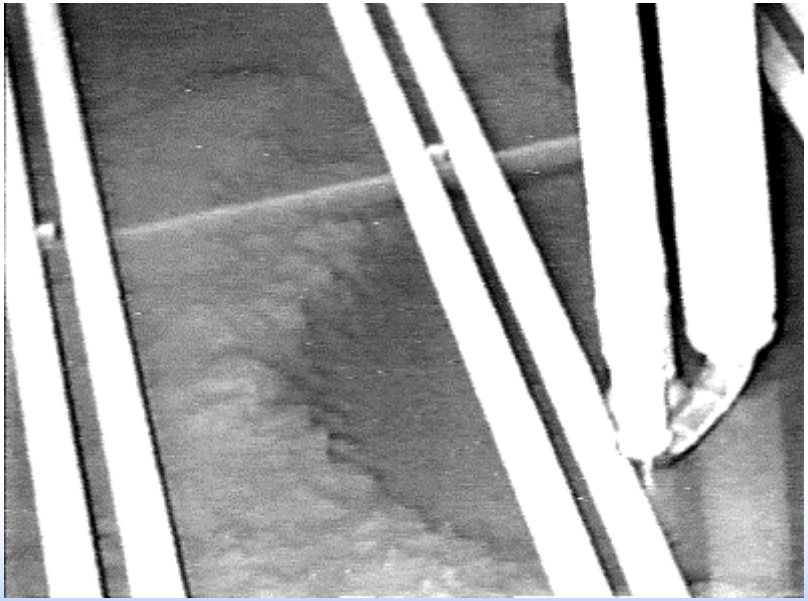


A.3.205

Idaho National Laboratory

INL TF Solids Handling + Retrieval

- Not stir up solids in tank farms
  - Static solids in bottom of tanks
- Leave in tank while treating liquid
- Specially equipped slurry transfer for retrieval
  - Wash Ball (spin jet system) and hand nozzle
  - Steam jet removal (replaced steam jet for higher flow)



A.3.206

Idaho National Laboratory

TF Plugging Case 1, Valve Positioning

- Valve on 3” pipe did not open fully (globe valve not ball valve)
- Solids clogged restricted passage
- Valve was removed and repaired, system returned to normal

A.3.207

Idaho National Laboratory

TF Case 2, Silica Gel

- Silica Gel Ruthenium Absorbers on the NWCF
- Periodically rinsed to regenerate (HNO3)
- Silica gel degenerates (decrepitates!)
- Transfer from NWCF to TF of regeneration solution caused blockage in complicated “trombone” pipe section.

Evaporator (PEW) plugs

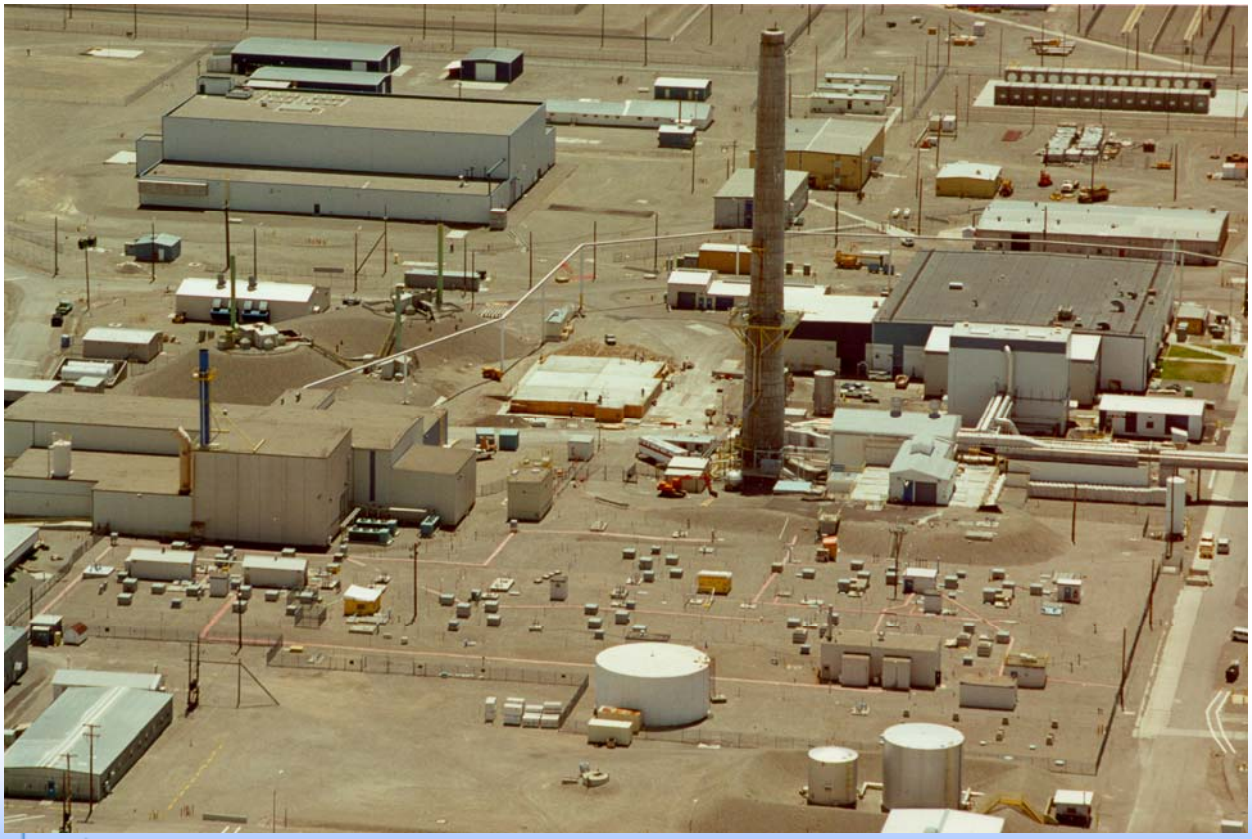
- Most low level solution waste from process equipment is sent to evaporator (PEW)
  - Thermo-siphon evaporator
  - Receives wastes from all over plant
- Complicated chemistries begin to prevail under evaporation (sometimes past solubility limits)
- Solids from concrete degradation and removal
- Evaporator systems become scaled

Calcination Plugs

- Most plugs occurred in instrumentation lines
  - Generally cleared with HP air ("minipump" 1000 psi)
- Some plugs in feed nozzles
  - Small orifice, generally campaigned with others (built-in backups)
- Some plugs in calcine system
  - Calcine agglomeration
    - Dissolution with nitric acid
    - Some pipe sections had to be removed (shutdown and cleanout)

Summary

- SS construction, redundancy, acidic solutions
- Integration of processing and management of solutions to avoid solidification



Appendix A.4

Keynote Talks

Improving Technical Success in Projects at DuPont

Jan 2008

Rick Bockrath

The miracles of science™

Improving Technical Success in Projects Agenda

- Purpose and Corporate Background
- DuPont Experience
- Keys to Success
- "High Technology" Definition
- A Call to Action
- Third-Party Reviews
- Details/Accountability
- Findings thus Far
- Questions/Discussion

A.4.2

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Purpose of Today's Discussion

- Specifically, to share changes made in our approach to managing technology risk in all of our projects in the hopes that this interaction may help us and others learn how to improve our processes.
- To share DuPont culture, history, perspective, and processes around bringing new technology to commercialization.

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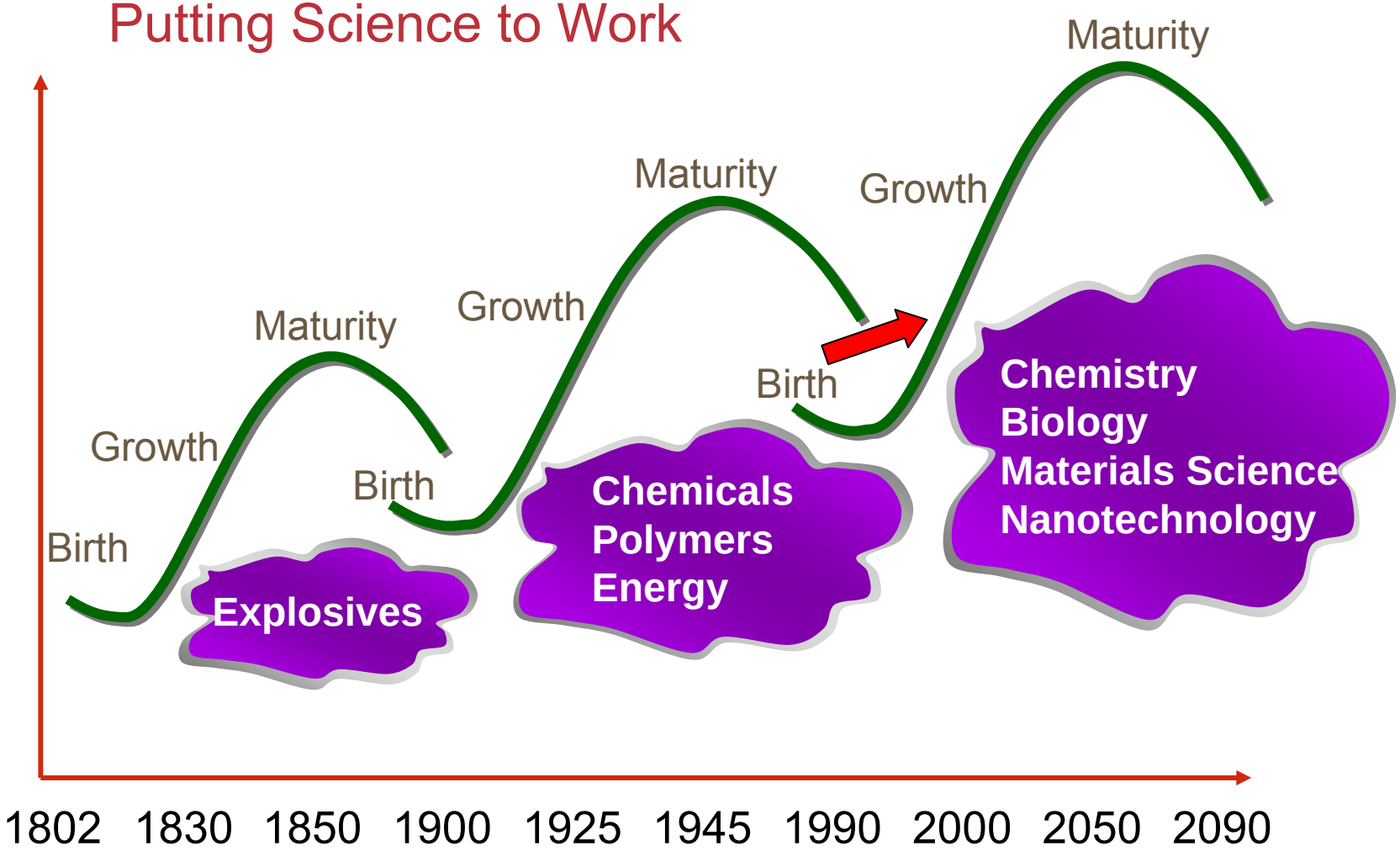
DuPont Innovation Over Two Centuries

Putting Science to Work



A.4.4

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Innovation Across 3 Centuries at DuPont

Hagley Powder Mills

"Birthplace of DuPont"

DuPont Chemicals & Polymers

- Nylon, Spandex, Teflon®,Tyvek®, Kevlar®, Nomex®, Sontara®, Corian®, TiO_2, Suva®, etc.
- Engineering Resins – Delrin®, Zytel®, Rynite® Surlyn®
- Elastomers - Neoprene, Viton®, Hypalon® ,Kalrez®

DuPont Leadership in Industrial Biotechnology

- Seeds: Pioneer®
- BioFuels
- Biomaterials: PDO, Sorona®, Hytrel®

A.4.5

Five Growth Platforms

DuPont Electronic & Communication Technologies

$3.8 B

- Electronic Technologies
- Displays Technologies
- Imaging Technologies
- Fluoroproducts

DuPont Performance Materials

$6.9 B

- Engineering Polymers
- Packaging & Industrial Polymers
- DuPont-Teijin Films
- Performance Elastomers

DuPont Coatings & Color Technologies

$6.3 B

- Performance Coatings
- Titanium Technologies

DuPont Safety & Protection

$5.6 B

- Advanced Fiber Systems
- Chemical Solutions
- Nonwovens
- Safety Resources
- Surfaces

DuPont Agriculture & Nutrition

$6.3 B

- Pioneer Hi-Bred
- Crop Protection
- Nutrition & Health
- Qualicon

2006 Segment Sales

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Improving Technical Success in Projects

DuPont Experience

- Long history of technical innovation.
- Long history of economic success based on discovery and/or development of new technology.
- Today's marketplace demands continued technological advances and the ability to bring them to commercialization faster. There can be no tolerance for major failures.

A.4.7

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Improving Technical Success in Projects

DuPont Experience in 1999-2001

- **Four major projects totaling greater than $600MM were not performing or under performing.**

- **Our overall success rate in implementation of new technology projects was not satisfactory as evidenced by Independent Project Analysis benchmark data.**

A.4.8

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Consequences of Poor Technology Choices

A.4.9

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Consequences of Poor Technology Choices

After

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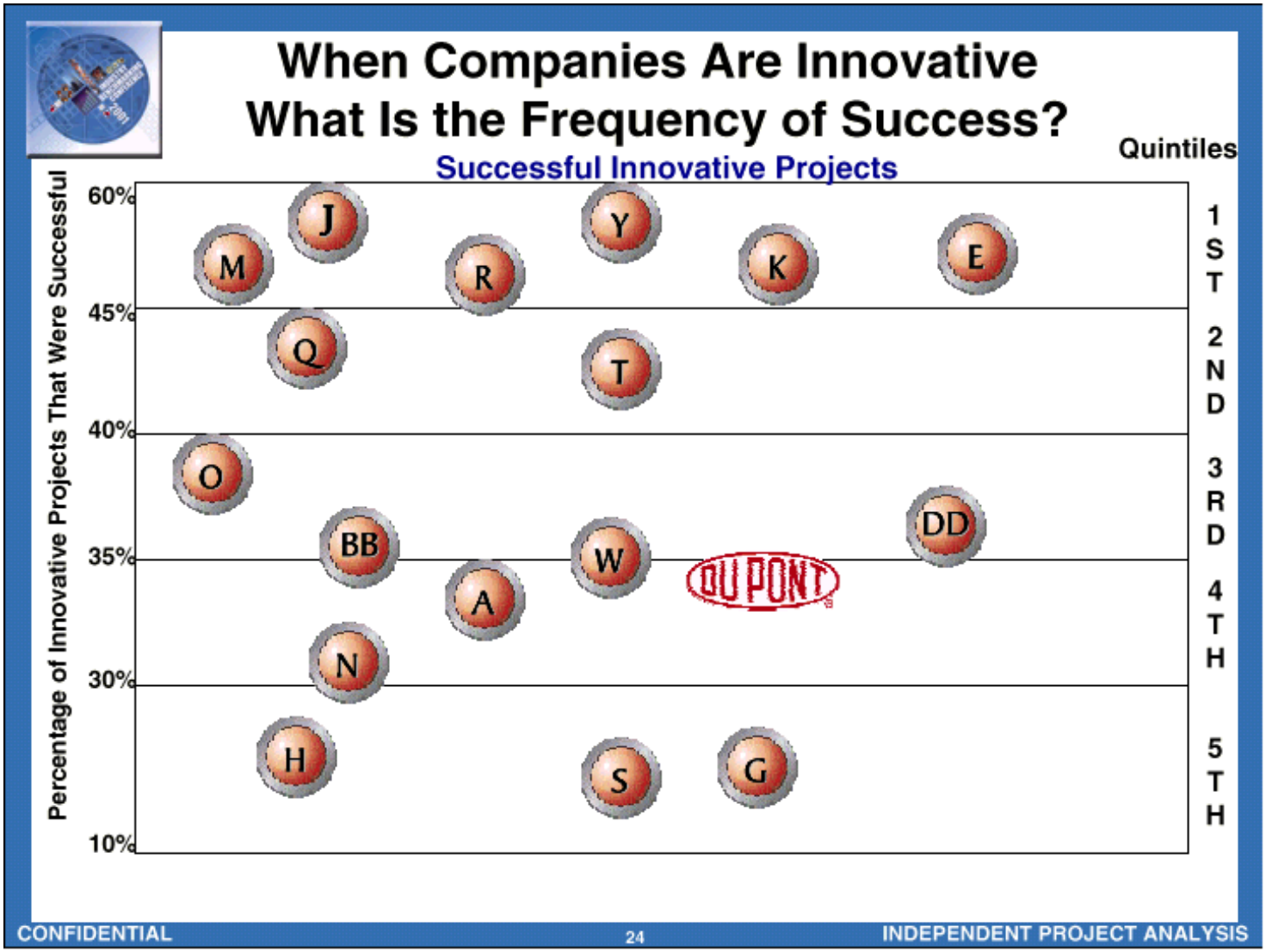
Track Record Per IPA (2001)



A.4.11

Improving Technical Success in Projects

Keys to Success

- Per Independent Project Analysis' study "Successful Commercialization of New Technology," there are four key practices critical to controlling innovation risk:
  - Recognizing innovation is present
  - Scheduling by accomplishment
  - Thoroughly defining innovation projects
  - Developing effective teams

A.4.12

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Improving Technical Success in Projects

Keys to Success

Reflection within DuPont

Those projects where teams actively pursued input from outside the natural work group rarely encountered major failures.

A.4.13

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First Step is to Recognize the True Level of Technical Difficulty in a Project

- If the technology is complex or difficult, then the challenges will be greater, plan accordingly:

  Staffing, adequate timing, realistic milestones

- Look for the signs of high technology content

A.4.14

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Signs of a High Technology Project

- High degree of new technology, including;
  - Process is new to the world, the industry or your company
  - Major process modification in 2 or more steps - especially primary reactor
  - Unit operations are known to company but not practiced in that specific sequence

A.4.15

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Signs of a High Technology Project (cont.)

- Process has more than 4 major complex steps
- Process has one or more recycle streams
  - impurity build-up, enough purge points?
- Process involves solids handling - especially moving solids
- Tough material of construction issues
- Technology knowledge makes it a "Product by Process" facility
- Specifications are complex and significant - customer qualification requirements exist

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High Technology Projects will likely have a Tough Time when:

- Flowsheets not generated/converged or
- Process only simulated on computer or
- Process only piloted at bench scale or
- Process piloted but not with integrated recycle streams
- R&D is still occurring during production design
- Market development quantities not tested with customers
- Impurities not well characterized
- Poorly understood Vapor/Liquid Equilibria
  - Tight separations, foaming potential

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High Technology Projects will likely have a Tough Time when:

- Inadequate corrosion testing if exotic materials used
  - Don't forget seals, gaskets, etc
- Significant solids handling issues
- Complex waste handling issues
- Feedstock quality is critical,
- Process uses a new catalyst or an improved catalyst
- Tough to control dynamically
- Business demands a tight timeline so R&D is compressed

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Common Outcome on High Technology Projects

- R&D takes twice as long as estimated
- Project costs 33% more than estimate
- Start-up takes 15 months longer
- Production in first year less than 50% of design
- Over 40% of moderate and high innovation projects were outright failures
- Fewer than 20% of them delivered all that was promised at authorization time – but many created great profits anyway!

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Improving Technical Success in Projects

A Call to Action!

- Team sponsored in early 2001 by the CEO and Chief Technology Officer to address technical success in capital projects at DuPont
- Coordinated by VP Engineering and directors of R&D, Operations and Engineering
- Initiated a two-stage approach

  1) Interim containment plan

  2) Longer term approach using Six Sigma methodology

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Improving Technical Success in Projects

Third-Party Reviews

- After review of history and input from many involved people, the interim containment team decided to concentrate on Third-Party Reviews.
  - Gatekeeping process by knowledgeable people outside of the business and project team
- To be utilized at minimum of two stages during the project process:

  Technology selection

  Final Basic Data review

A.4.21

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Improving Technical Success in Projects

Third-Party Reviews

The team developed:

- **Detailed process for third-party reviews along with formal audit process.**
- **Accountability definition for technology and basic-data development.**
- **List of potential reviewers across DuPont.**

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Improving Technical Success in Projects

Third-Party Reviews

Third-Party Review Definition

A review by knowledgeable people not associated with the project or business unit that can assess:

- Stage of technology development and readiness to proceed
- Potential pitfalls for project teams to address

For a major project, typically involves 6-8 people spending a day or more reviewing the state of the technology with the project team at various stages of the project.

A.4.23

Process Development Mantras

- Time is your Enemy
- If they actually give you enough time then:

  All Questions Will Be Answered in Every Case

  In enough time to plan?

  Just in time to survive?

  When doing the work over?

- Since nothing is a "given", the name of the game is prudent risk management

A.4.24

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Improving Technical Success in Projects

Third-Party Reviews

Guidelines

- Should be held early and often
- First review "before you're ready"; at least before Pre-Project Authorization (PPA)
- Final review completed after basic data and before project authorization
- More reviews for larger, complex projects
- Representation from Central Research and Central Engineering Technology organizations required

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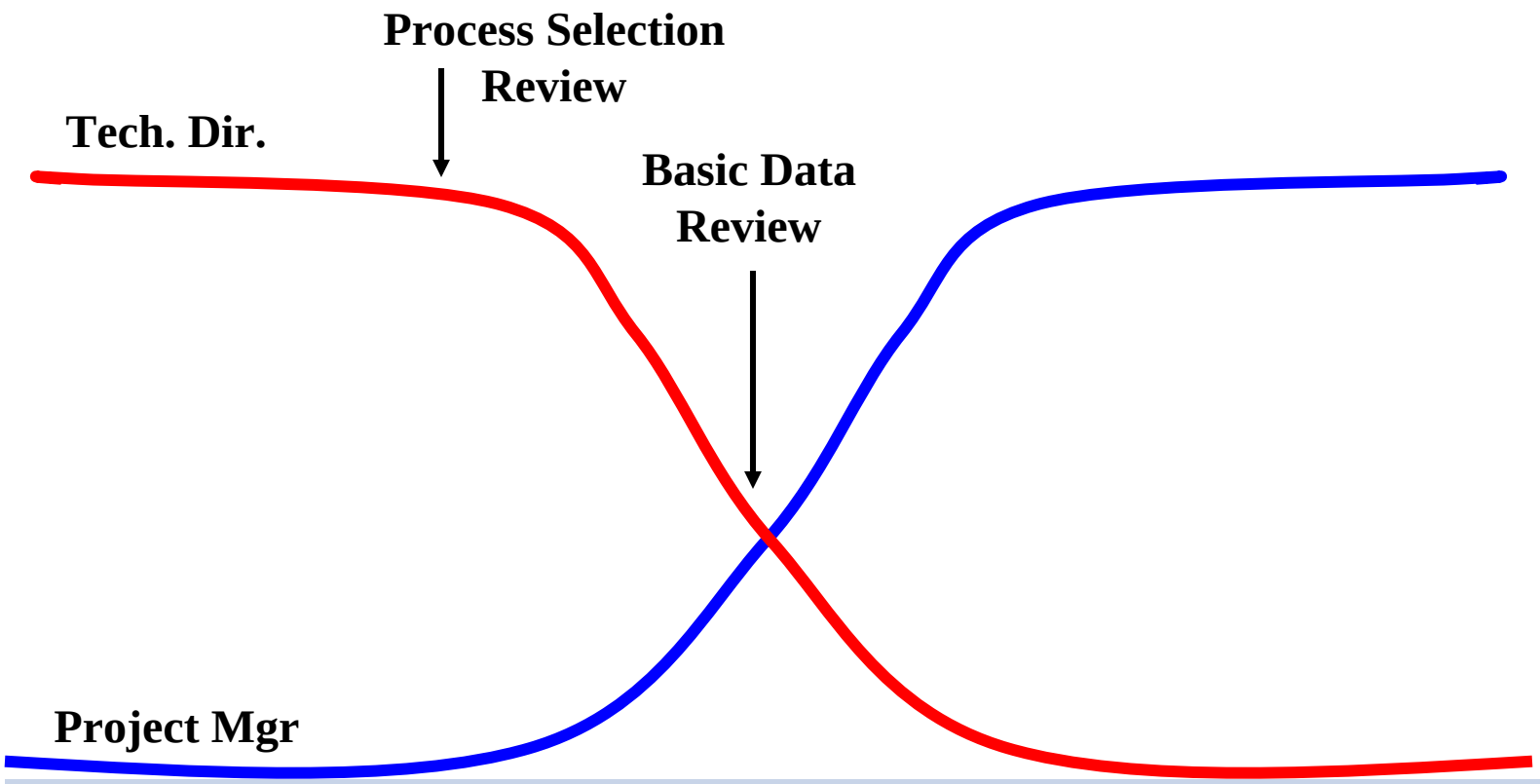
Improving Technical Success in Projects

Accountability – Technology Vs Implementation



GPI	FEL 1	FEL 2		FEL 3	
PACE	PHASE 1 (IDEA)	PHASE 2 (CONCEPT)	PHASE 3 (DEVELOPMENT)	PHASE 4 (READINESS)	PHASE 5 (SCALE-UP)

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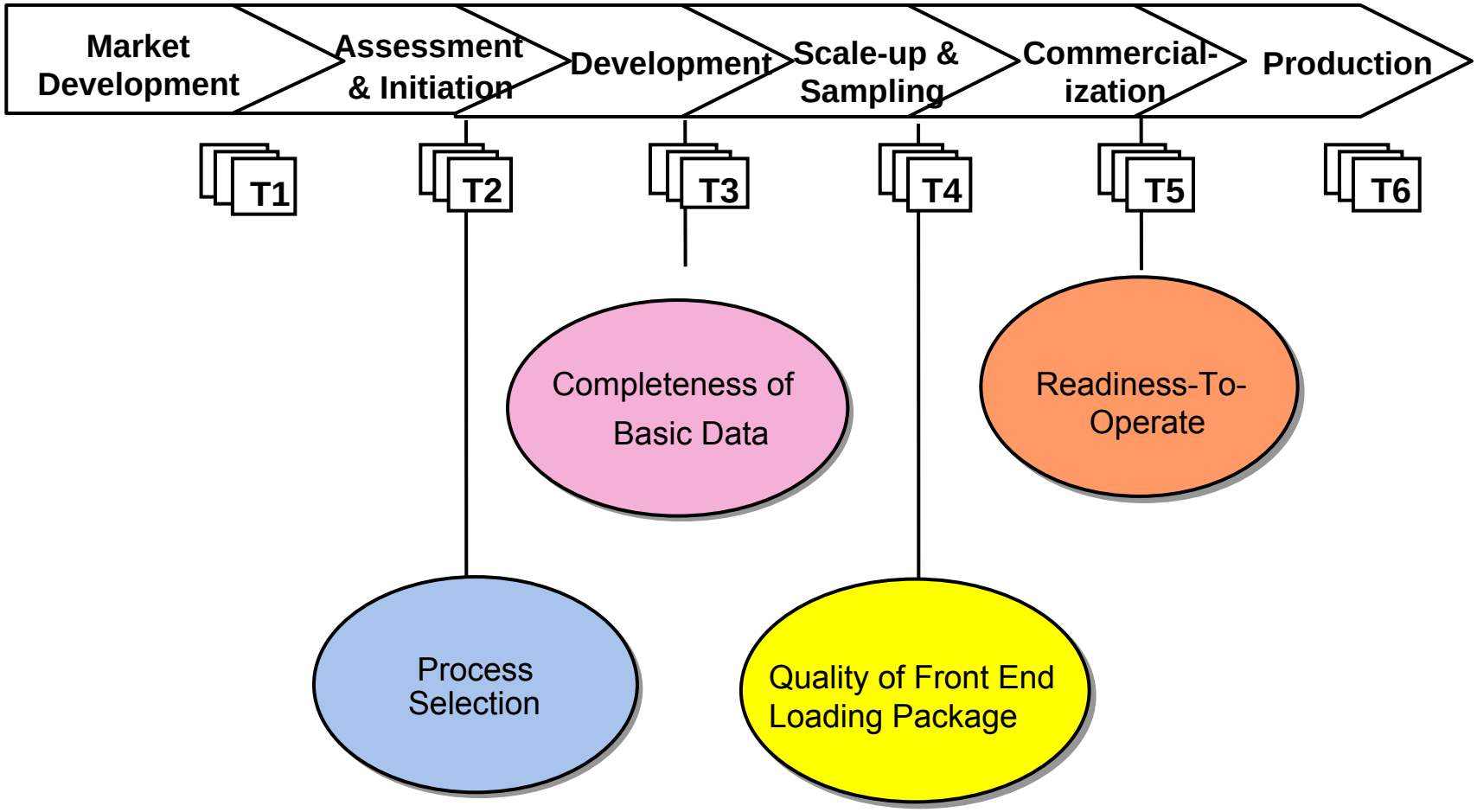
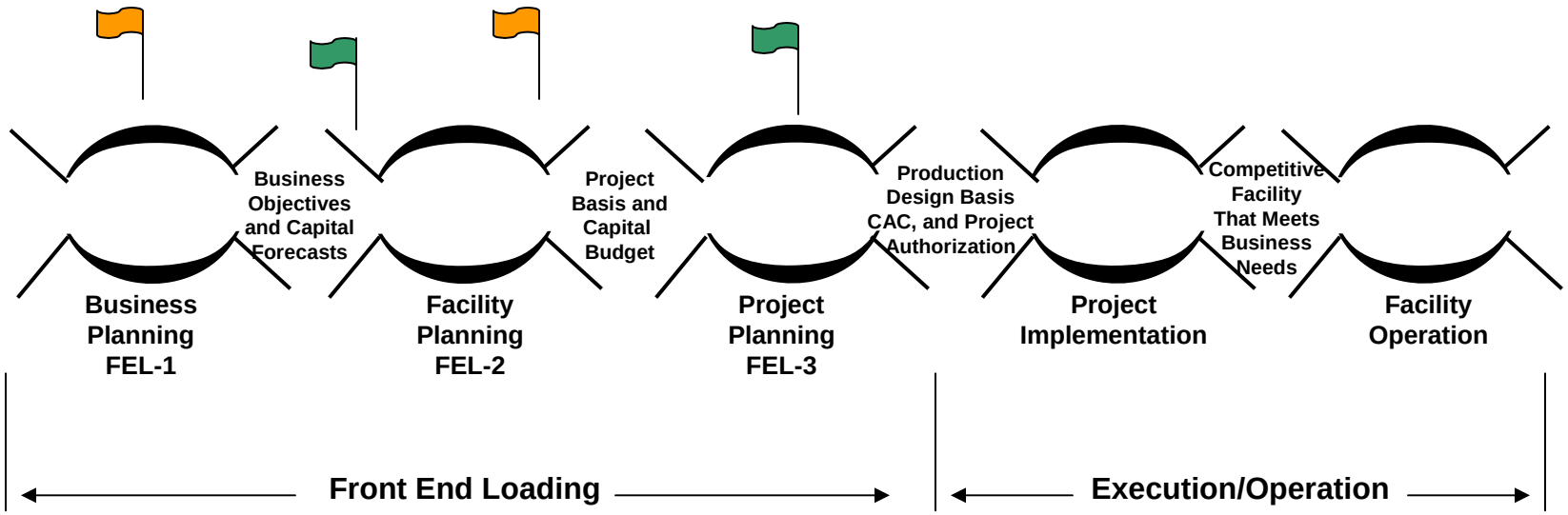
Third Party Review Timing

Integrated Application Development, Process & Product Design



Improving Technical Success in Projects

Third-Party Reviews



Minimum third-party review requirements

Additional reviews as needed for larger/more complex projects

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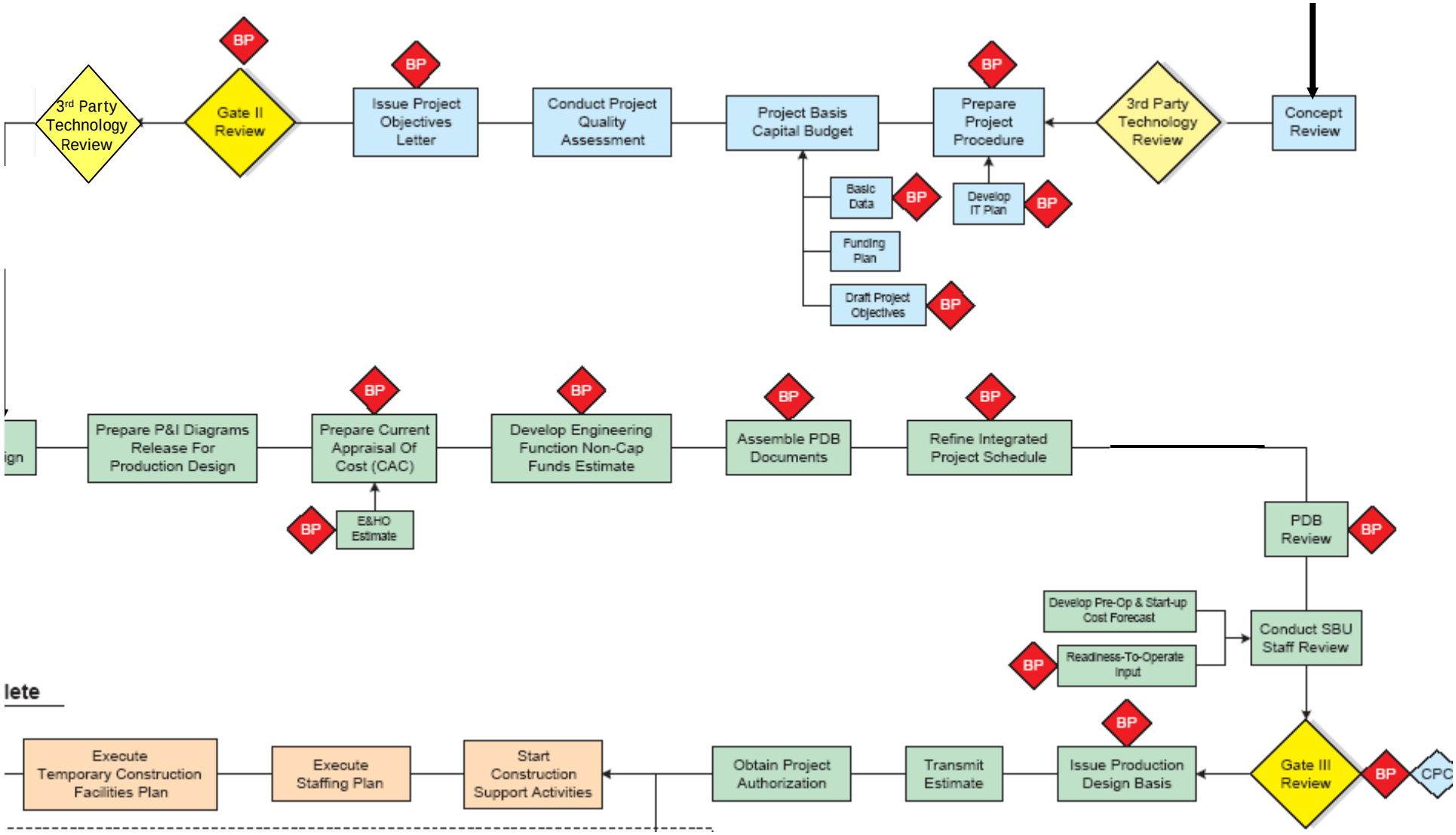
3rd Party Review Positioning



A.4.29

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Improving Technical Success in Projects

Third-Party Reviews

Incorporated into our project process and documentation

Audit process defined requiring formal review prior to authorization:

- **Engineering Technology Director for projects greater than $5MM**
- **Engineering VP, Chief Technology Officer for projects greater than $10MM**

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Improving Technical Success in Projects

- **Typical Output**
  - **Summary report with "Likelihood of Occurrence" versus "Severity of Consequence" analysis.**
- **Typical Findings**
  - **Usually identify one or two new technical issues.**
  - **By holding reviews earlier in the commercialization process, teams have time to manage the new issues.**

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Typical 3rd Party Review Ouput

Concern Number	XXX Expansion 3PR BMP 23/1212 May 9, 2006 Concerns	Business Objective Impacted	Likelihood Ranking High/Medium/ Low	Severity Ranking High/Medium/Low	Mitigation Strategy to reduce Severity and/or Liklihood
1		DCP operations	high	low	
2		shea	medium	high	
3		SHEA	low		
4		Cost	low		
5		SHEA, Capacity	medium	medium	
7		SHEA, PTO	low	high	
8		SHEA, PTO	medium	high	
9		Cost, Schedule	medium	medium	
10		Schedule	high	high	

CONCERN'S FORMAT: IF this were to happen, THEN what is the consequence

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Key Roles in 3rd PR Gatekeeping

- Central Engineering Director
  - Ensures quality 3rd PR process is in place
  - Authorizes variance via same decision-makers
- Business Technology Director
  - Ensures basic data is adequate and timely
  - Initiates & justifies variance, as appropriate
  - Directs follow-up work to mitigate risk
- Business Engineering Manager
  - Ensures all projects follow the process
  - Ensures basic data meets needs of project
  - Schedules more reviews if technology changes

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Roles & Accountability

3rd Party Review Process for New Technology Projects - RACI Diagram -

TASKS/Deliverables	PROJECT LEADER	3RD PARTY REVIEW FACILITATOR	TECHNOLOGY DIRECTOR	BUSINESS ENGINEERING MANAGER	DuET MANAGER LINK TO SBU	DuET OPERATIONS MANAGER	DuET DIRECTOR		
Gatekeeping for 3rd PR Process									
Quality process maintained in GPI, appropriately resourced			C,I	C,I	R	R	**A**		
Ensure new technology projects follow 3rdPR process	R	R	R	**A**	R	C,I	C,I		
Ensure adequate project basic data is available for 3rdPR	R		**A**	R	C,I	I	I		
or, Request for variance and documentation of basis	R		**A**	C,I	C,I	I	C,I		
Authorization of variance			C,I	C,I	R	I	**A**		
Conduct 3rd Party Reviews of New Technology Projects									
3rdPR in FEL schedule or variance requested	R	C.I	C,I	**A**	C,I	R	C,I		
Preparations completed to ensure quality review	R	**A**	C	C	R	I			
Review conducted & minutes issued	**A**	R	R	R	R	I	I		
Documentation of review, i.e. prioritized risks, followup plans	**A**	R	I	I	C,I	I	I		
Follow-up on 3rd PR Findings									
Review with business re risks & mitigation plans	**A**	C,I	C,I	C,I	C,I				
Go/no go decision on project re technical risk	R		**A**	R	C,I	I	I		
3rdPR findings to IPA - for use to set contingency, S/U timing	**A**	I	I	I	R	R			
Followup work on technology risk mitigation plans	R		**A**	C,I	C,I				
Require followup 3rdPR if change technology basis after FEL3	R		C,I	**A**	R	I	I		
Get feedback on effectiveness of 3rd PR process on project	C	I	C	C	**A**	I	I		

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Improving Technical Success in Projects

Findings thus Far

Current Status

- Practice has been in place for 6 years
- Has been utilized on 70+ projects
- Business units by and large have been receptive; some enthusiastic about incorporating this into the process
- Usually find 3+ issues that require addressing
- Three projects significantly slowed/stopped once business better understood the risks.

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The miracles of science™

INDEPENDENT PROJECT ANALYSIS, INC.

New Technology and Solids:

A Difficult Combination

U.S. DOE

Slurry Retrieval, Pipeline Transport & Plugging, and Mixing Workshop

David A. Gottschlich

January 17, 2008

Confidentiality

- **Much of the information is confidential to IPA**
- **Less detailed version of briefing will be provided later**

Purpose of this Presentation

- DOE has an ambitious and difficult waste processing mission
- Demonstrated methods will often not exist
  - New technology must be developed
- Most of the materials will not behave well
- Combination of poorly behaved materials and new technology often leads to failure
- Failure has severe consequences

Degree of Innovation Matters

- Each new step, on average, reduces operability ~10 percent in months 7 – 12 for all types of facilities
- Each new step, on average, increases startup duration by ~2 months for all types of facilities
  - ~3 months for solids processing facilities
- Facilities with three or more new steps are at much higher risk of outright failure
- More innovative and complex technologies need more extensive development facilities

Outline

- IPA background and methodology
- Industry history with similar projects
- DOE project history
- Reasons for project failure
- Best Practices for success
- Conclusions

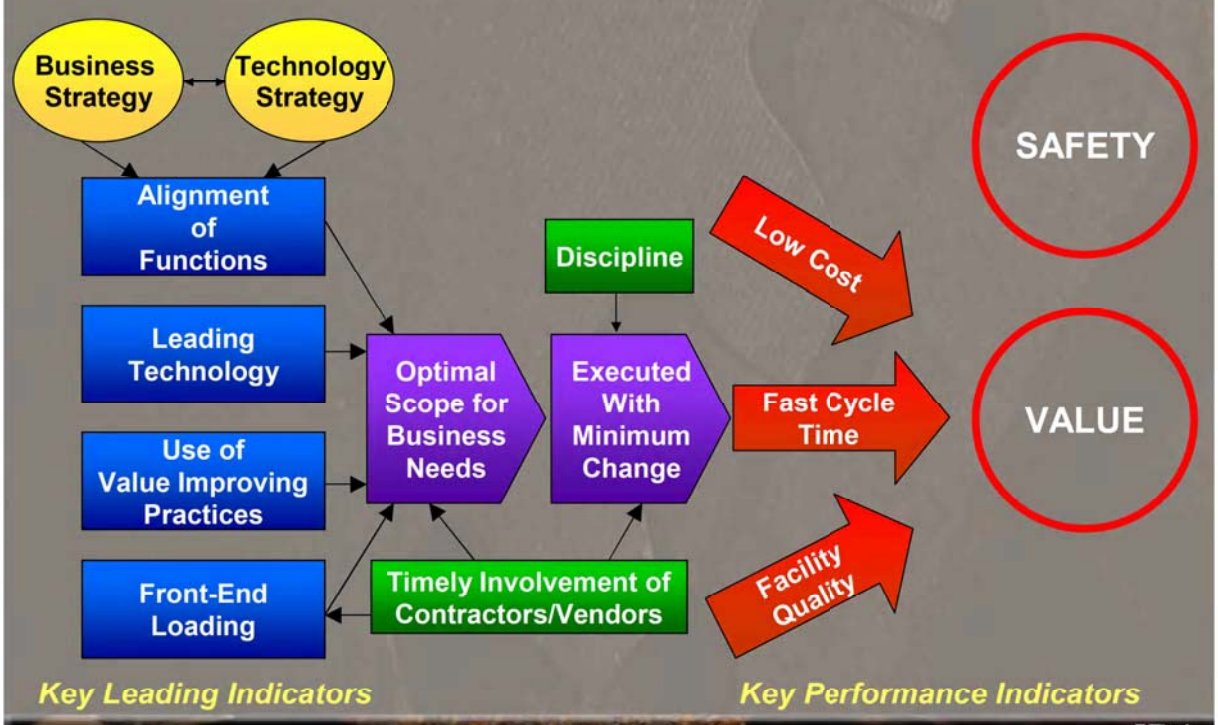
Outline

- *IPA background and methodology*
- Industry history with similar projects
- DOE project history
- Reasons for project failure
- Best Practices for success
- Conclusions

IPA Background

- Independent Project Analysis (IPA) evaluates capital projects for the processing industries around the world
  - Benchmarking and Best Practices company
  - Work for most of the major oil and chemical companies
- Started 20 years ago
  - Original work began about 10 years before that at Rand
  - Much of the original work was for DOE
- Now nearly 200 employees
- We collect detailed project histories of about 800 projects each year
- We now have detailed databases of about 11,000 process facilities

Elements of Capital Effectiveness



Database (1)

- Total of about 12,000 projects
  - Average start of execution: 1Q2000
  - Size range: $15k – $10B
  - Process facilities, non-process facilities, E&P projects, etc.
  - About 150 DOE projects (DP and WM) from the '70s, '80s, and '90s
- About 1,000 projects with new technology
- About 1,700 projects with solids processing

Database (2)

- Our goal is to examine difficulties in solids processing and innovation
- Therefore, we selected samples heavily weighted toward new technology projects
- Focus on major projects rather than smaller, plant-based projects
- We will use minerals industry as a proxy
  - Difficult materials handling
  - Materials typically cannot be characterized well—raw solids
  - Difficult processing

Some Definitions

- **Startup:** the period from mechanical completion of facilities (all units physically able to run) to routine operation
  - Facilities are making on-spec product but may not have achieved sustainable nameplate
- **Early operability:** production as a percent of nameplate capacity in months 7 through 12 after mechanical completion
- **New steps:** processing steps (chemical or physical) that are new in commercial use
  - New equipment, new match of equipment and feed, new chemical processing
- **Heat and Material Balances Known:** portion of balance equations for plant/processing train based on commercial experience

Defining New Technology (1)

- New process technology is any of the following:
  - Process chemistry that has not been used commercially
  - Incorporation of major equipment that is commercially unproven
  - New match of feed and equipment
- Scale-up of commercially proven technology is not new
- "New-to-company" is not necessarily new

Defining New Technology (2)

- IPA measures new technology in several ways:
  - Number of process steps that use new technology
  - Percentage of investment in new technology
  - Technology stepout scale

Technology Stepout Scale (1)

- Off the shelf technology
  - Project nearly duplicates proven process with modifications confined to site tailoring
- New integrations only
  - New configurations of existing, commercially proven process steps
- Minor process modification
  - One or more steps are new but order and functions of steps are unchanged from current practice

Technology Stepout Scale (2)

- Major process modification
  - Two or more steps new but core technology unchanged or can be isolated from up- and downstream
- Substantially new process
  - Analogous process may be in use but this is new path or approach
  - Core technology new and effects cannot be isolated
- All new process
  - No process like this one in use anywhere
  - Many steps new or substantially changed from prior plants

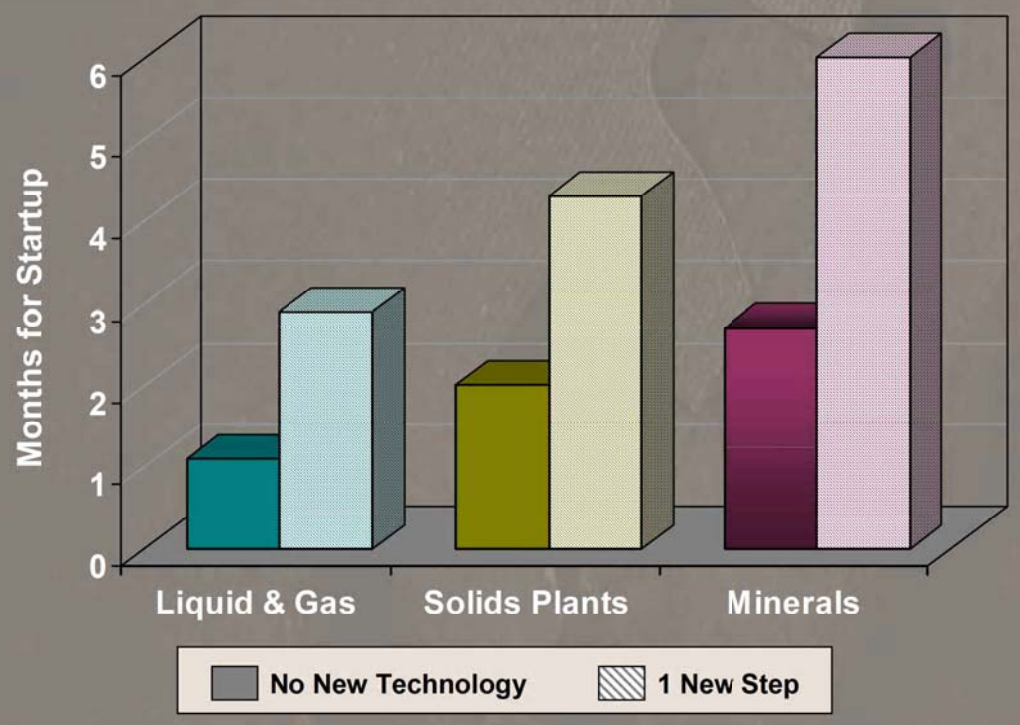
Outline

- IPA background and methodology
- *Industry history with similar projects*
- DOE project history
- Reasons for project failure
- Best Practices for success
- Conclusions

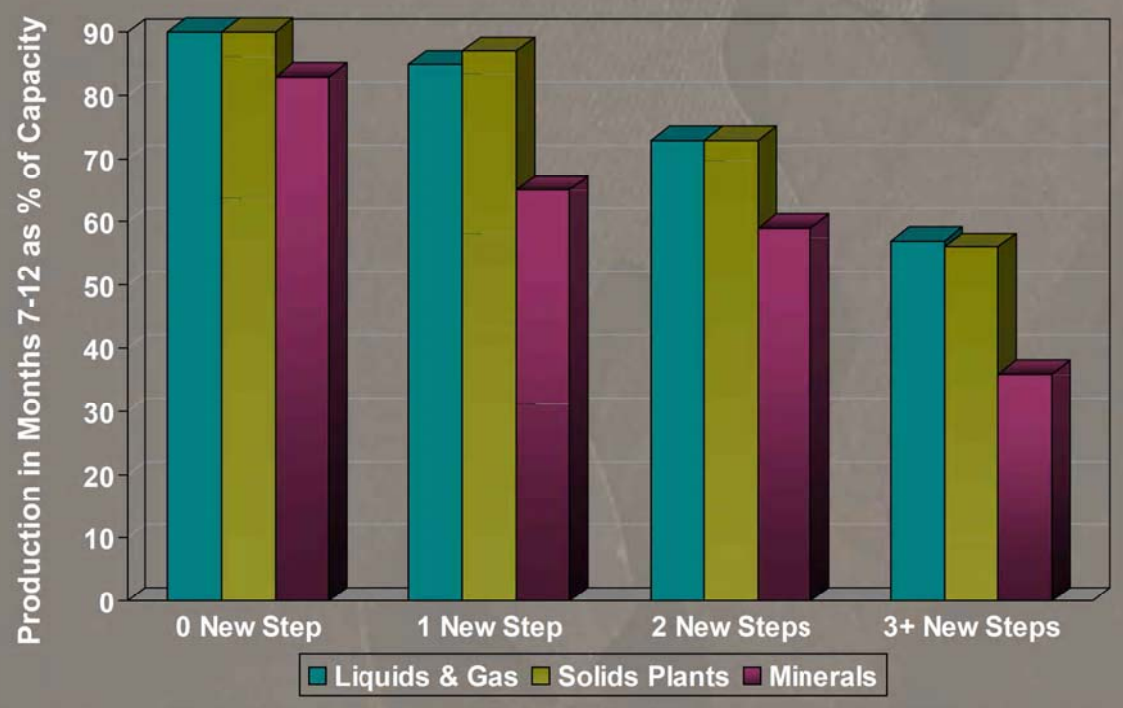
What History Tells Us...

- Many introductions of new technology have been business disasters!
- New technology substantially increases project failure chances
- Historically, new technology is linked with:
  - Much higher average cost growth
  - Difficult startups
  - Poorer operability
  - >40 percent of moderate and high innovation efforts were outright failures
  - <20 percent delivered all of what was promised
- Difficulties can be (partially) mitigated by proper understanding and management

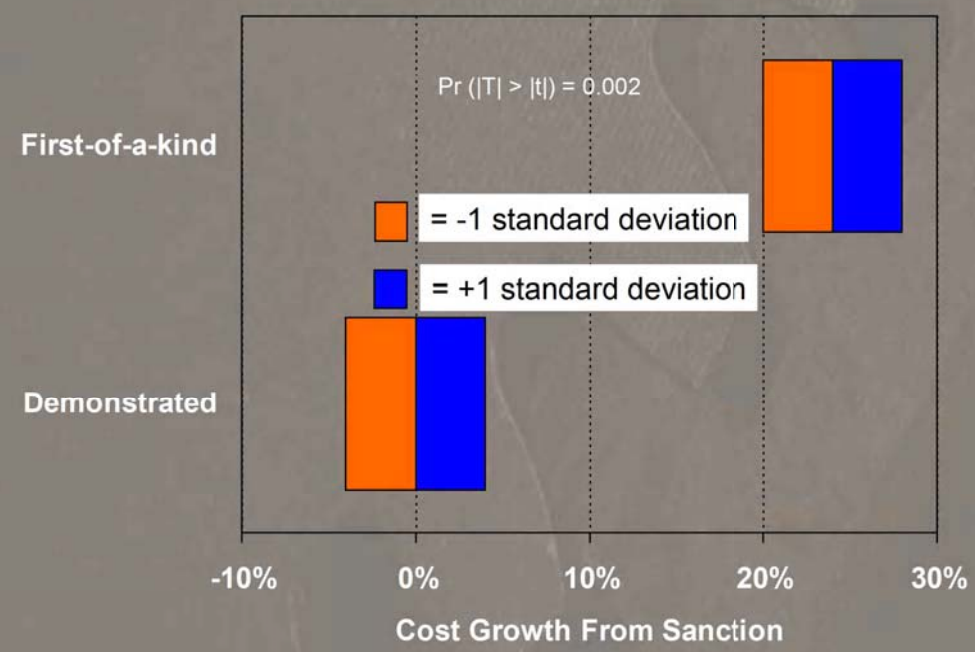
Startup Time



New Technology and Minerals Are a Difficult Mix



Cost Growths of First-of-a-Kind Processes Are Greater Than Ones Previously Demonstrated



Not controlled for other factors

What's Behind the Difficulties?

- Waste handling problems
- Feedstock impurity problems
- Weak basic technical data

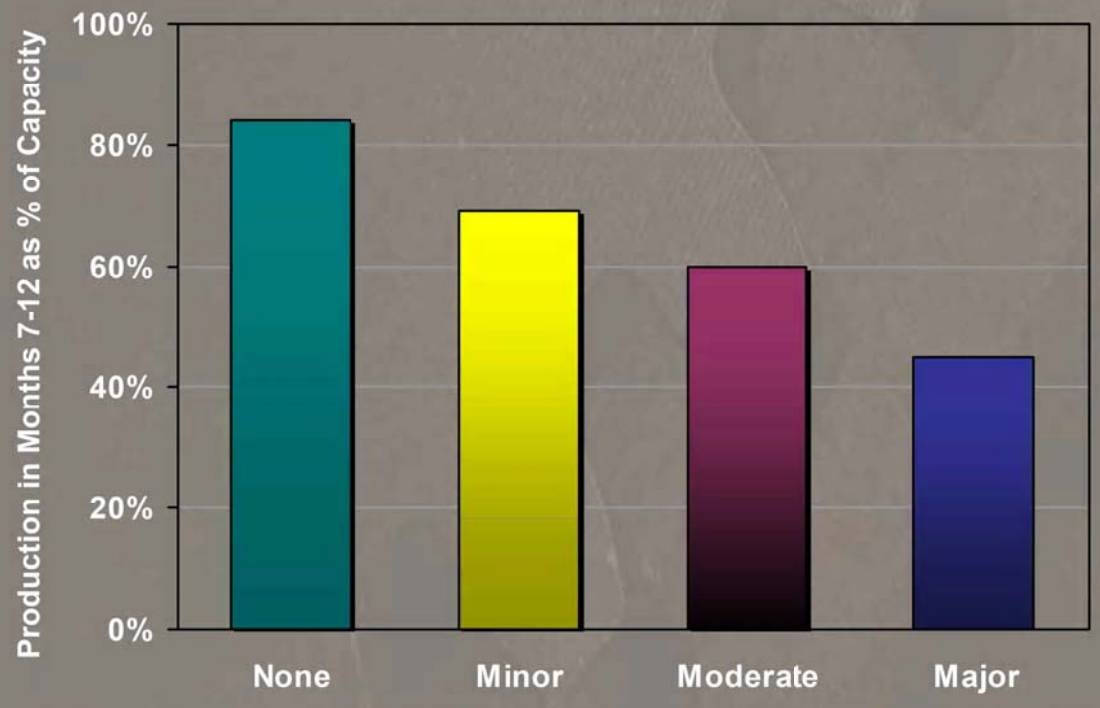
Waste Handing Problems

- 40 percent of innovative minerals processing facilities experience waste handling or treatment problems in startup
  - Versus 24 percent of other innovative process plants (pr<.01)
- In non-minerals processing plants, effects were short-lived and did not affect operability in 3rd and 4th quarters
- In minerals processing facilities, waste handling problems were associated with losses up to 22 percent of capacity in the 2nd six months (pr.<.0001)

Unexpected Impurities in Feedstocks

- Feedstock impurities are more common in minerals processing than chemical processing generally
- When they occur, they punish early operability severely, regardless of the level of innovation

Feedstock Impurity Problems Damage Performance in Minerals



Outline

- IPA background and methodology
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DOE Benchmarking

- IPA benchmarked DOE projects at least twice
  - Defense Programs in 1990
  - Waste Management in 1995
- Project performance substantially lagged Industry
  - Poor cost predictability
  - Long schedules with substantial slip
  - Long startups and below nameplate operation

Why?

- Long DOE funding cycle
  - Disrupted team continuity
  - Made FEL uncertain
- Changing objectives
- Frequent lack of commercially available solutions
- Fundamentally more difficult problems
  - But even simple projects suffered the same problems

Outline

- IPA background and methodology
- Industry history with similar projects
- DOE project history
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- Best Practices for success
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Weak Basic Technical Data

- Unlike liquids and gases, data for minerals processing do not scale well from smaller experimental facilities
- Minerals facilities are intrinsically more difficult to instrument than liquid and gas processing facilities making data less available from prior units
- Ambient conditions affect minerals handling and processing characteristics in ways that are unusual with liquids and gases
- As a result, basic technical data are weak for new technology minerals processing plants
- Consequences are seen in plant operability

Why Do New Technology Projects Fail?

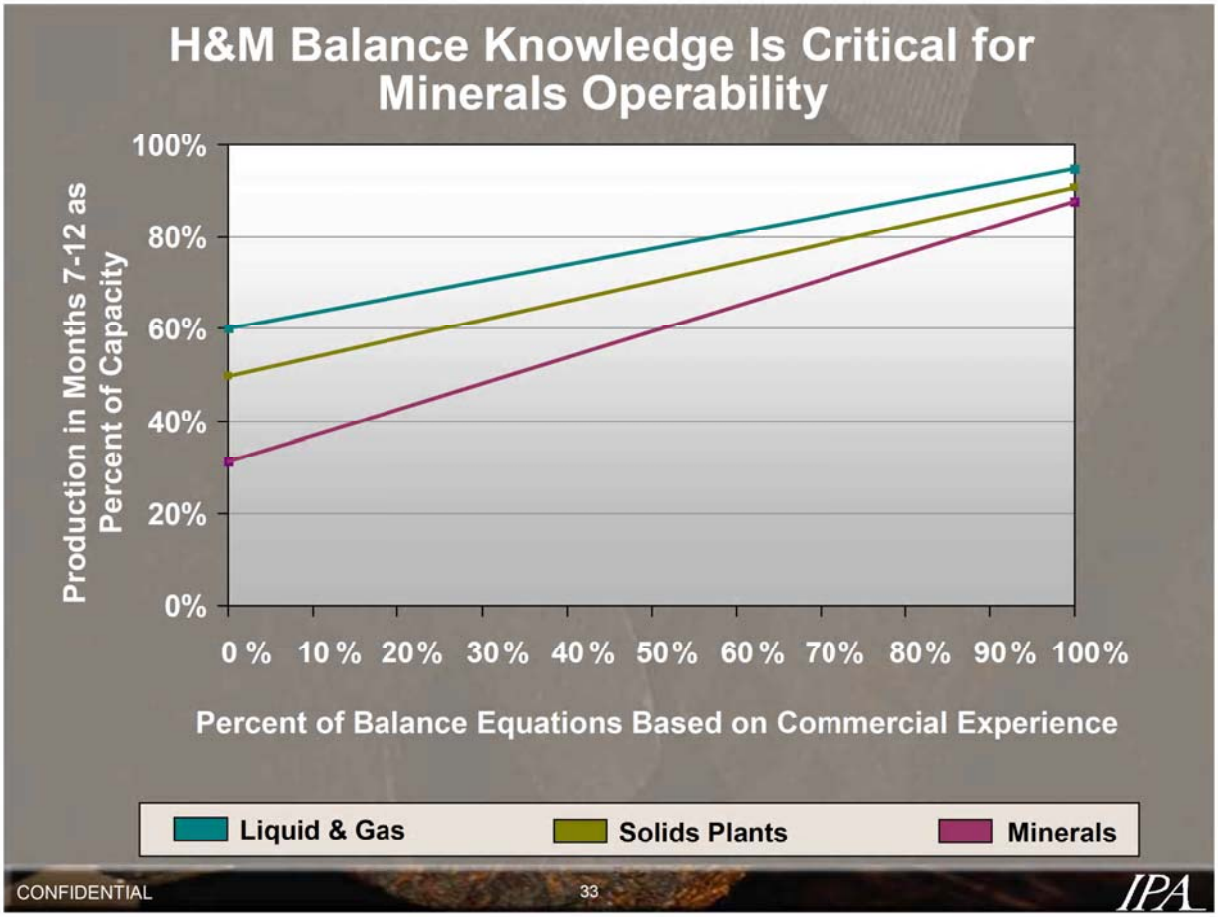
- Push for speed prevents obtaining necessary basic data
  - As a result, everything is fast-tracked except startup
  - Shortcuts that lead to failure:
    - > Skipping the integrated pilot
    - > Short-cutting FEL
    - > Accelerating execution

Basic Data Are Basic!

- If basic data are wrong, the design will be flawed, often in fundamental way
- Because basic data underpin design, design conservatism does not help
  - You cannot engineer your way out of bad basic data
- Errors in basic data are the most common cause of failed new technology projects

Good Engineering Cannot Substitute for Complete Basic Data

- Reaction products, especially side reaction products
- Separation technology verified
- Heat and mass balances established
- Materials of construction (corrosion and erosion cannot be simulated)
- Temperature and pressure control
- Engineered equipment specifications
- Mechanical equipment selection


H&M Balance Knowledge Is Critical for Minerals Operability
Production in Months 7-12 as Percent of Capacity
100%
80%
60%
40%
20%
0%
0 %
10 %
20%
30%
40 %
50%
60 %
70%
80%
90%
100%
Percent of Balance Equations Based on Commercial Experience
Liquid & Gas
Solids Plants
Minerals
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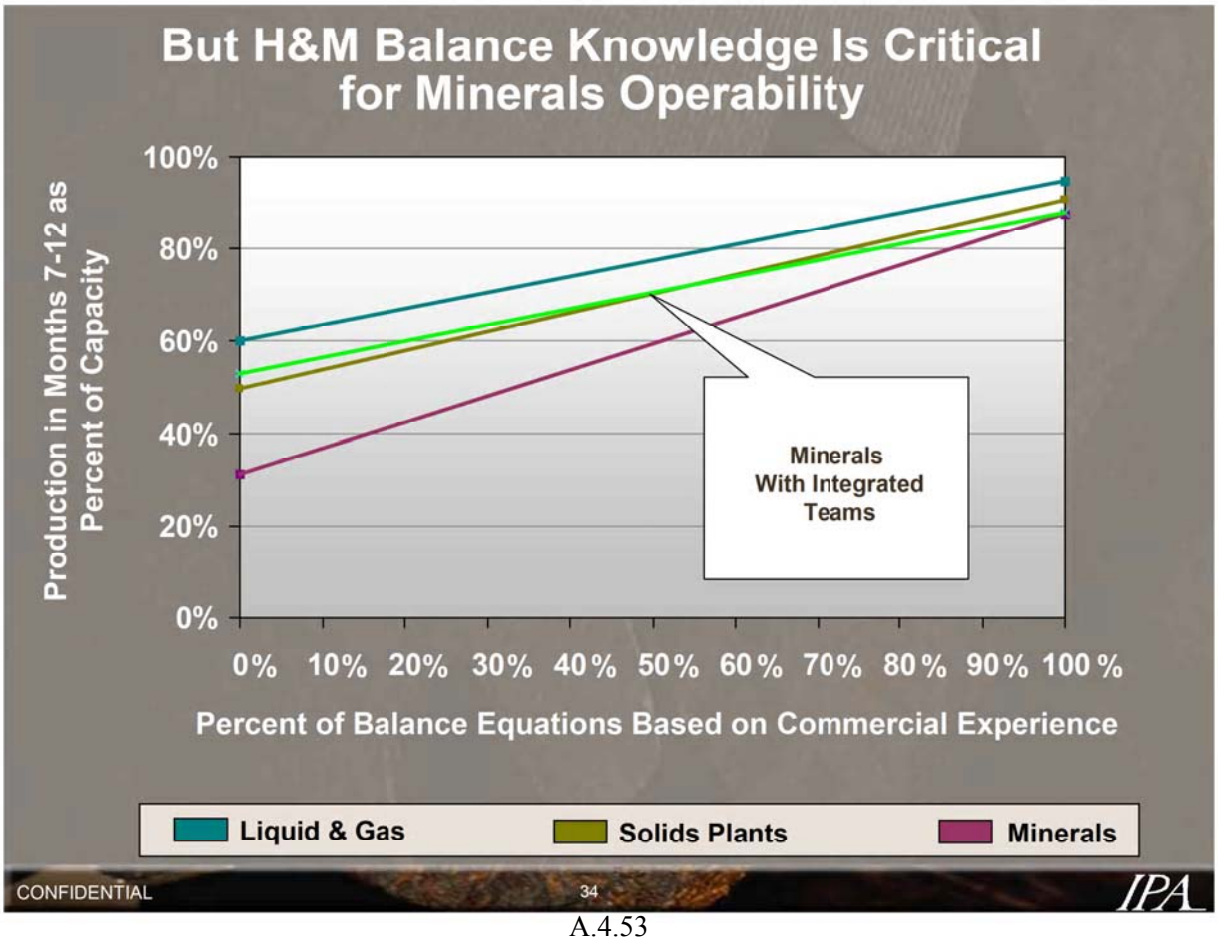
33
IPA


But H&M Balance Knowledge Is Critical for Minerals Operability
Production in Months 7-12 as Percent of Capacity
100%
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0%
0%
10%
20%
30%
40 %
50%
60 %
70%
80 %
90%
100 %
Minerals With Integrated Teams
Percent of Balance Equations Based on Commercial Experience
Liquid & Gas
Solids Plants
Minerals
CONFIDENTIAL
34
IPA

Processing Solids

- Solids hate mechanical equipment and seek to destroy it by force
- Solids processing is highly empirical
- Processing characteristics cannot be safely predicted from theory or small-scale experiments
  - Behavior of solids under process conditions often scale-dependent
    - > Fluid beds change characteristics with size
    - > Slurry reactors experience wall effects
    - > Speeds required at scale induce carryover of materials

Outline

- IPA background and methodology
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- *Best Practices for success*
- Conclusions

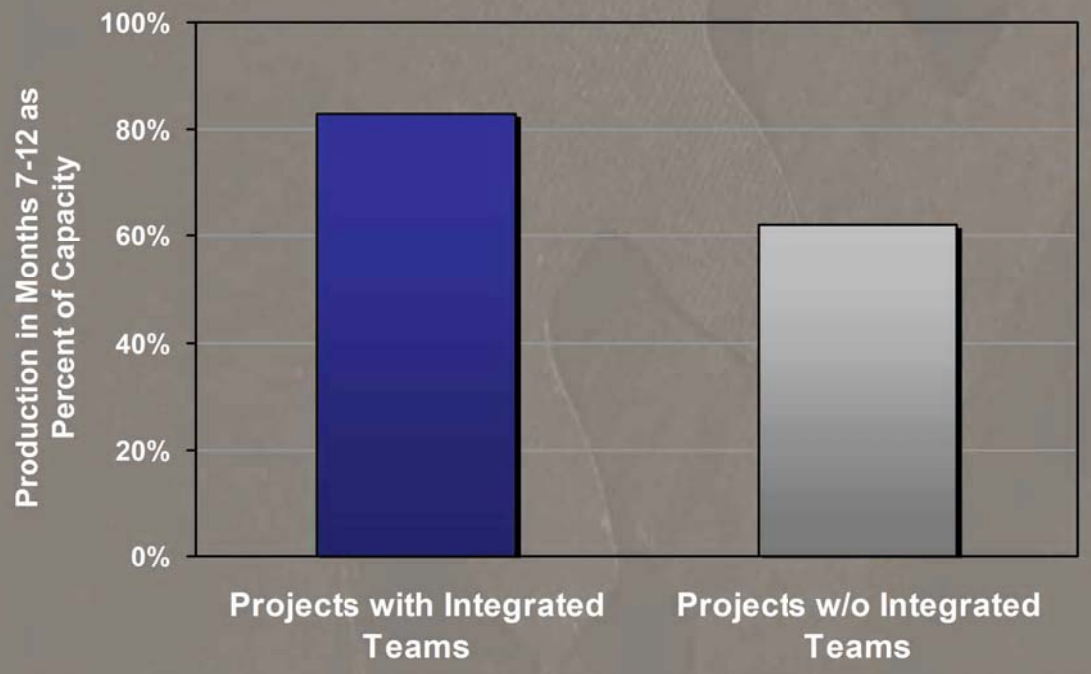
Defining the Integrated Project Team

- An integrated project team has all owner functions fully represented on the team starting in front-end loading. At least the following are present:
  - Environmental
  - Planning & Scheduling
  - Geology
  - Mine Eng./planning
  - Business
  - Engineering
  - Construction
  - R&D
  - Maintenance
  - Mine/Plant operations
  - Health & Safety
  - Startup
- These team members are identified prior to project authorization and have specific responsibilities that are defined and understood by all team members
- The team members have the authority to make decisions for their function
- Reasonable continuity must be maintained

Staffing New Technology Programs

- Three critical factors in new technology staffing:
  - Core team should have continuity from pilot(s) through commercialisation of first unit
  - Core team must include science/technology base, solid project expertise, and high-quality process engineers
  - Additional resources must be made available as needed and requested

Team Integration Directly Improves Operability of Minerals Plants



Team Integration also...

- Reduces deleterious effects of not knowing the heat and material balances by 45 percent!
- Reason is straightforward:
  - If you lack strong empirical basis, next best substitute are knowledgeable people, especially operations, maintenance, startup, and core R&D personnel

Outline

- IPA background and methodology
- Industry history with similar projects
- DOE project history
- Reasons for project failure
- Best Practices for success
- *Conclusions*

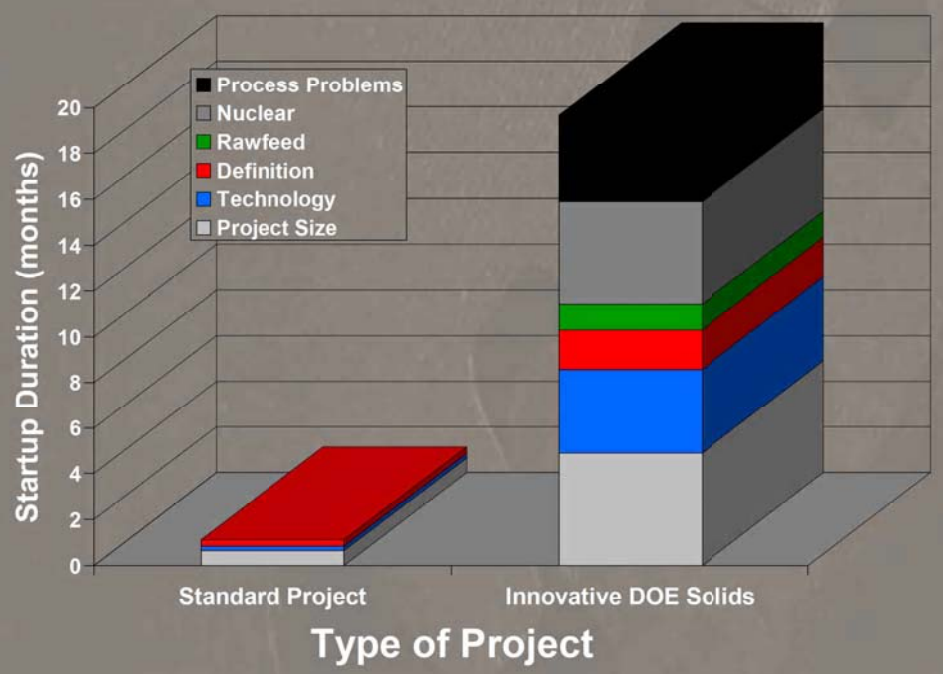
Summing Up

- Raw solids processing projects face some unique challenges when trying to introduce new technology
  - Problems that are easily resolved in other types of processing such as waste handling and feedstock problems are more stubborn
  - Transfer of basic technical data from prior plants is both more important and more difficult

What Does This All Mean for a DOE Project Startup?

- $100 million project
- DOE project:
  - Minor process modification with 2 new steps (6 total)
  - Medium level process problems
  - Nuclear
  - Raw solid feed
  - Poor FEL
- Industry (non-nuclear) project
  - No new technology, so no process problems
  - 6 total process steps
  - No raw solid feed
  - Good FEL

Startup Duration Example



Some Don’ts of New Technology Program Management

- Remember, most R&D is just plain work and can be planned, scheduled, and resourced as such
- Don’t substitute good engineering for bad basic data
  - All you get is a conservatively designed, inoperable plant
- Never fast-track R&D
- Never fast-track new technology projects

More Don’ts

- Don’t understate risks to get approval
- Don’t do new technology projects without doing excellent front-end definition; risks will kill you
- Don’t mismatch business objectives and new technology development realities
  - If success requires short-cutting to achieve cycle time, cancel the project
  - If business goal is an incremental improvement, a long, costly R&D effort will never work

Thank you for your attention!

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ARC
AUSTRALIAN RESEARCH COUNCIL

THE UNIVERSITY OF MELBOURNE

D. V. Boger Research Interests

- Viscoelastic Fluid Mechanics
- Particulate Fluids
- Environmental Rheology
- Hydrocolloids

D.V. Boger
Particulate Fluids Processing Centre
Department of Chemical & Biomolecular Engineering
The University of Melbourne
Victoria Australia

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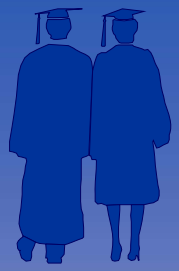
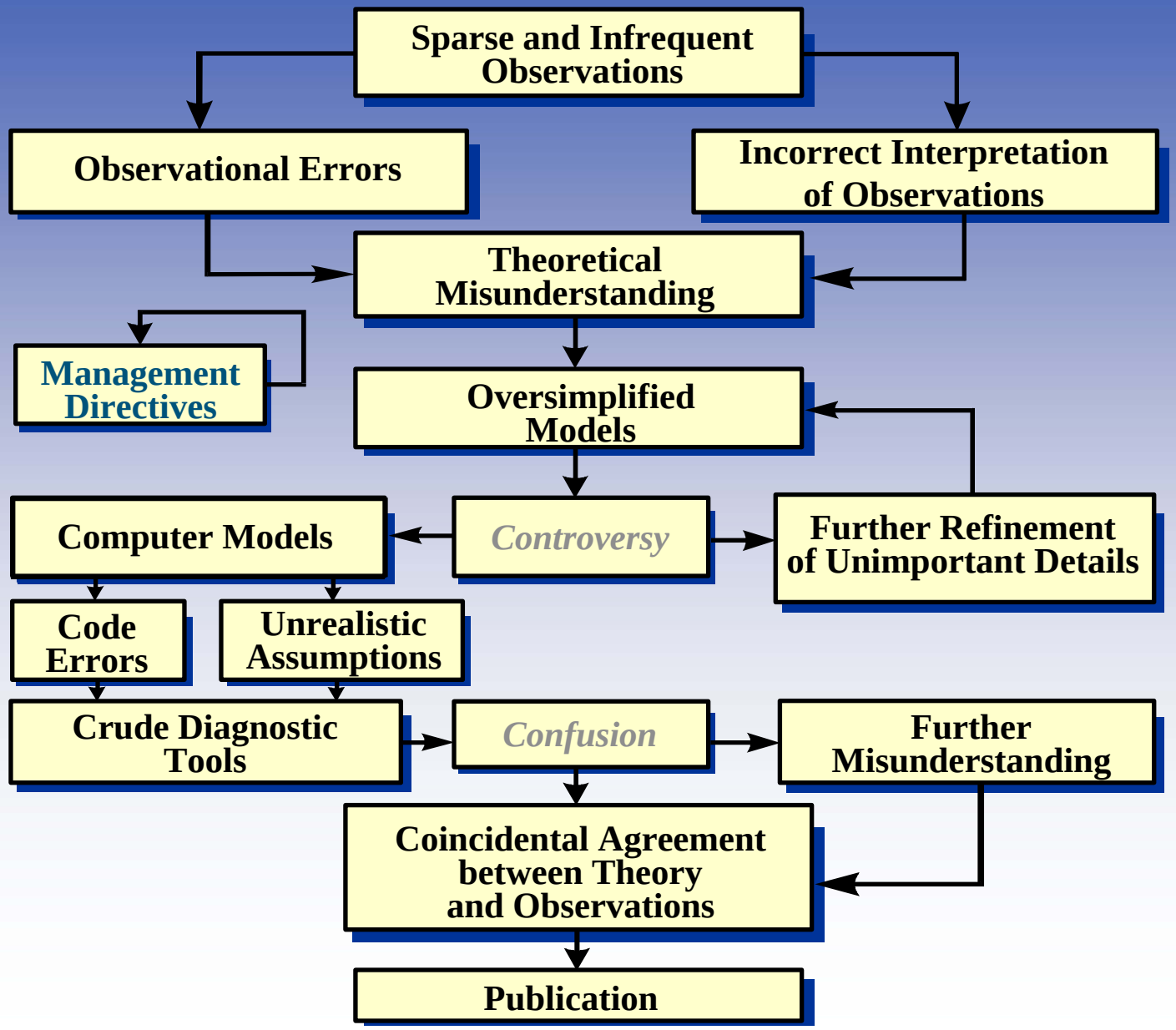
The Course of Science





The Rheology and Surface Chemistry of Slurries

David V Boger
Laureate Professor
The University of Melbourne
Australia

Particulate Fluids – Suspensions

- Settling Suspensions
- Non-Settling Suspensions

Ratio of Time Scales = N_{Se}

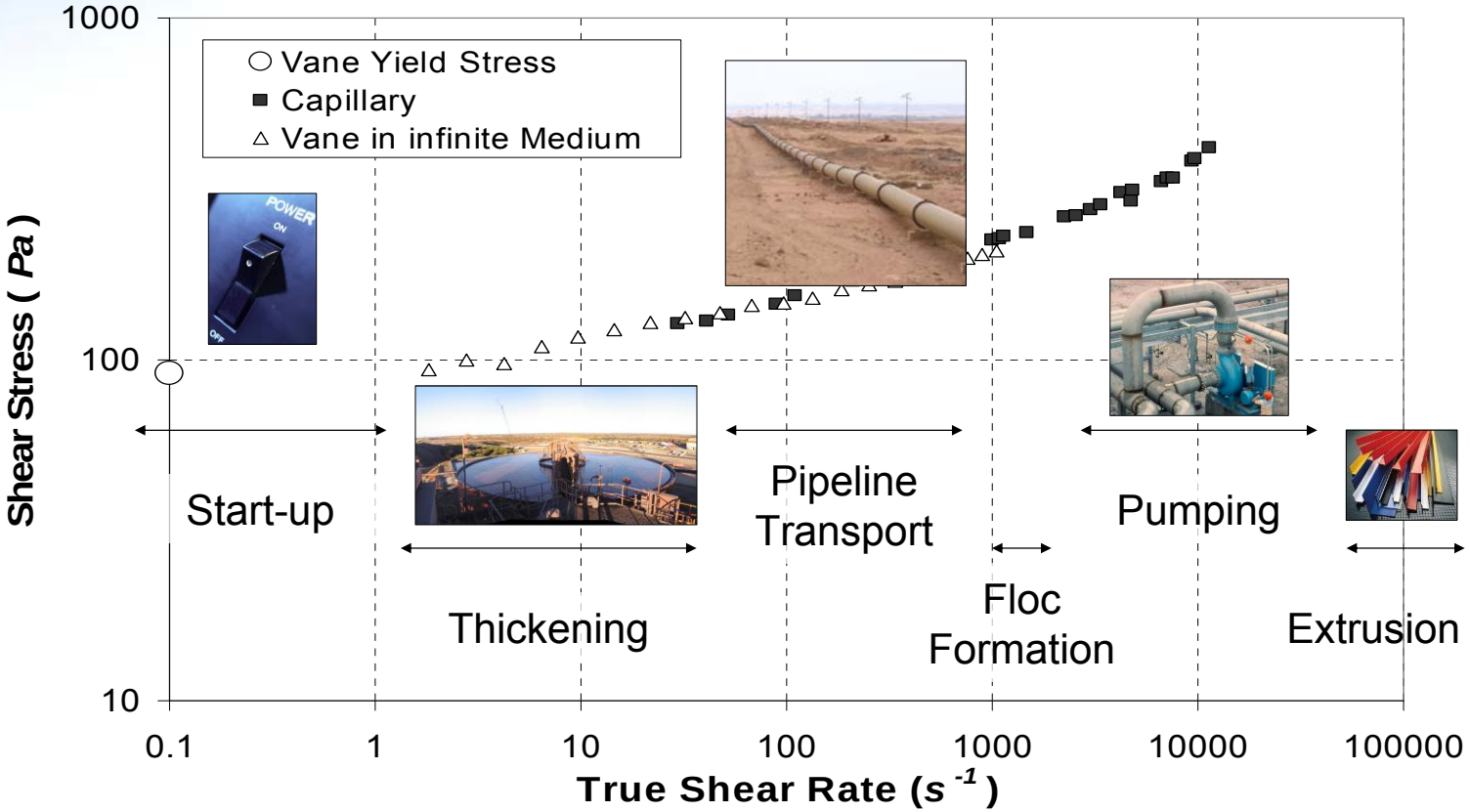
$$N_{Se} = \frac{\tau}{t_S}$$

τ = Characteristic time of the process

t_S = Characteristic time of settling

N_{se} <<1 Non-Settling (Rheology)

Why do we want to Characterise Rheology?



Bauxite Residue Disposal

1974 - Alcoa of Australia (Consulting)
1978 - G. Sarmiento (Ph.D)
1983 - Q.D. Nguyen (Ph.D - Alcoa)
1986 - N.J. De Guingand (M.Eng.Sc - Alcoa)
1992 - PROBLEM SOLVED ?
1993 - N. Pashias (Ph.D - Alcoa)
2000 - F. Sofra (Ph.D - Alcoa)
2005 - D. Cooling (Ph.D - Alcoa)

Exploiting the Rheology of Mineral Tailings

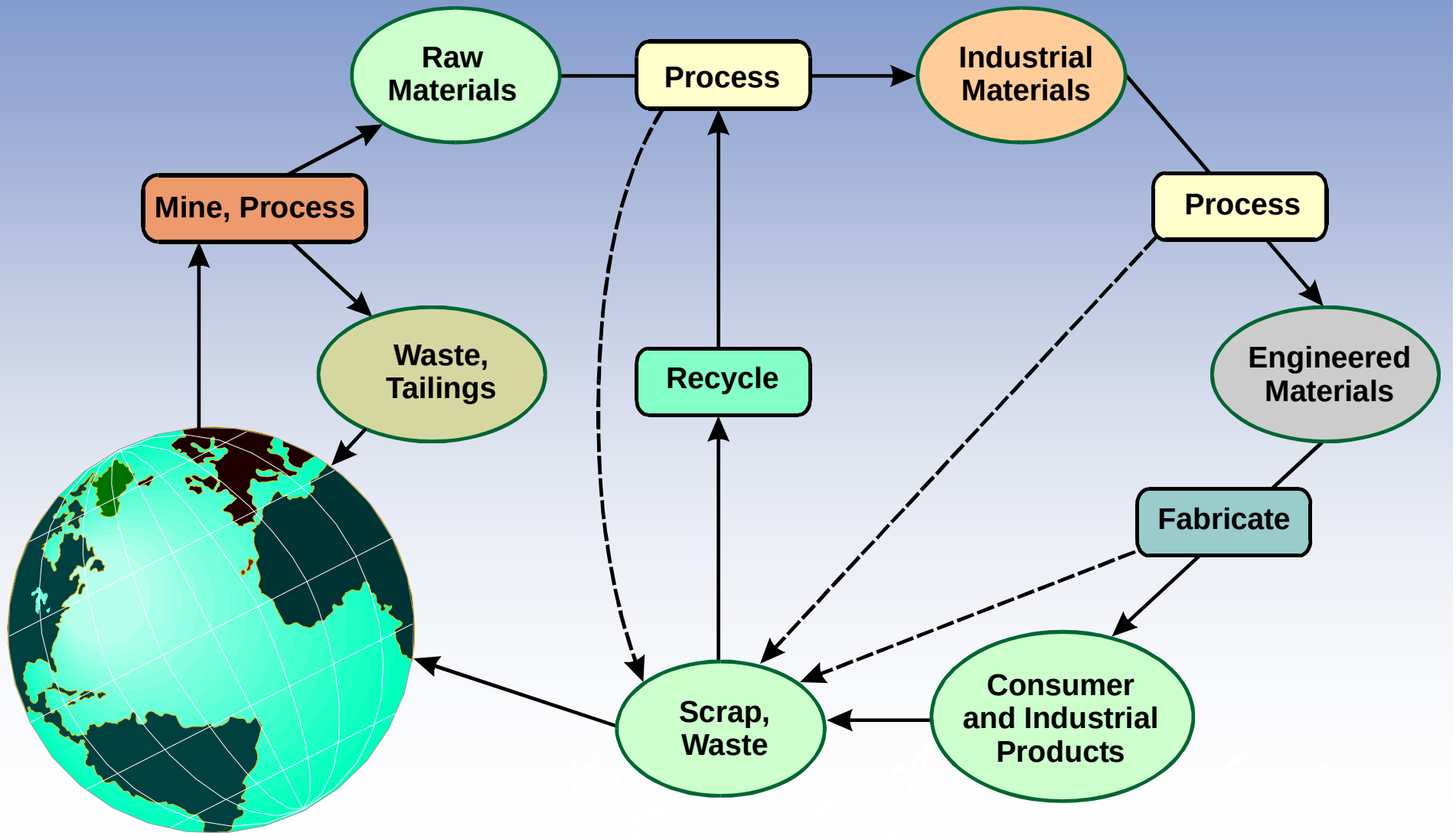
D.V.Boger

Particulate Fluids Processing Centre
Department of Chemical & Biomolecular Engineering
The University of Melbourne
Victoria Australia

Environmental Rheology

D.V.Boger
Particulate Fluids Processing Centre
Department of Chemical & Biomolecular Engineering
The University of Melbourne
Victoria Australia

Sustainability, Recycling and Environmental Modelling



Escondida

D. V. Boger

BHP Billiton Fellow

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'94 4 3

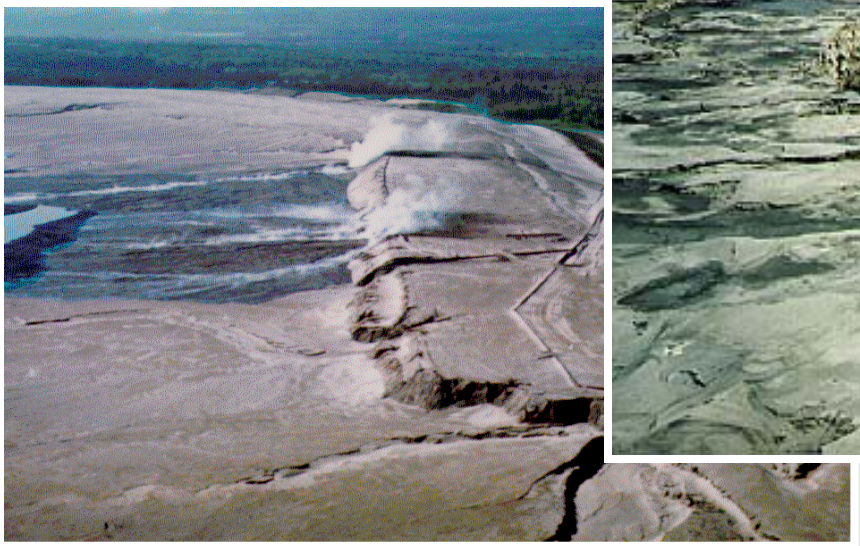
The Canadian Oil Sands Industry

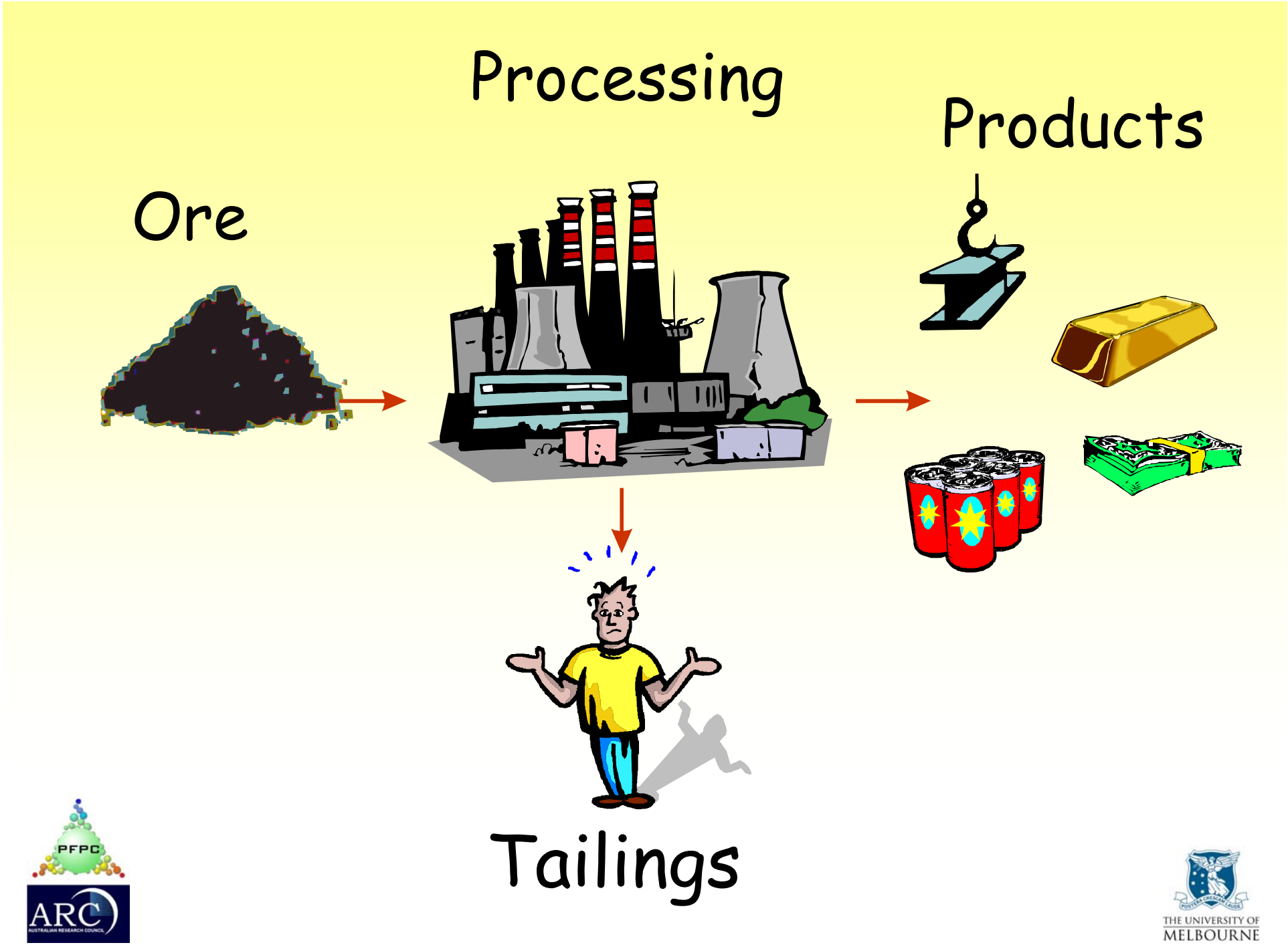
"By 2010 the oil sands industry will be producing about one million barrels of oil per day from surface mined oil sands. This equates to about one million cubic metres of coarse tailings deposit per day and 0.2 to 0.3 million cubic metres of fine tailings per day. To date the industry has produced 400 million cubic metres of fine tailings."

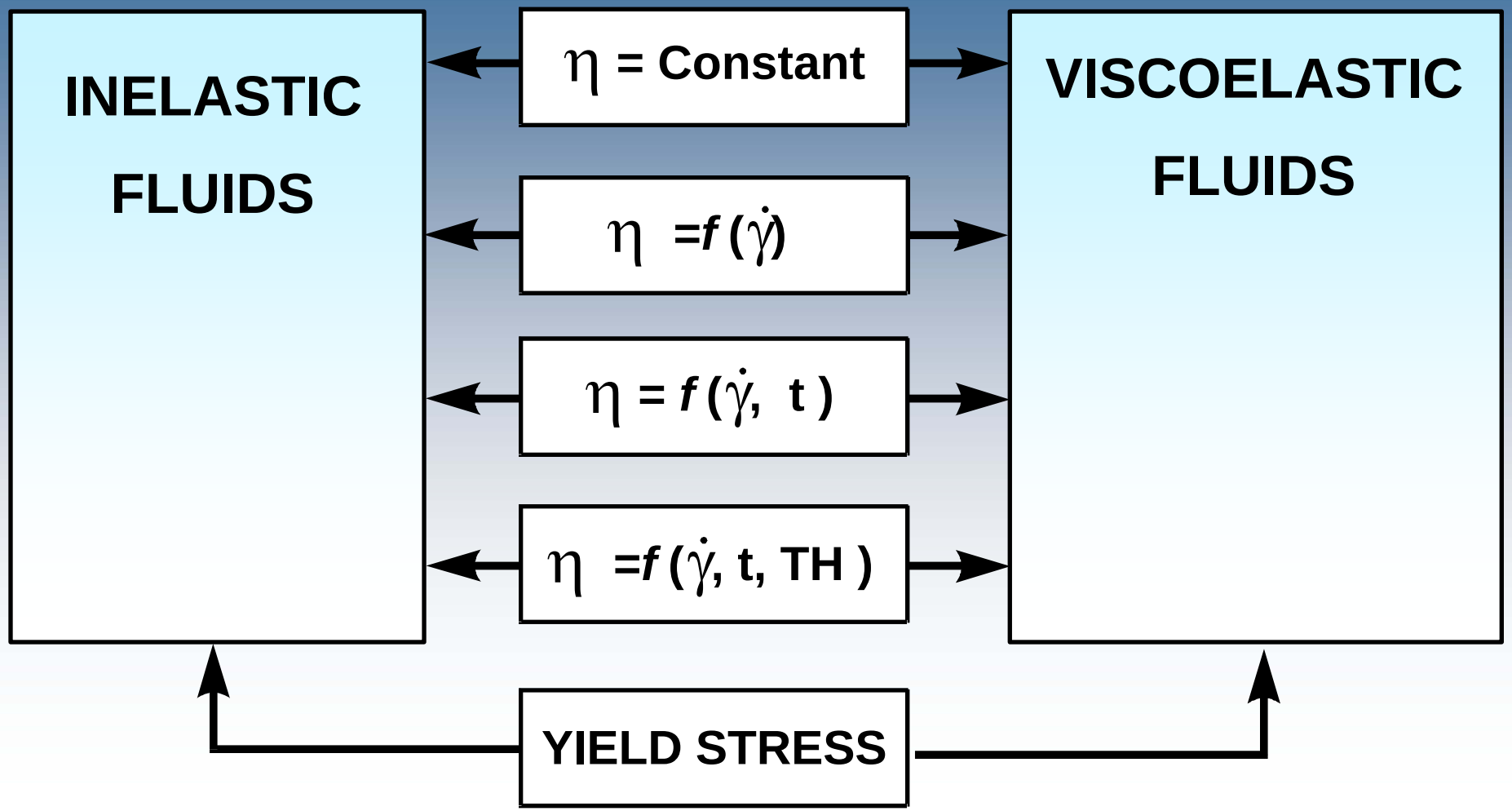
D.V.Boger

Particulate Fluids Processing Centre
Department of Chemical & Biomolecular Engineering
The University of Melbourne
Victoria Australia

Oil Sands Tailings Deposition




Processing
Products
Ore
Tailings


INELASTIC FLUIDS
η = Constant
η =f (γ̇)
η = f (γ̇, t)
η =f (γ̇, t, TH)
VISCOELASTIC FLUIDS
YIELD STRESS

Rheometry

PFPC

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AUSTRALIAN RESEARCH COUNCIL

THE UNIVERSITY OF MELBOURNE

Developments motivated by applications in minerals industry

- The Vane Yield Stress Rheometer
  (J. Rheol., 27, 321, 1983; and 29, 335, 1985)

- The Conical Slump Test for Yield Stress Measurement (50 cent Rheometer)
  (J. Rheol., 40, 1119, 1996)

- The Bucket Rheometer
  (J. Rheol. *In press*, 2007)

Yield Stress Measurement

D.V. Boger

Particulate Fluids Processing Centre
Department of Chemical & Biomolecular Engineering
The University of Melbourne
Victoria Australia

Colgate

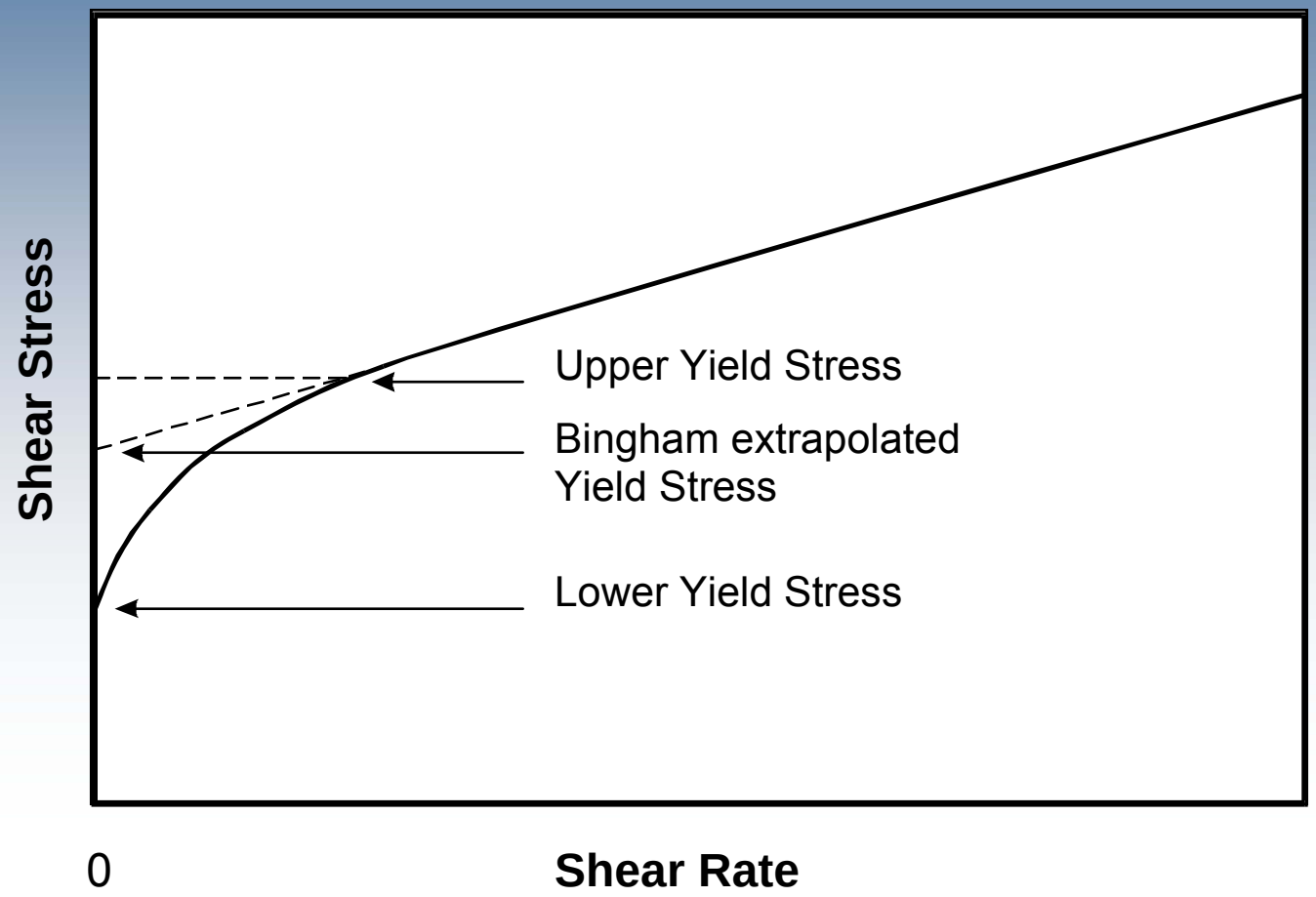
Colgate

Colgate

Typical Yield Stress Values

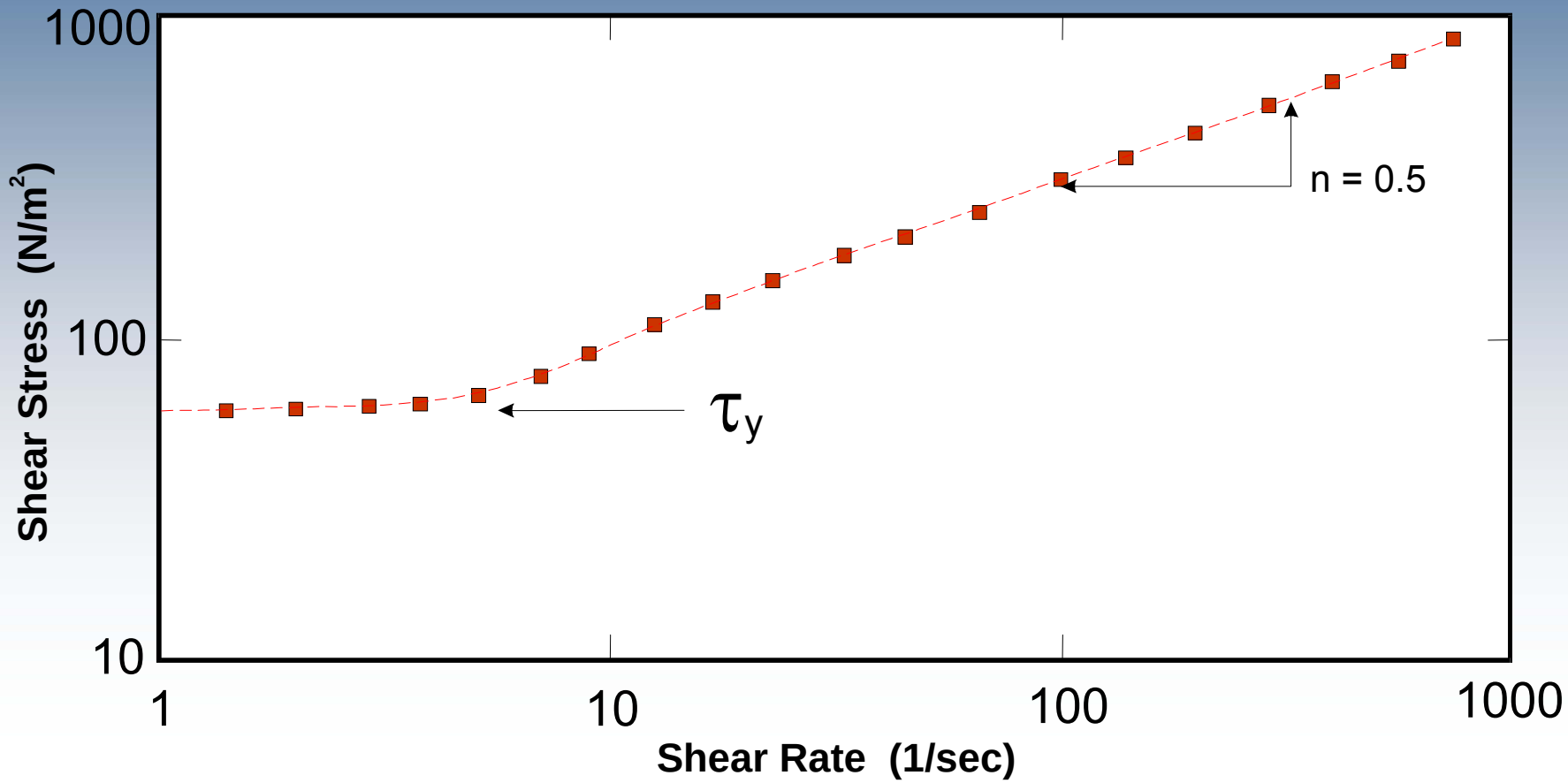
Tomato Sauce (Rosella)	15 Pa
Yoghurt	80 Pa
Toothpaste	110 Pa
Peanut Butter	1900 Pa
Thickened Tailing Disposal	30-100 Pa
Mine Stope Fill	250-800 Pa

The Yield Stress

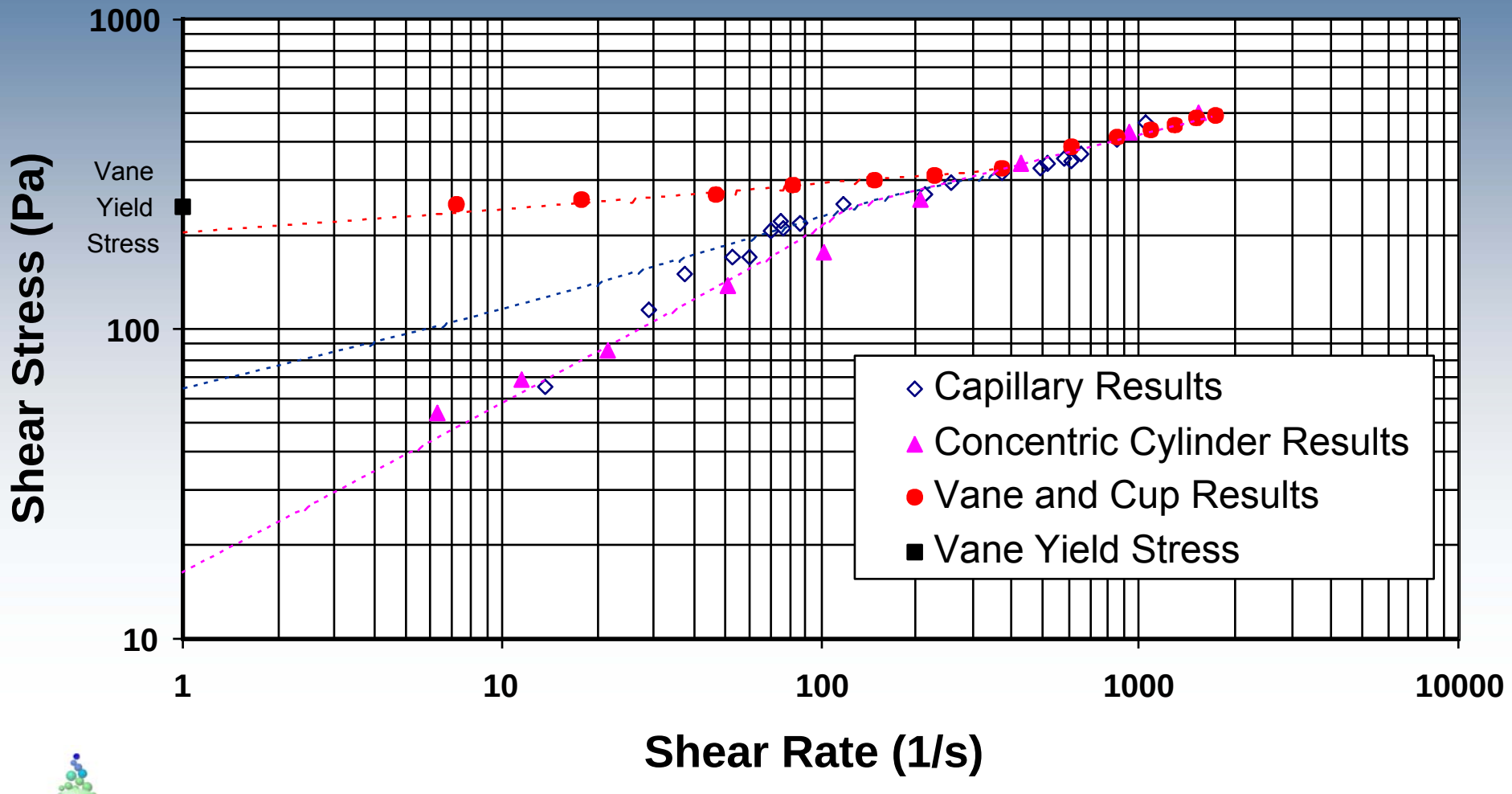


Tomato Soup Concentrate

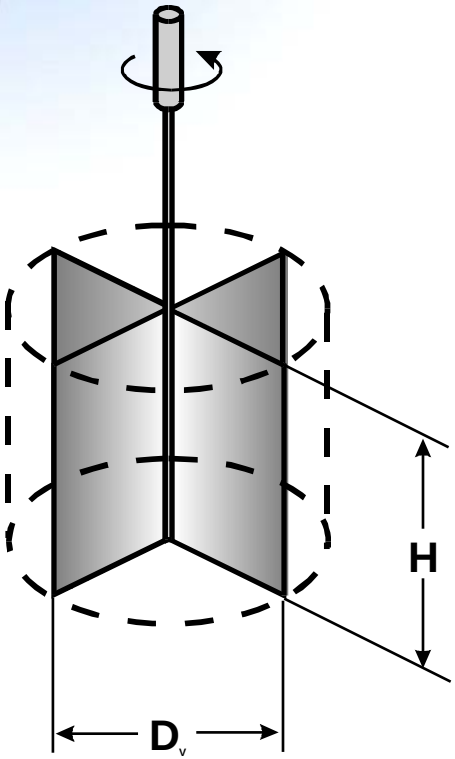
Temperature 20 °C

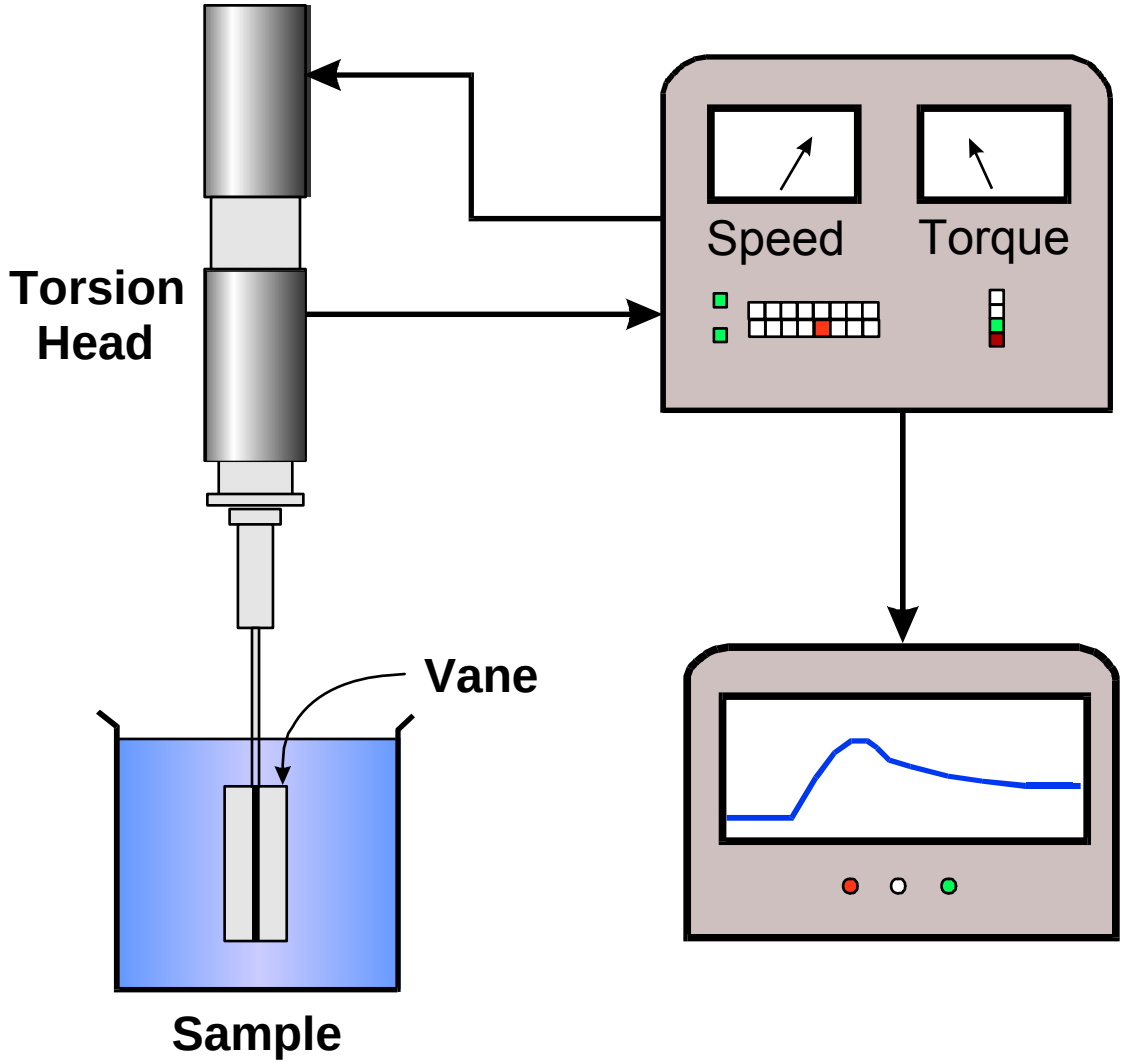


Flow Curves for Paste Sample - Yield Stress = 250 Pa



Vane Technique





The Vane Method

$$T_m = (\pi\, dl)\frac{d}{2}\tau_y + 4\pi\int_0^{d/2} \tau_e\,(r)\, r^2\, dr$$

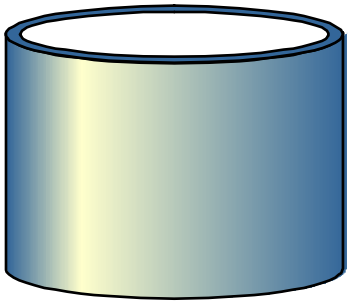
If $\tau_e = \tau_y$ then

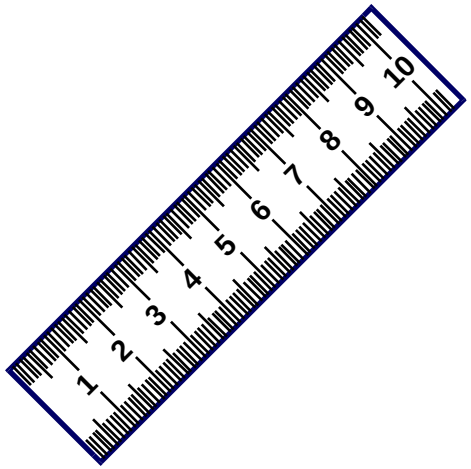
$$T_m = \frac{\pi}{2} d^3\left(\frac{1}{d} + \frac{1}{3}\right)\tau_y$$

"Yield Stress Measurement for Concentrated Suspensions" (with Q.D. Nguyen), J. of Rheology, 27, 321 (1983)"

"Direct Yield Stress Measurement with the Vane Method" (with Q.D. Nguyen), J. of Rheology, 29, 335 (1985)"

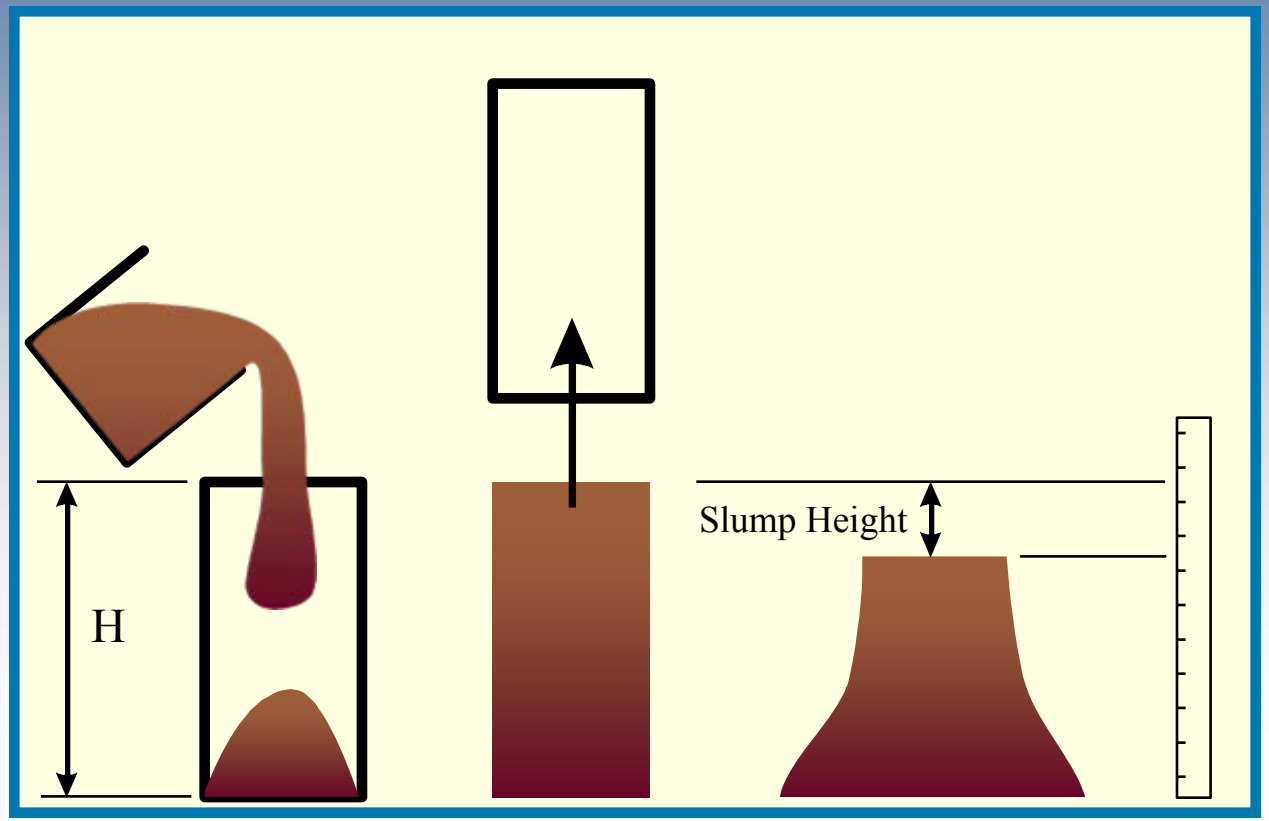
“D#Liw#hqw#Khrphwu#ru \hogwhv#Phdvxuphqw“





M#Khro/#73+9,/#14:<#1<<9

The Slump Test



1. Fill slump mould
2. Remove mould vertically
3. Measure distance between mould and top of sample

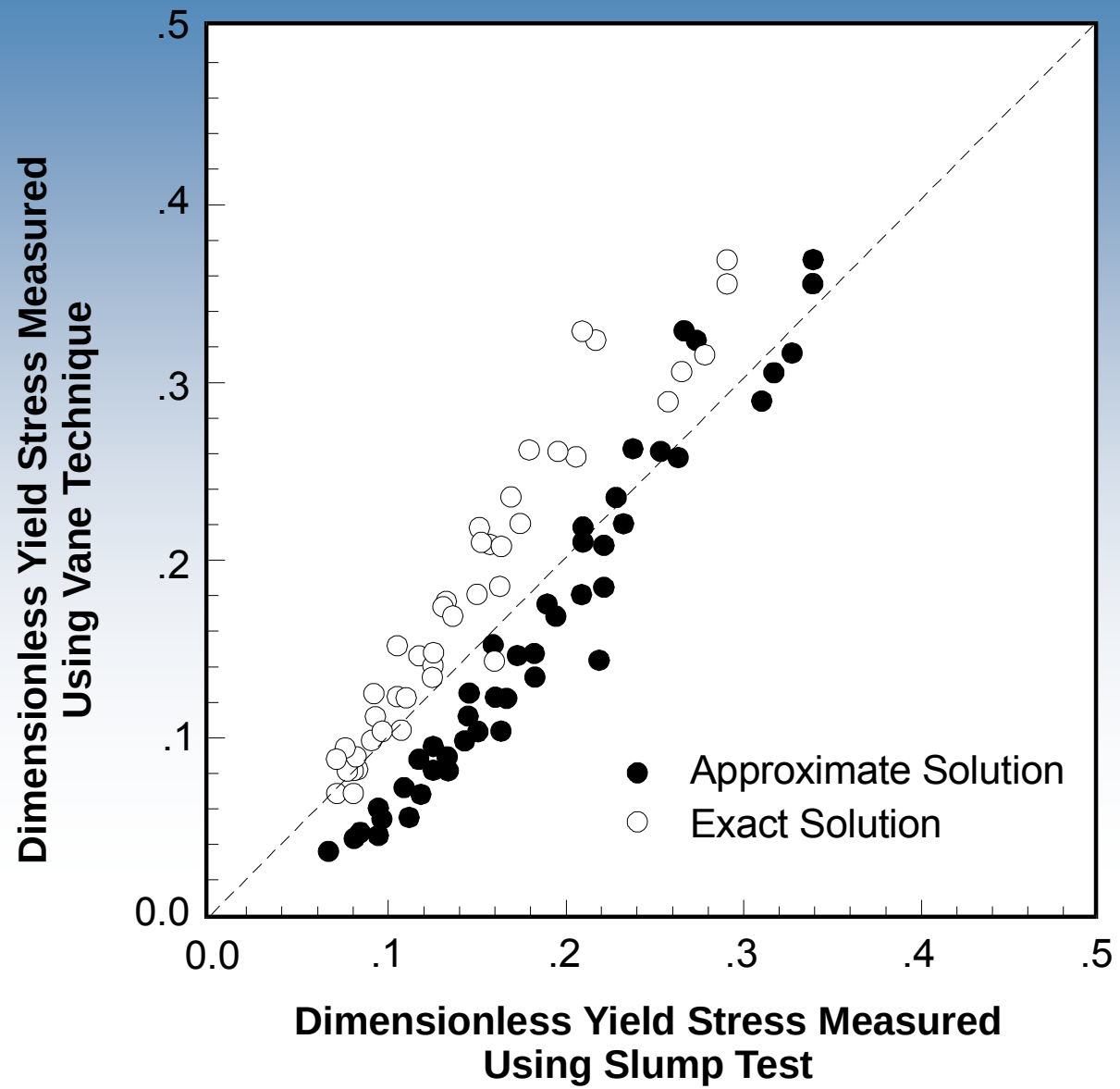
Slump Test

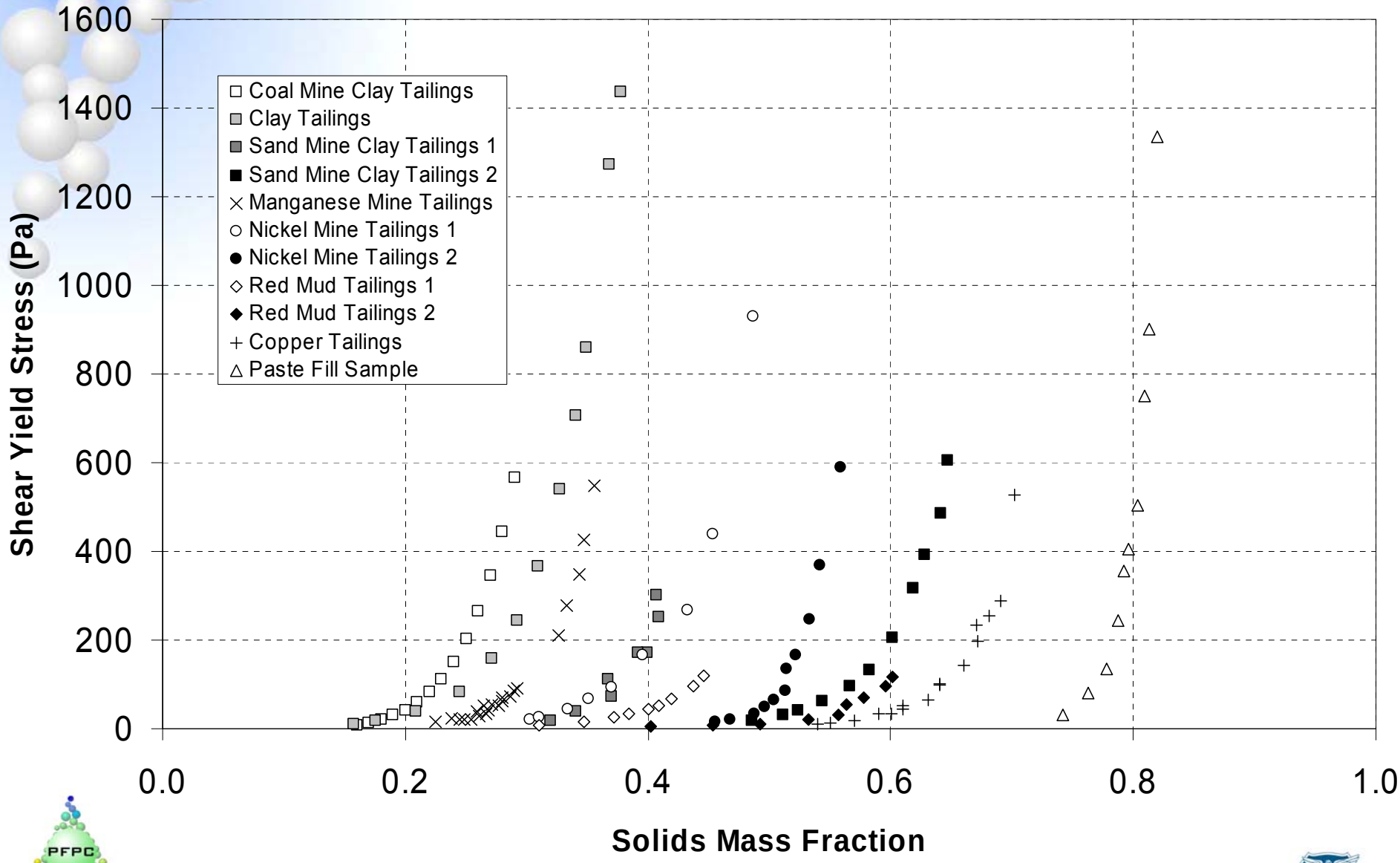
Approximate Result:

$$\tau_y' = \frac{1}{2} + \frac{1}{2}\sqrt{S'}$$

The Fifty Cent Rheometer
D.V. Boger
N. Pashais
D.J. Glenister
J. Summers

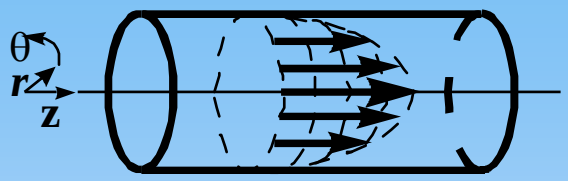
Comparison of Yield Stress Measuring Techniques



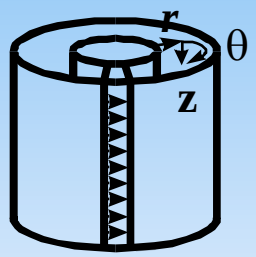

Shear Yield Stress (Pa)
1600
1400
1200
1000
800
600
400
200
0
0.0
0.2
0.4
0.6
0.8
1.0
Solids Mass Fraction
Coal Mine Clay Tailings
Clay Tailings
Sand Mine Clay Tailings 1
Sand Mine Clay Tailings 2
Manganese Mine Tailings
Nickel Mine Tailings 1
Nickel Mine Tailings 2
Red Mud Tailings 1
Red Mud Tailings 2
Copper Tailings
Paste Fill Sample

Viscometric flows used for viscosity measurement

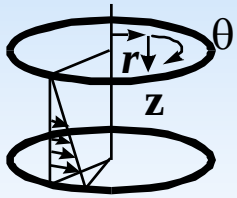
Poiseuille Flow



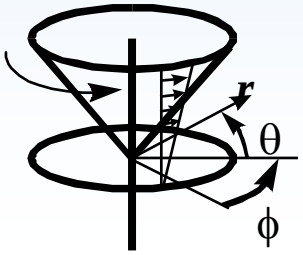
Couette Flow



Parallel Plate Torsion



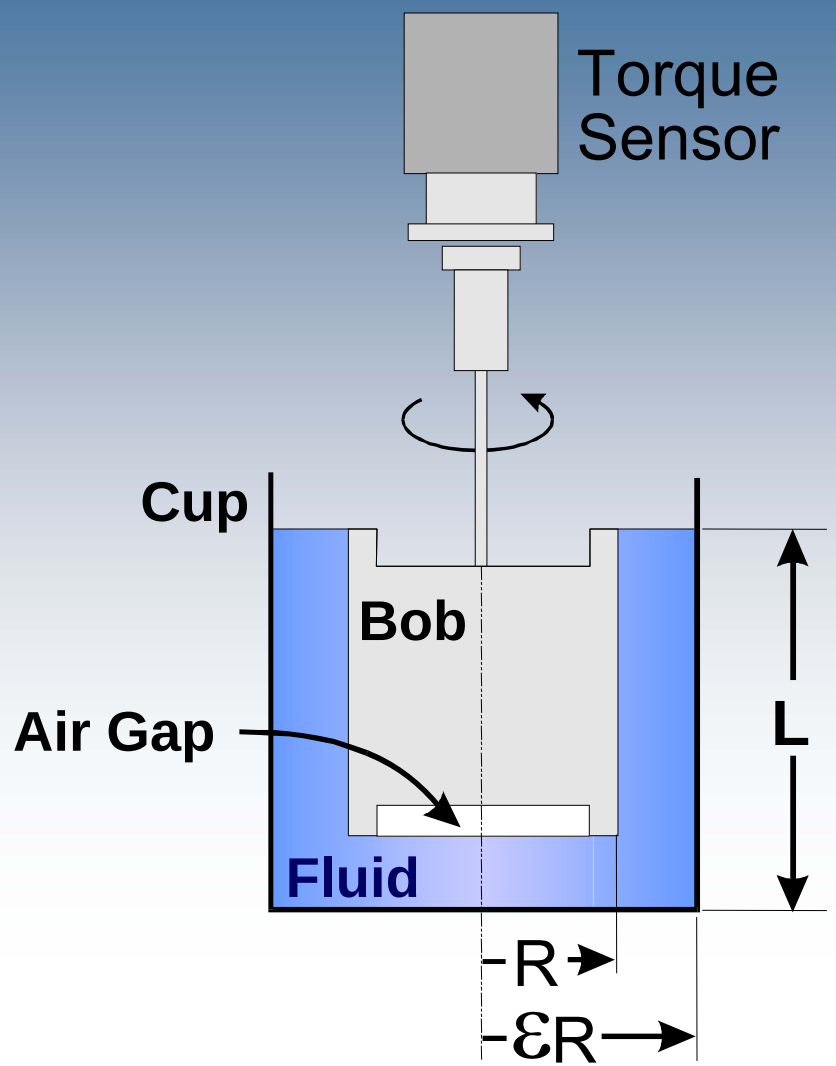
Cone and Plate Torsion



Current Methods of Characterisation

Capillary Rheometry

Rotational Rheometry


Torque
Sensor
Cup
Bob
Air Gap
Fluid
L
R
εR

Couette Viscometry

Basic Equations:

$$\dot{\gamma} = f(\tau) = -r\,d\omega/dr$$

$$\tau = T/2\pi L r^2$$

$$\tau_1 = T/2\pi L R^2 = \varepsilon^2 \tau_2$$

$$\Omega = \int_{\tau_2}^{\tau_1} \frac{f(\tau)}{2\tau}\, d\tau$$

where τ_1 and τ_2 are the shear stress at the surfaces of the bob (r=R) and the cup (r=εR), respectively.

Newtonian Fluid:

$$\dot{\gamma}_1 = f(\tau_1) = 2\Omega/(1-\varepsilon^{-2})$$

Overview of the Problem

- Samples are not stable over time.
- Some testing methods are susceptible to particle size effects.
- Slip between the sample and the shearing interface can occur.
- Some instrumentation is very large and not portable.

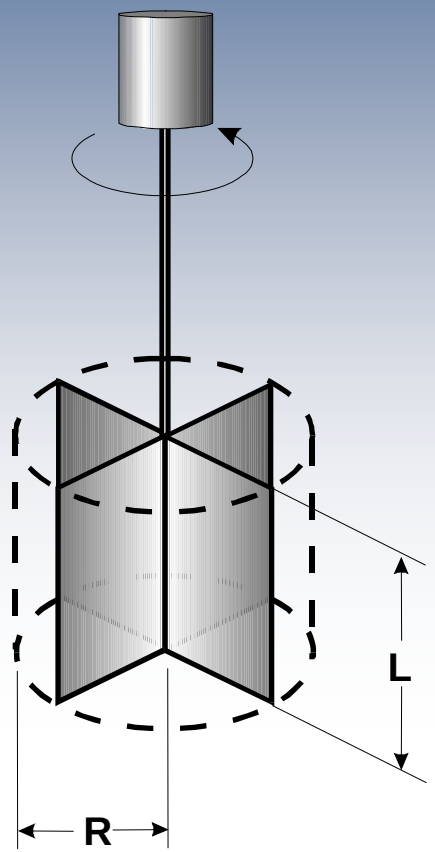
THE BUCKET RHEOMETER FOR THE VISCOSITY CHARACTERISATION OF YIELD STRESS SUSPENSIONS

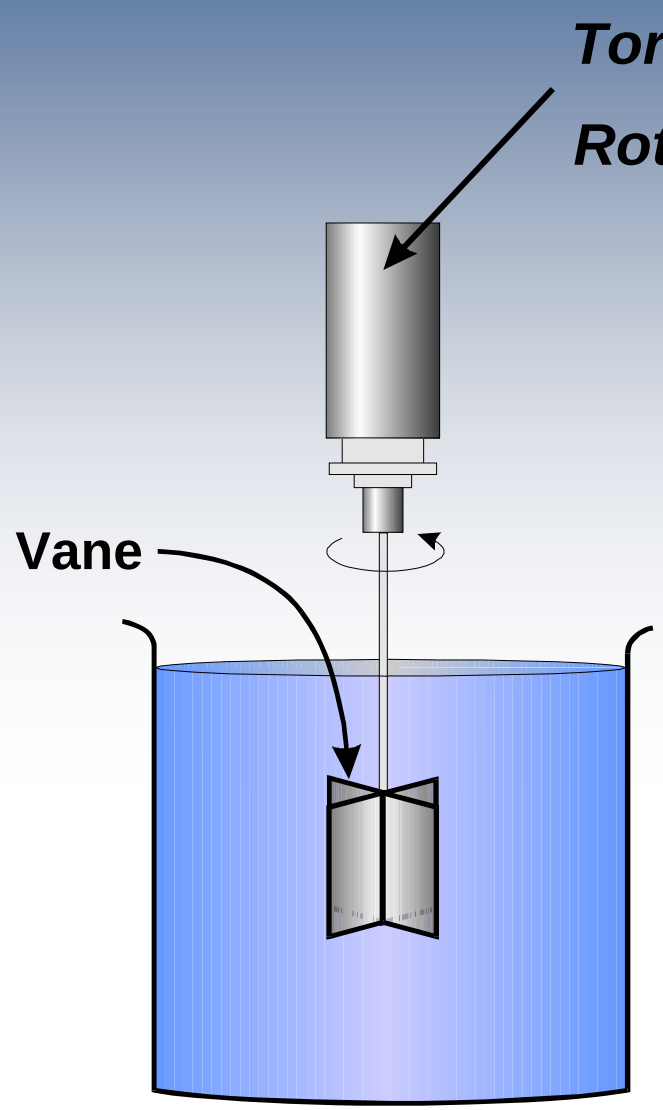
Daniel T. Fisher

Professor Peter J. Scales

Laureate Professor David V. Boger

The Bucket Rheometer





Torque **(T)**

Rotational Speed **(Ω)**

Shear Stress:

$$\tau = \frac{T}{2\pi LR}$$

where **R = vane radius**

L = vane height

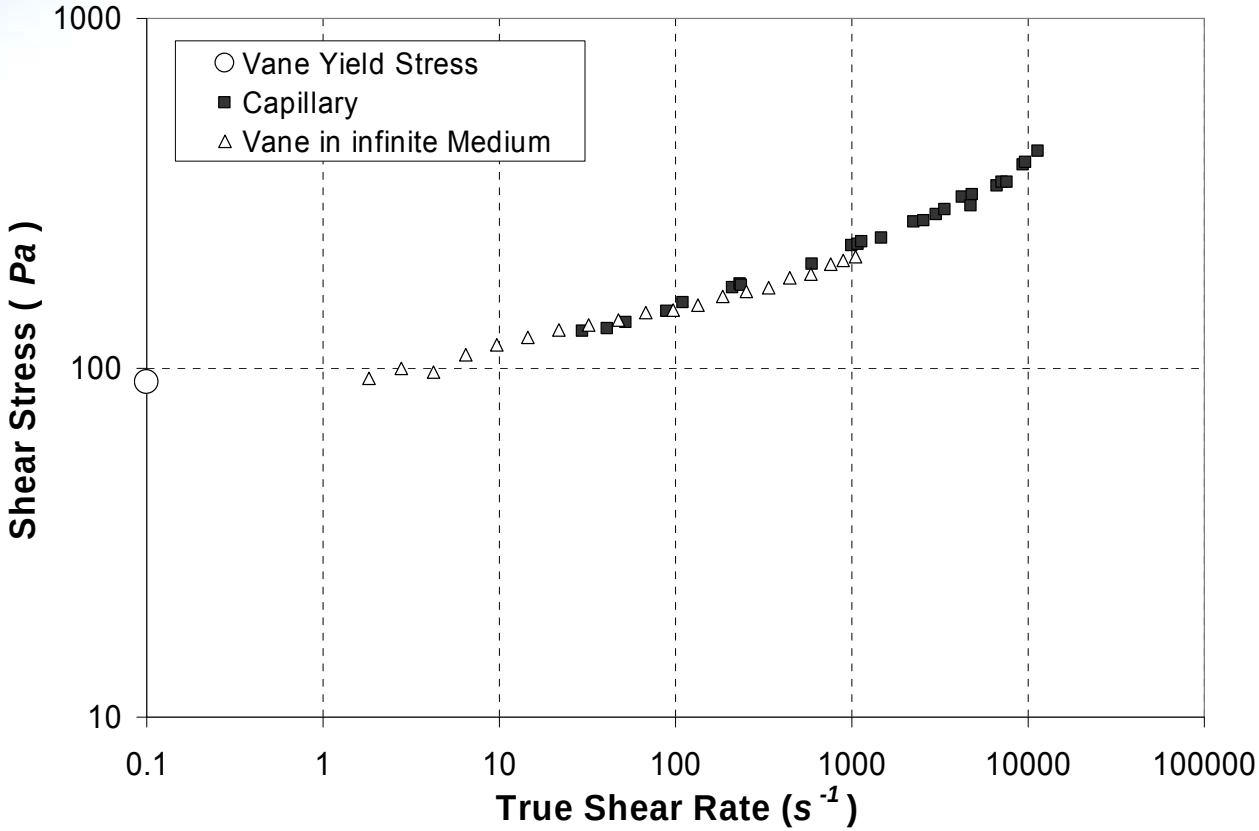
Shear Rate:

$$\dot{\gamma} = \frac{2\Omega}{S}$$

where $S = \frac{d\ln T}{d\ln \Omega}$

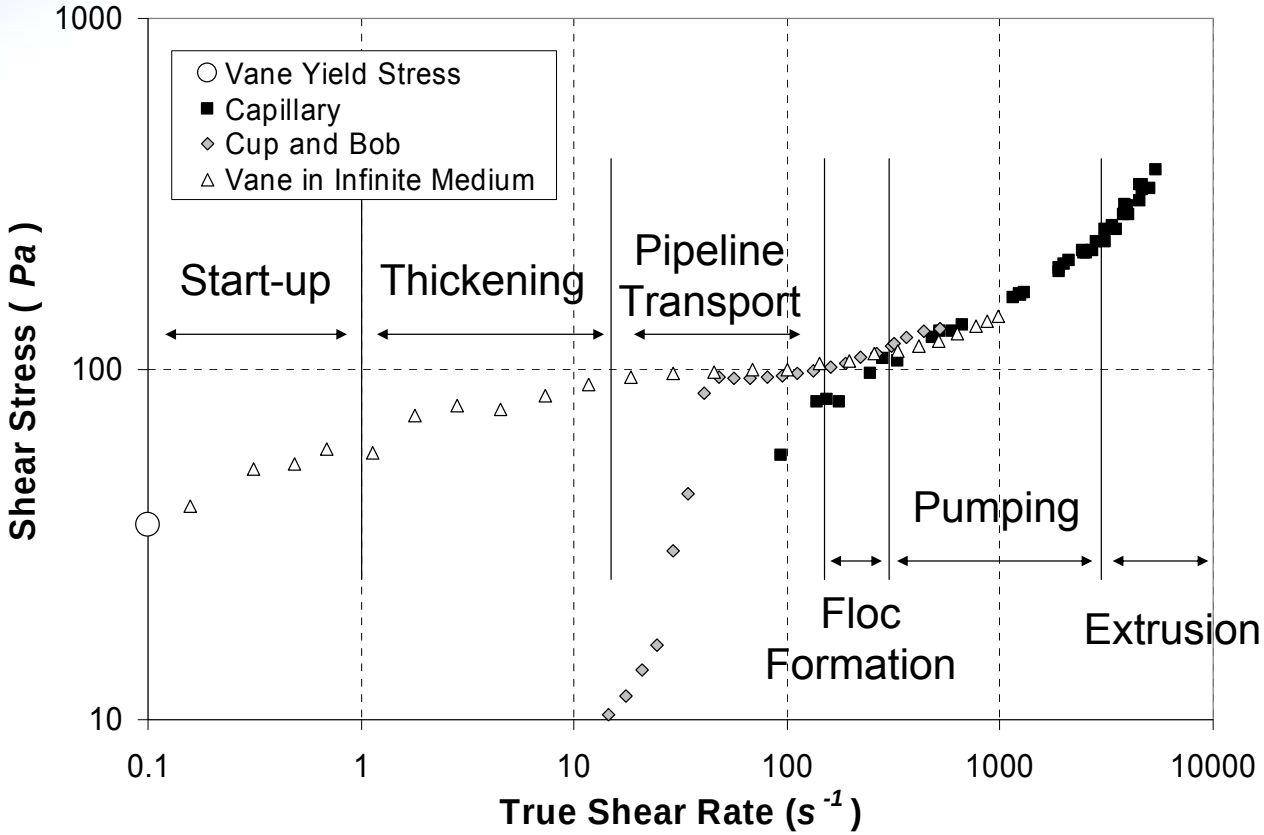
Alumina Paste (AKP50)

50.2%w/w and pH 7.60



Transitional Limonite Slurry

45.1%w/w and pH 6.34



Ideal Particulate Fluids

D.V. Boger

Particulate Fluids Processing Centre
Department of Chemical & Biomolecular Engineering
The University of Melbourne
Victoria Australia

Rheology of Suspensions

DEFINITIONS:

Relative Viscosity

Einstein

$$\eta_r = \frac{\eta}{\eta_0} = 1 + 2.5\phi + 6.2\,\phi^2 + 0(\phi^3)$$

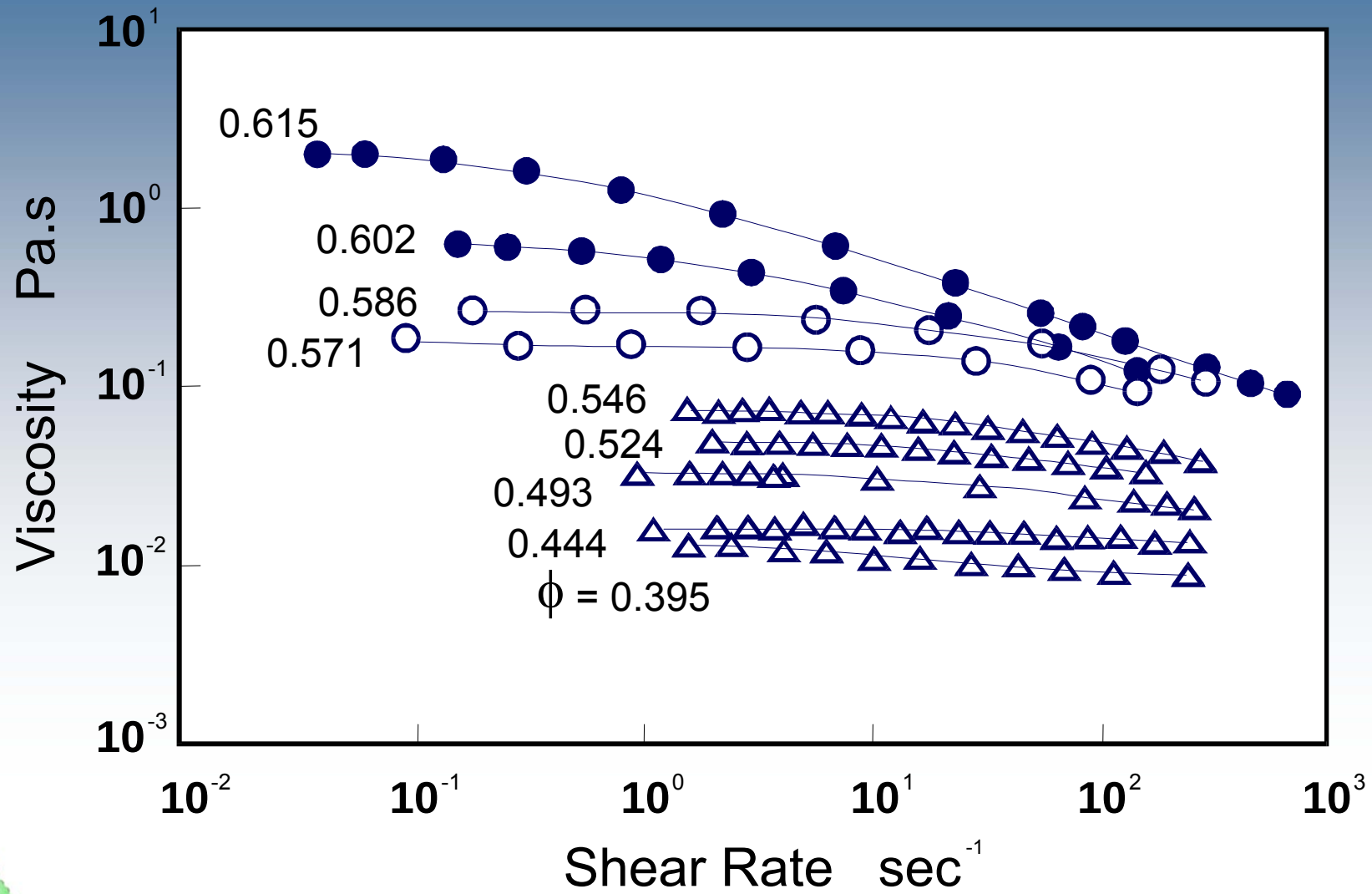
Bachelor

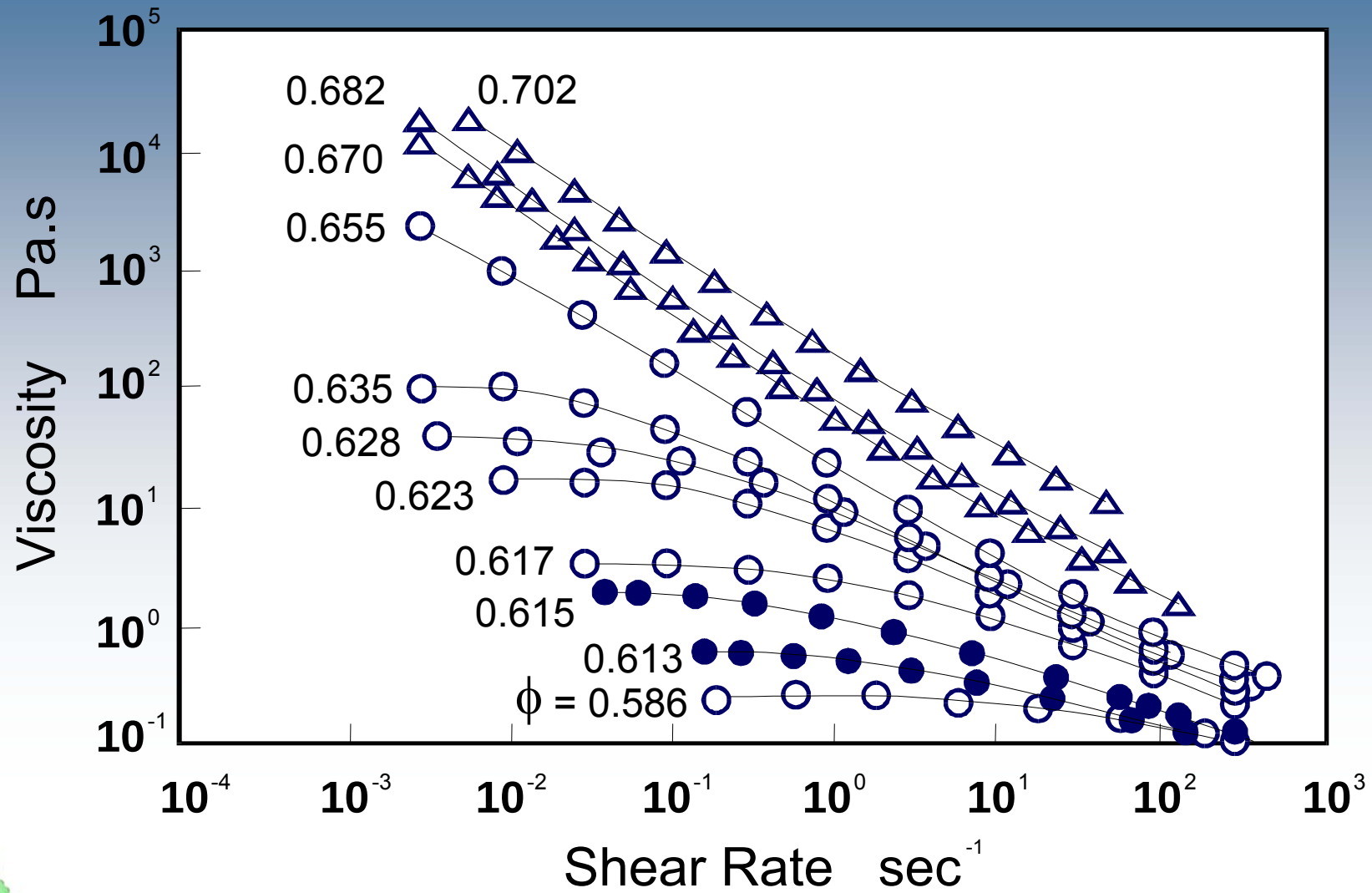
Specific Viscosity

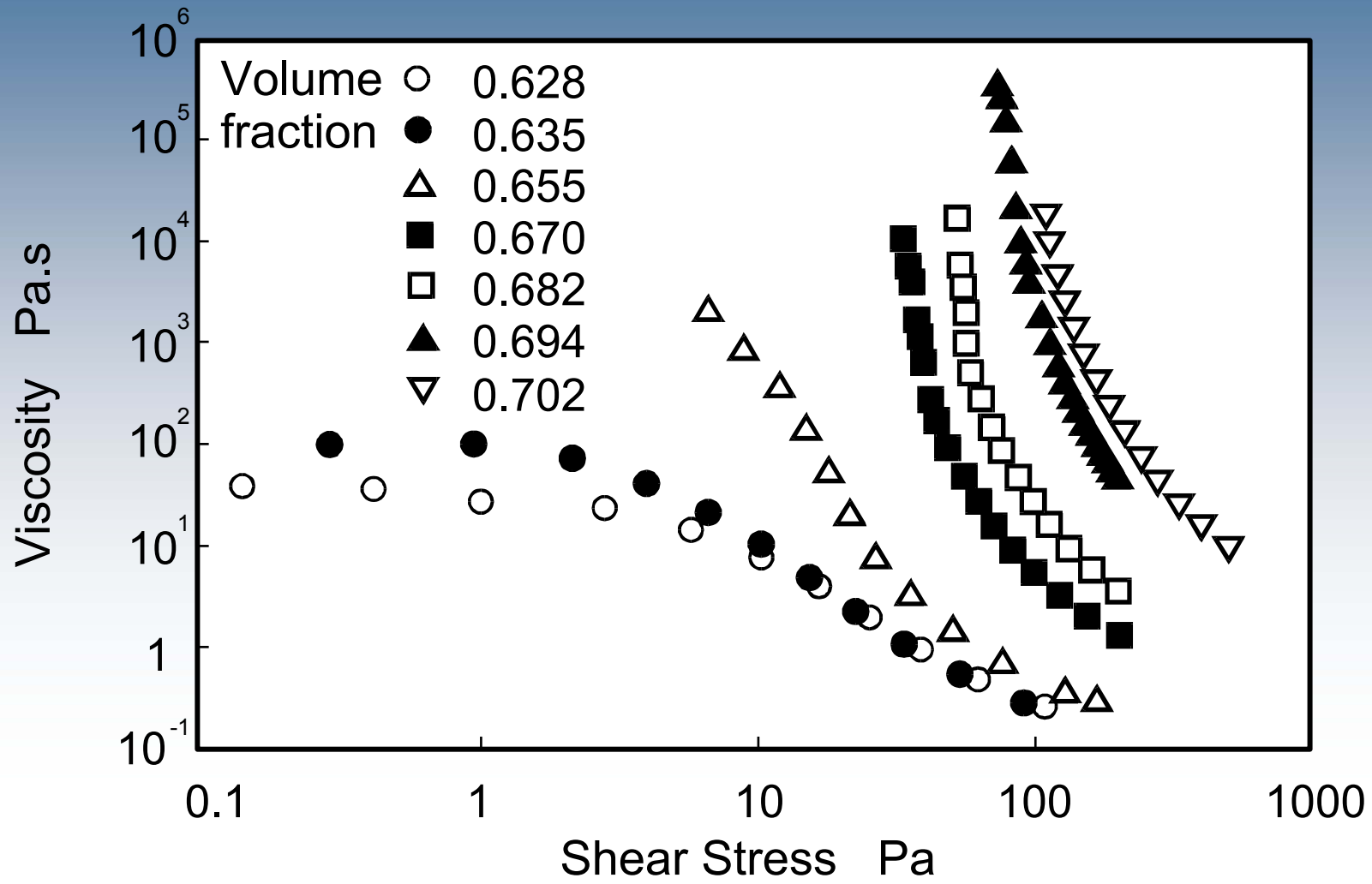
$$\eta_{sp} = \eta_r - 1 = \frac{\eta - \eta_0}{\eta_0}$$

Instrinsic Viscosity

$$[\eta] = \lim_{\phi \to} \frac{\eta_{sp}}{\phi}$$


Viscosity Pa.s
Shear Rate sec-1
0.615
0.602
0.586
0.571
0.546
0.524
0.493
0.444
ϕ = 0.395


Viscosity Pa.s
Shear Rate sec-1
0.682
0.702
0.670
0.655
0.635
0.628
0.623
0.617
0.615
0.613
ϕ = 0.586
10^5
10^4
10^3
10^2
10^1
10^0
10^-1
10^-4
10^-3
10^-2
10^-1
10^0
10^1
10^2
10^3


Volume fraction
0.628
0.635
0.655
0.670
0.682
0.694
0.702
Viscosity Pa.s
Shear Stress Pa
10^{6}
10^{5}
10^{4}
10^{3}
10^{2}
10^{1}
1
10^{-1}
0.1
1
10
100
1000

Yield Stress for Hard Sphere Suspensions

ϕ (Volume Fraction)	τ_y (Yield Stress, Pa)		
	Vane	Steady Shear	Creep
0.670	----	30 +/- 5	25 +/- 5
0.682	22 +/- 5	50 +/- 5	45 +/- 5
0.694	67 +/- 6	80 +/- 8	90 +/- 5
0.708	108 +/- 2	100 +/- 10	90 +/- 10

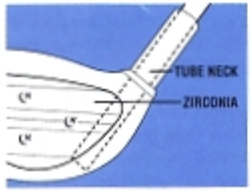
J. Coll. and Interfacial Sci., Vol 147, number 2, 479-495 (1991)

Our Ceramic Will Test Your Metal

Introducing The New Z-Driver From Coors CeraSports

Superior Performance. A well-hit ball with the Coors CeraSports Ceramic Z-Driver is an experience unlike any other in golf. The way it looks. The way it sounds. The way it feels. A confident feeling that translates into superior performance.

Precision Advantage. The secret is in the clubface. Computer-designed with optimum roll and bulge characteristic that maximize distance and accuracy. The Coors CeraSports Ceramic Z-Driver features the Zirconia (Z) clubface–a diamond-hard surface, precision-machined to the most exacting tolerances. So its accuracy and clean look are permanent. And in a game of unending variables, you'll welcome the consistency.



Consistent Accuracy. That's only the beginning. To give you every advantage, the Coors CeraSports Ceramic Z-Driver has an aerodynamically sleek tube neck design that maximizes clubhead speed. And because accurate distance is the real test, the tube neck design and deep penetrating shaft virtually eliminate twist and torque.

Drive after drive, your efforts translate into perfection off the tee.

Test Drive The Z-Driver.

Playing the Z-Driver is exhilarating. Visit your pro shop and ask for a test drive. Or call 1-800-252-PUTT. The ceramic difference will change the way you feel about golf. Forever.

Coors CeraSports Ceramic Z-Drivers

Model	Head Size	Shaft Type	Head Color
Men's Z	Standard	Graphite R,S,X	Gray glow
Men's Z	Standard	Steel	Gray glow
Men's ZX	Expanded	Graphite R,S,X	Black glow
Men's ZX	Expanded	Steel	Black glow
Senior' ZS	Expanded	Custom Graphite	Black glow
Ladies' ZL	Standard	Custom Graphite	Gray glow

Only right-handed Z-Drivers are offered at this time.

Ceramicon Designs, Ltd.
A Division of Coors Ceramics Company
600 9th Street, Golden, Colorado 80401
1-800-252-PUTT

Coors CeraSports

Discover The Ceramic Difference

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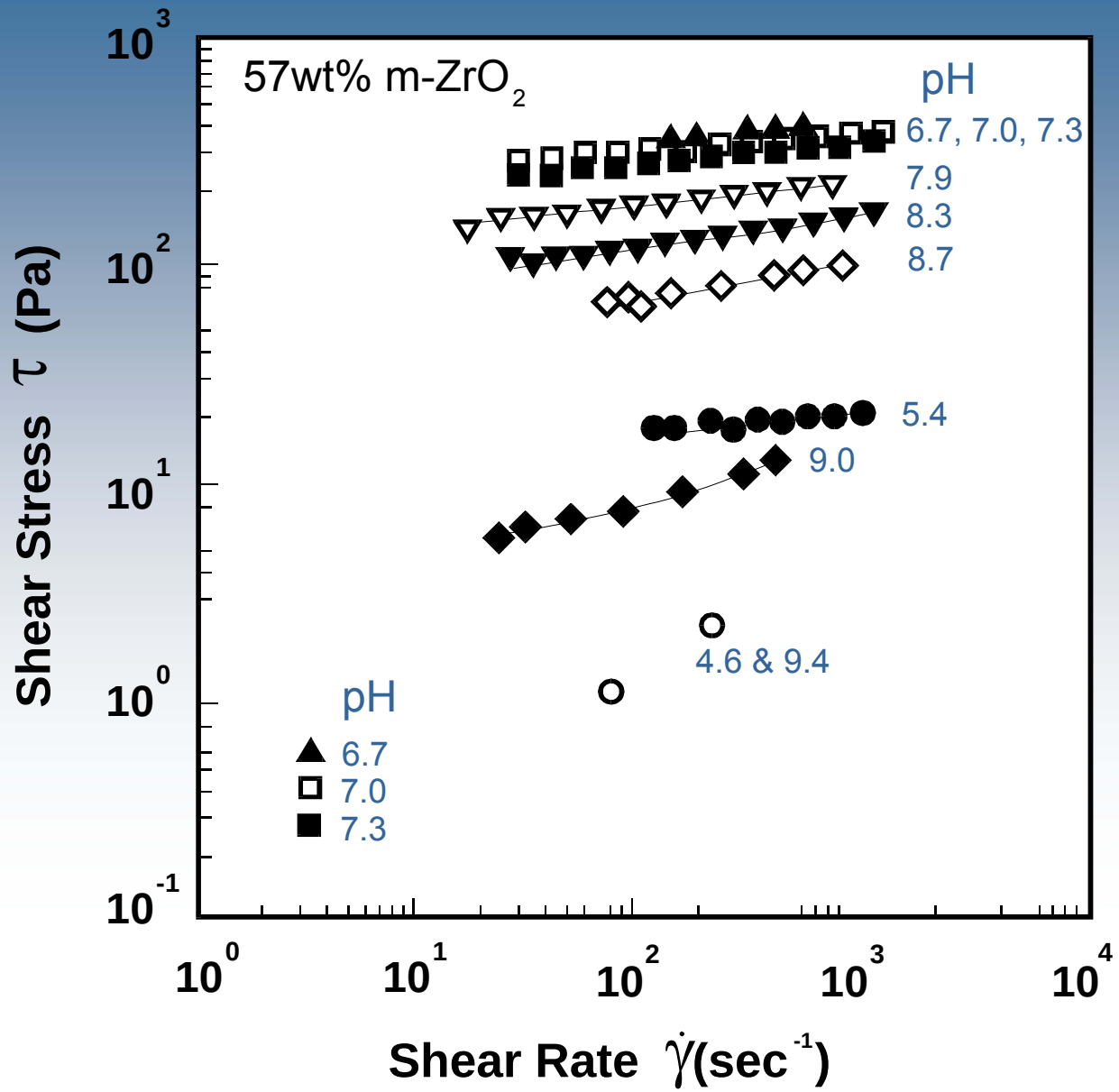
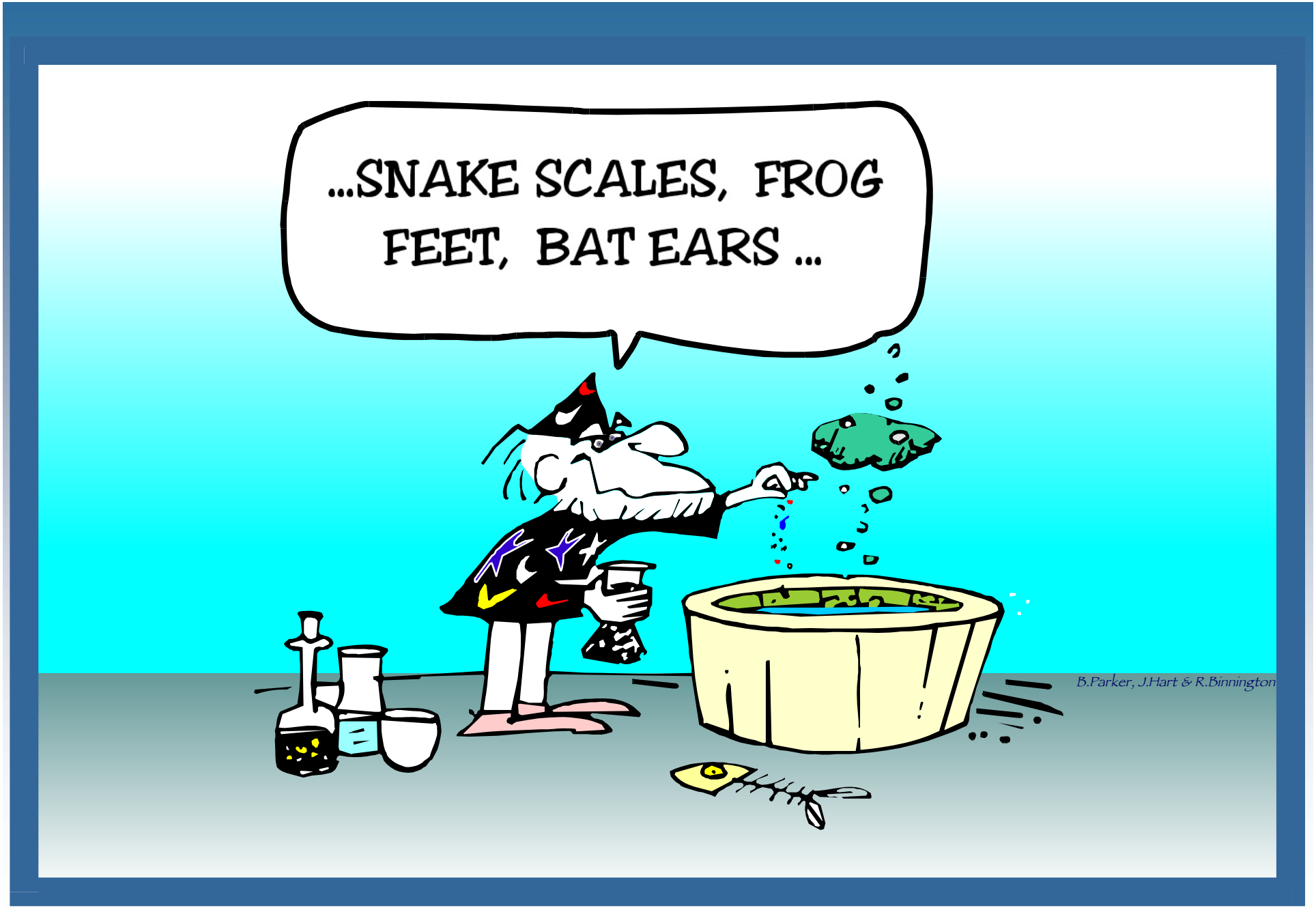
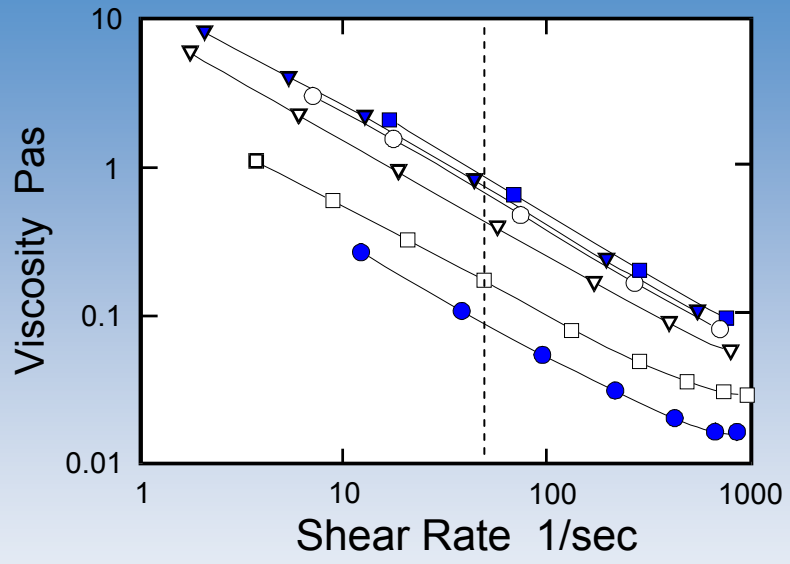

57wt% m-ZrO2
pH
6.7, 7.0, 7.3
7.9
8.3
8.7
5.4
9.0
4.6 & 9.4
pH
6.7
7.0
7.3
Shear Stress τ (Pa)
Shear Rate γ̇(sec-1)
10^3
10^2
10^1
10^0
10^-1
10^0
10^1
10^2
10^3
10^4


...SNAKE SCALES, FROG FEET, BAT EARS ...
B.Parker, J.Hart & R.Binnington

The Rheology of TiO_2 Pigment Suspensions

50wt% solids CR62

Viscosity Profiles as a function of pH



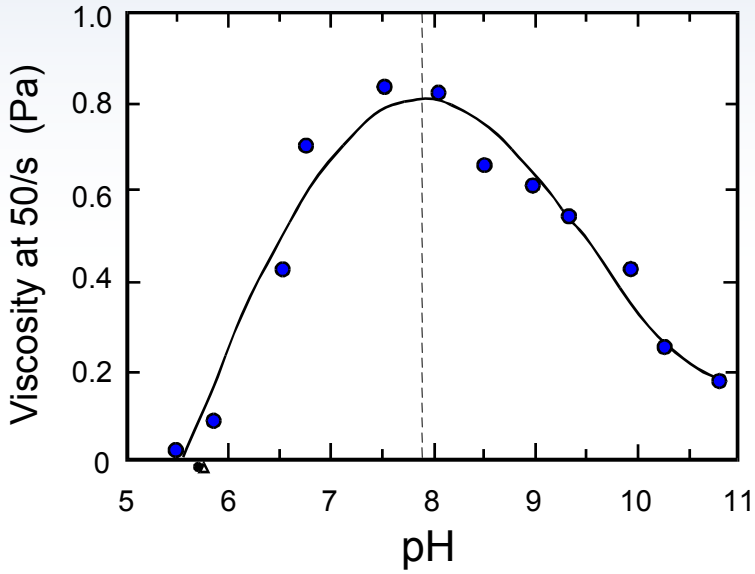
- pH = 5.9
- pH = 6.8
- pH = 7.5
- pH = 8.5
- pH = 9.9
- pH = 10.8

Power Law Behaviour

n = 0.2

2<K<20

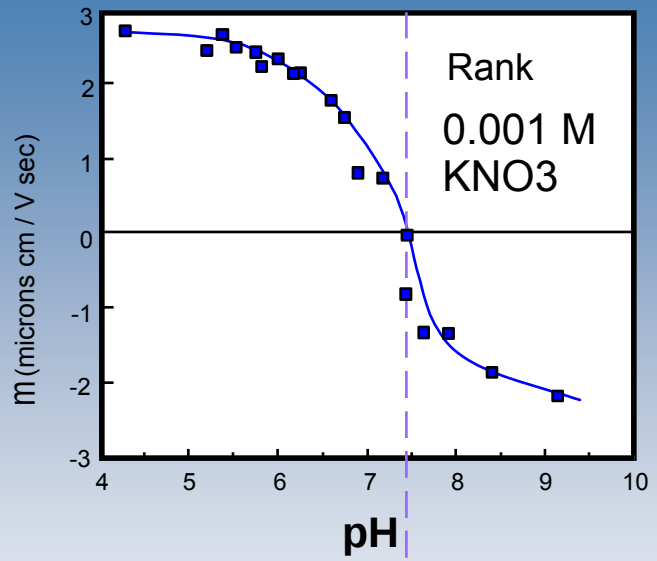
Viscosity Curve



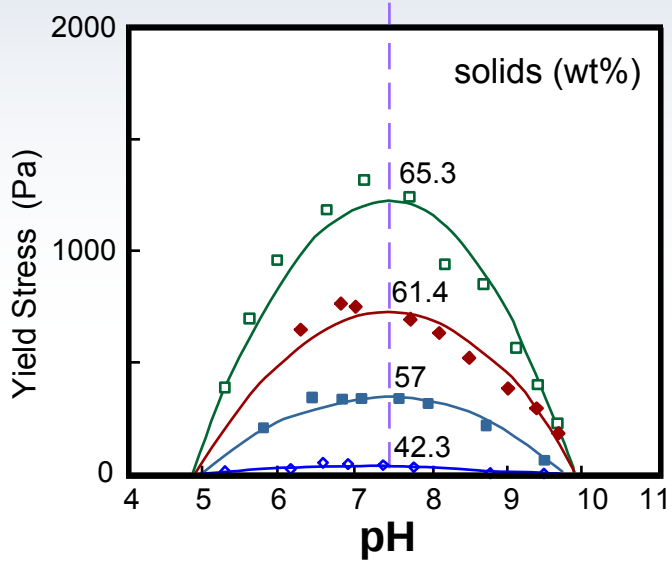
η_{max} occurs at pH = 7.9

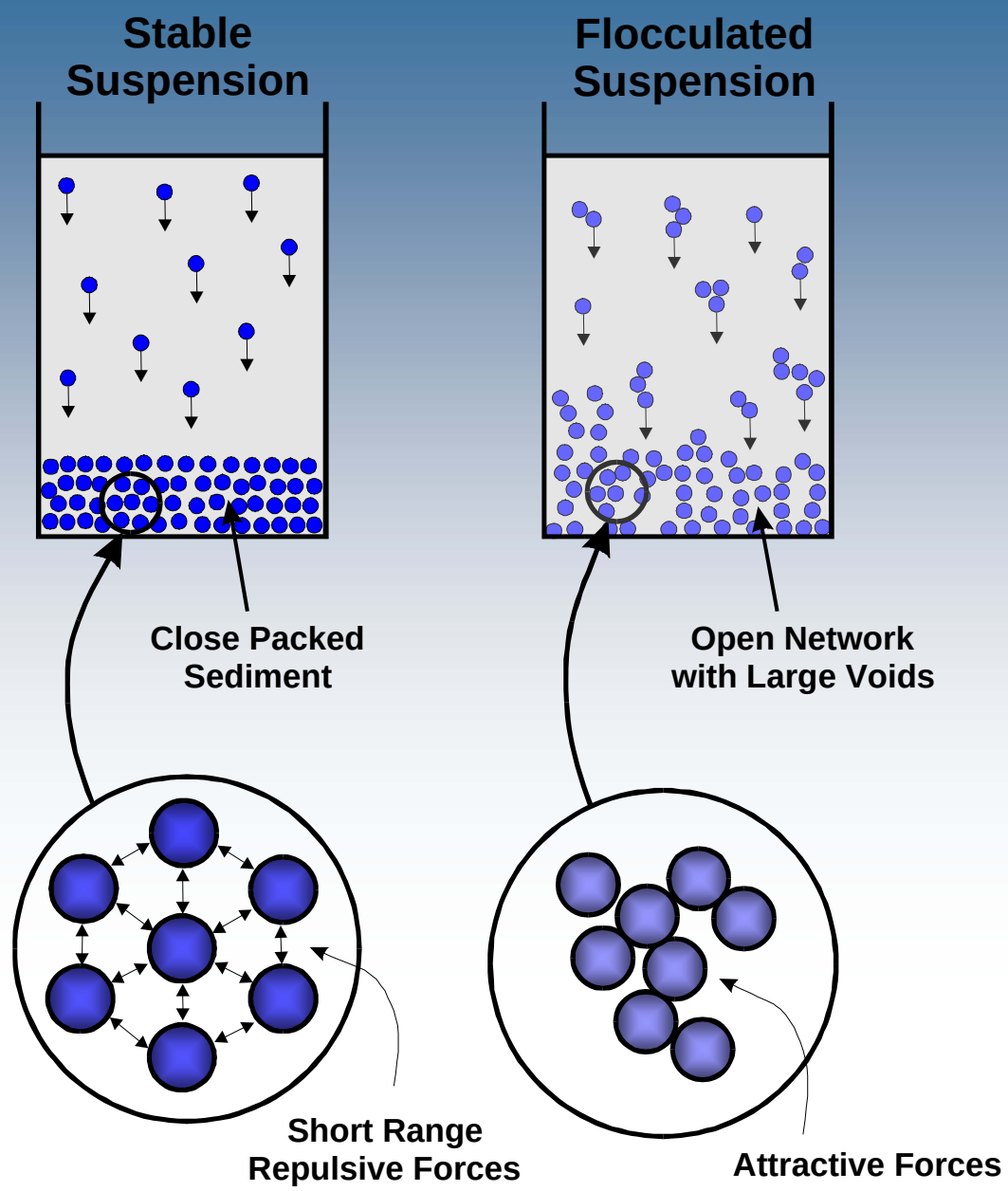
A.4.122

1. Electrokinetics

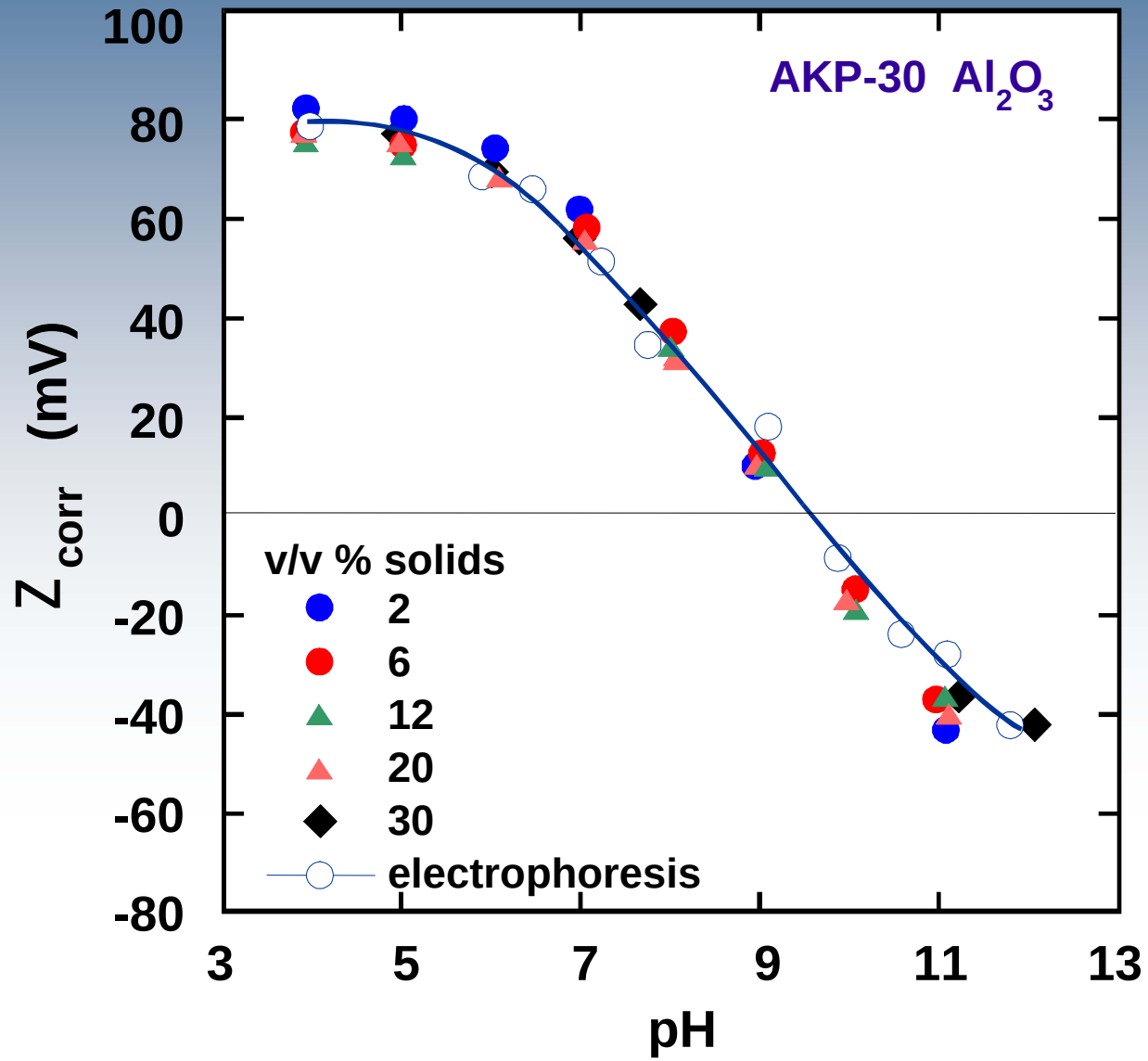


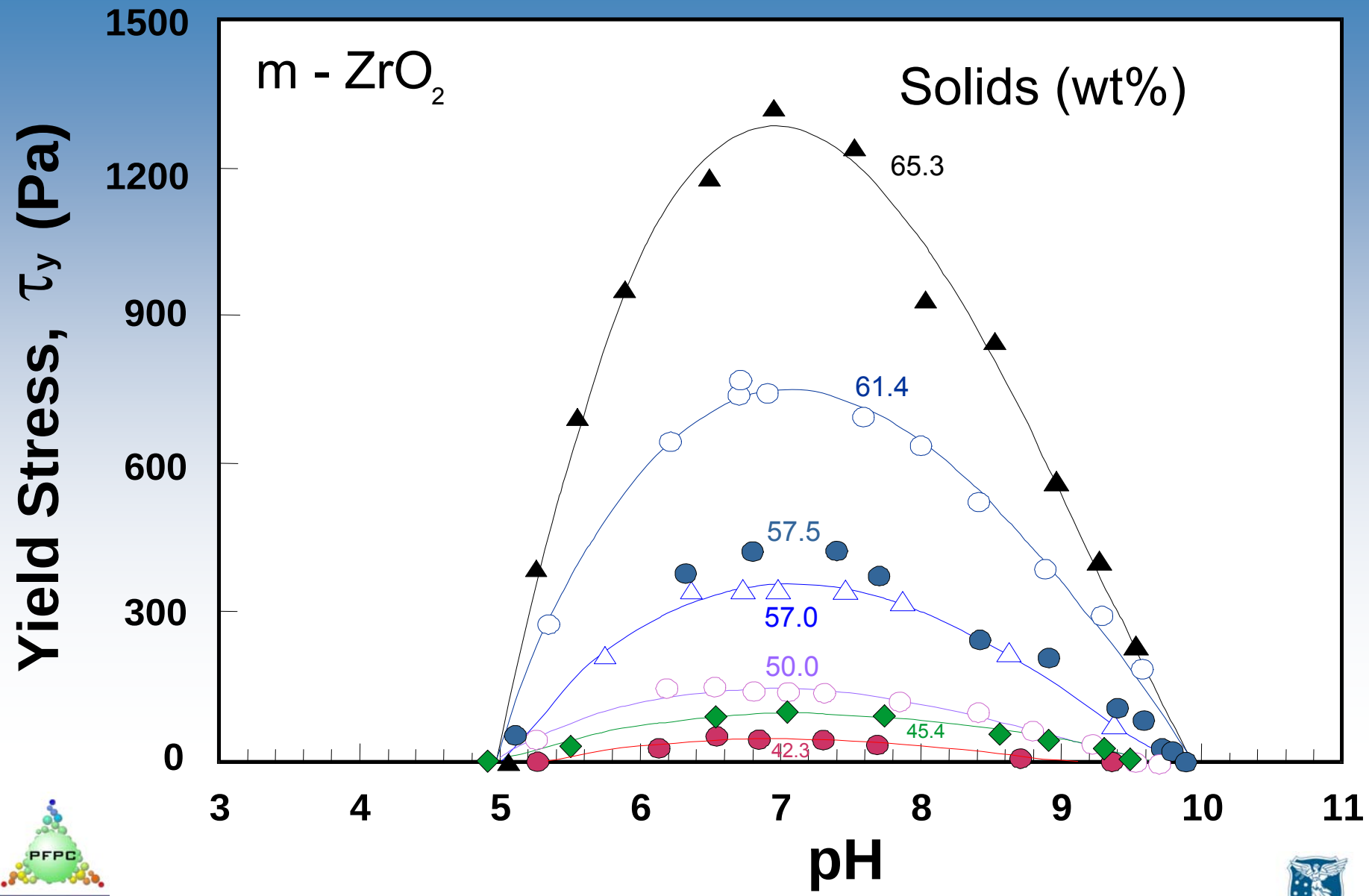
2. Rheology




Stable Suspension
Flocculated Suspension
Close Packed Sediment
Open Network with Large Voids
Short Range Repulsive Forces
Attractive Forces

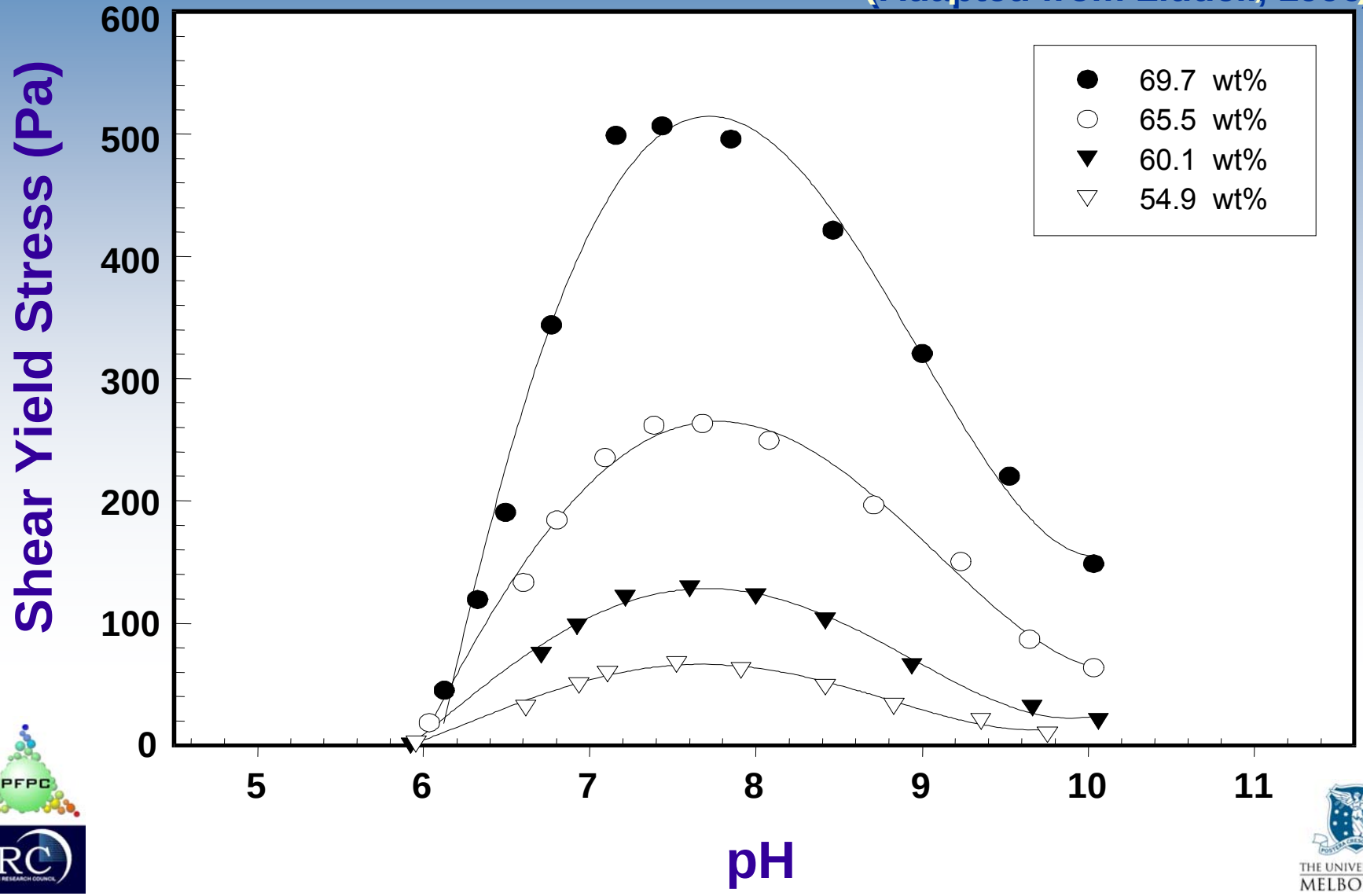
Alumina Electrokinetics




m - ZrO2
Solids (wt%)
65.3
61.4
57.5
57.0
50.0
45.4
42.3
Yield Stress, τy (Pa)
1500
1200
900
600
300
0
3
4
5
6
7
8
9
10
11
pH

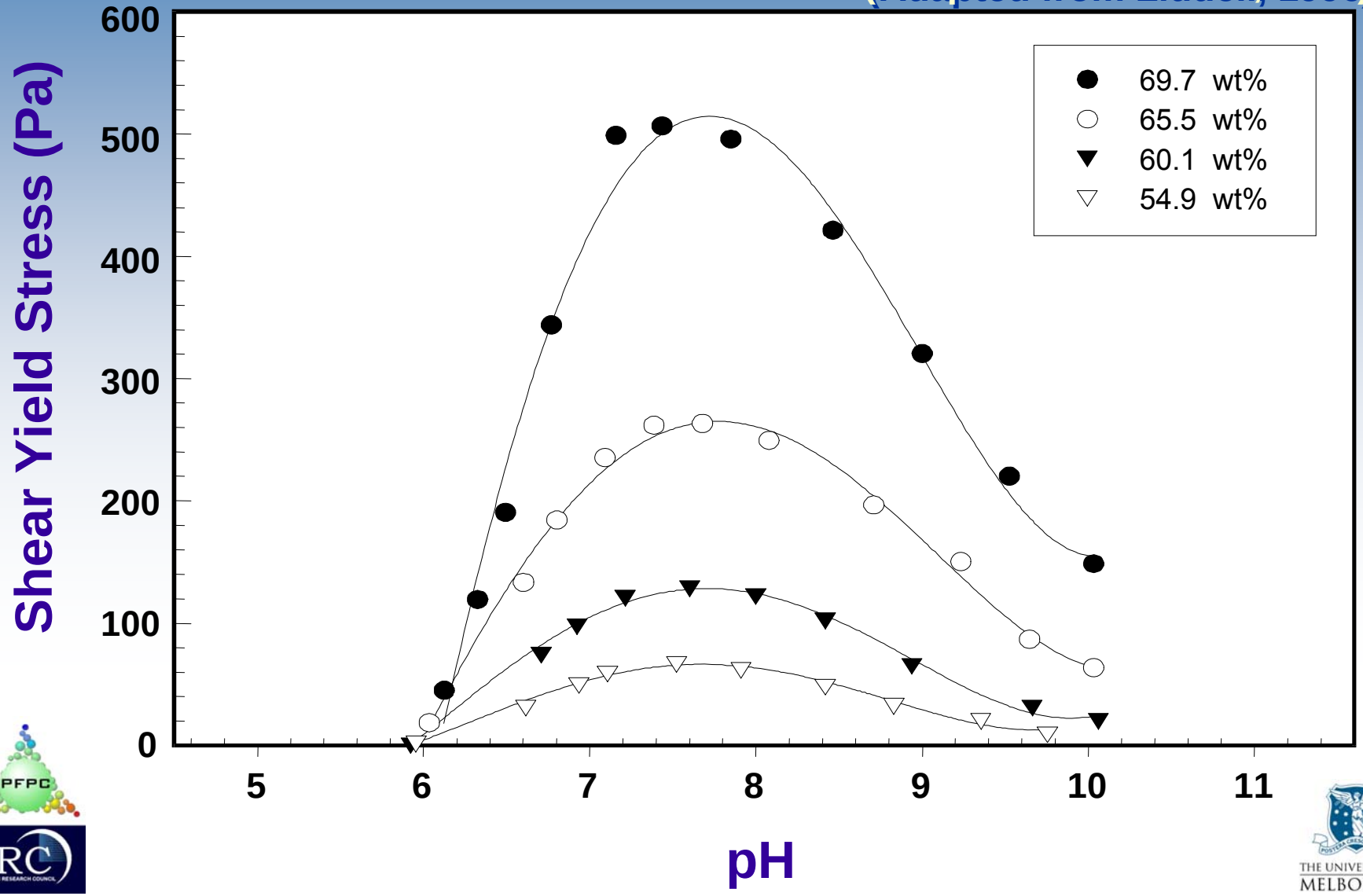
Yield Stress of Titania Suspensions as a Function of pH and Solids Concentration

(Adapted from Liddell, 1996)



Yield Stress of Titania Suspensions as a Function of pH and Solids Concentration

(Adapted from Liddell, 1996)



Additive Effects -Type of Interaction

a) STERIC INTERACTION

- **i) illustrated by small additive molecules**
- **ii) modelling to show the importance of additive molecular size**

b) BRIDGING FLOCCULATION

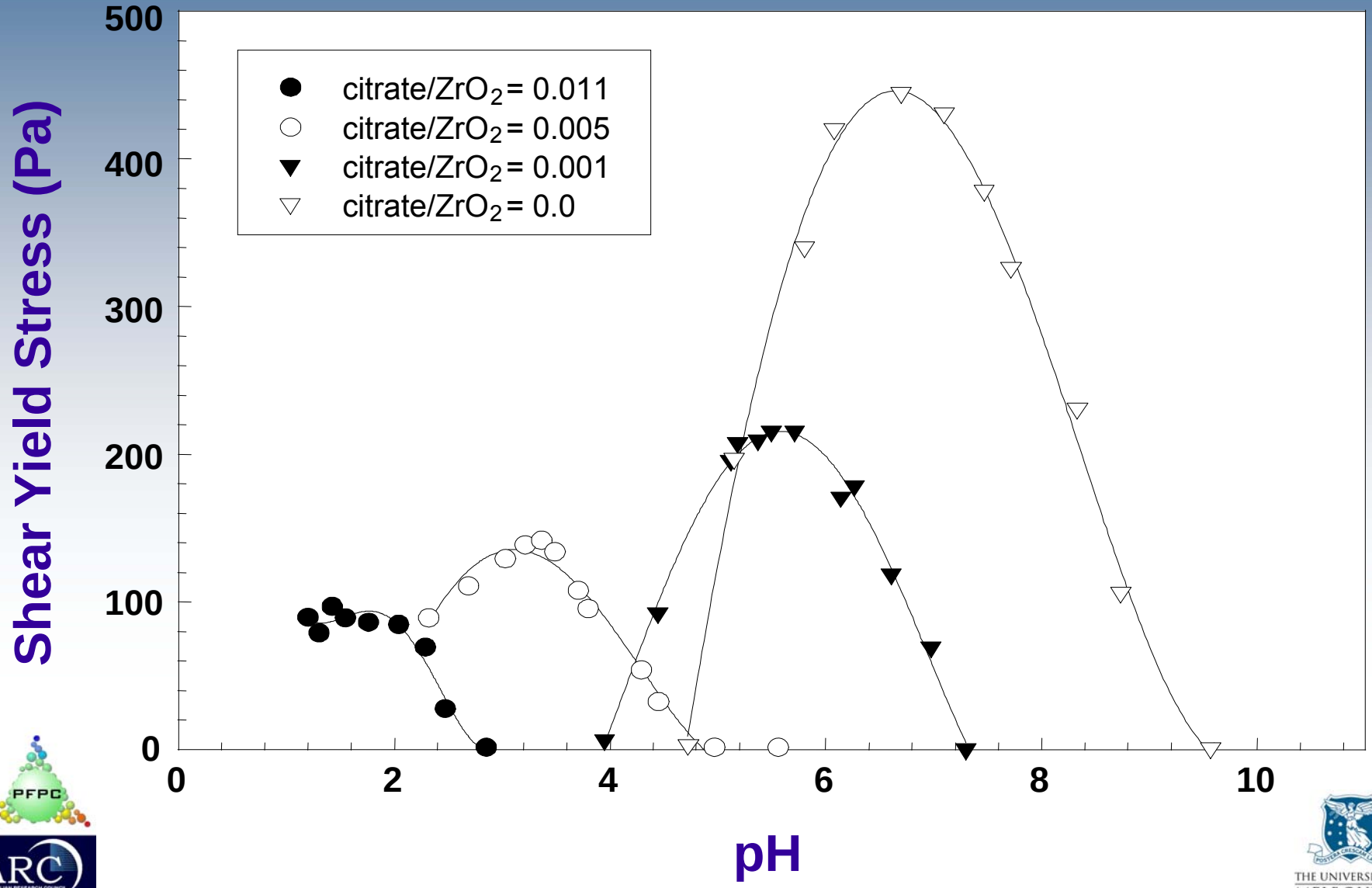
- **i) high molecular weight polyelectrolyte**

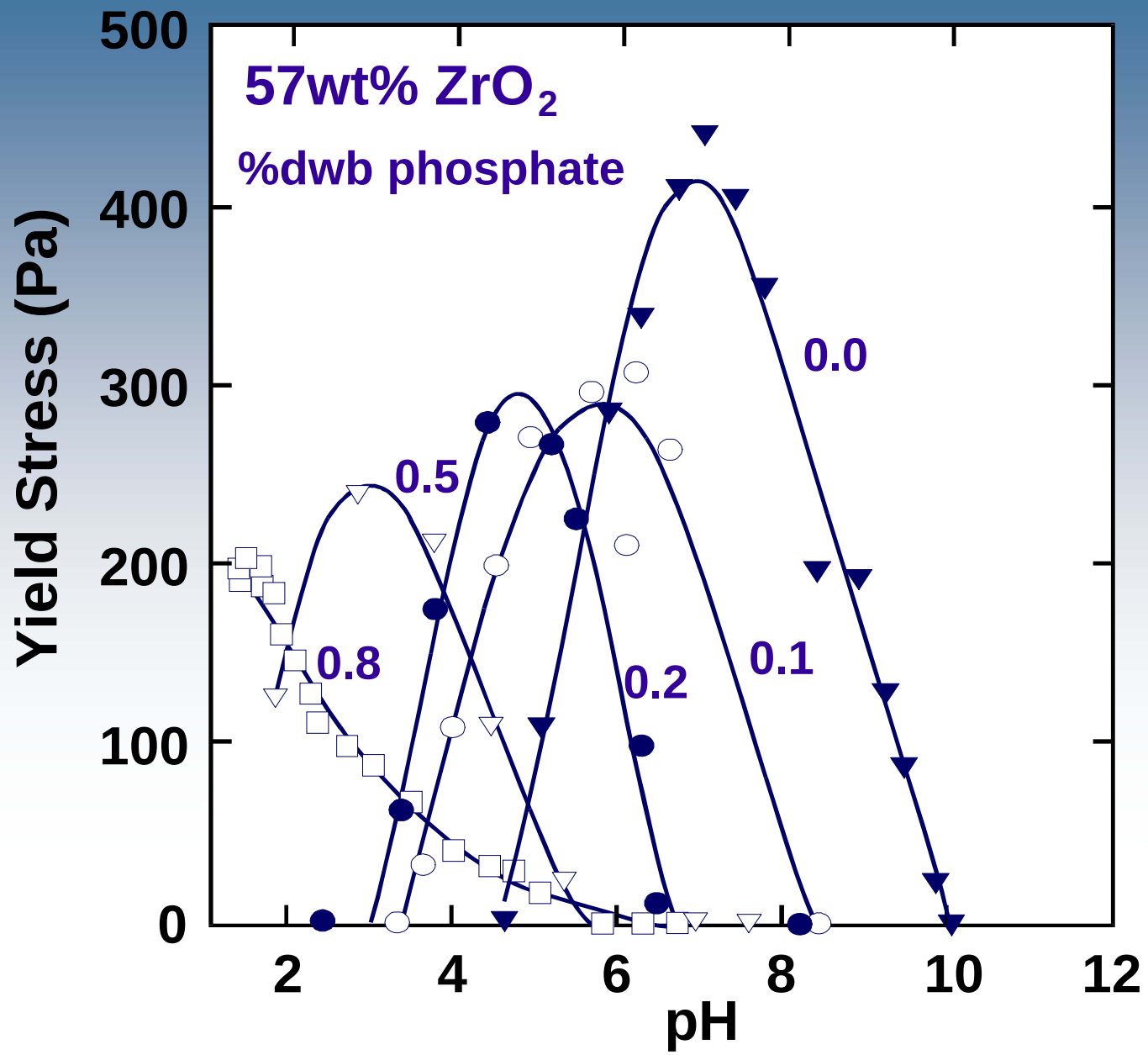
c) HYDROPHOBIC INTERACTION

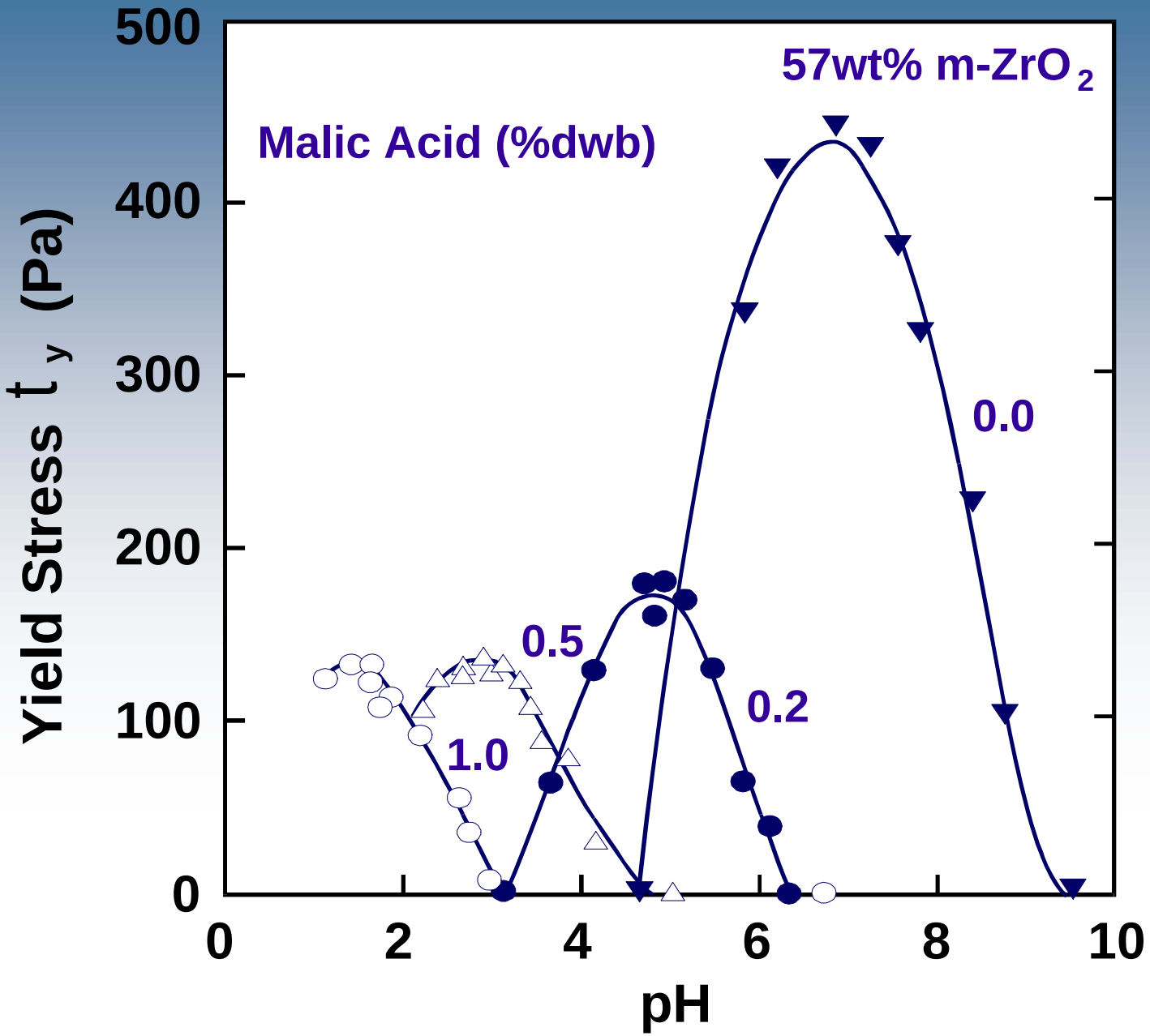
- **i) additive with a charge head group and a long hydrophobic tail, eg. dodecyl sulphate**

Note that most analyses were conducted at the IEP so as to eliminate complication arising from electrostatic interaction

Yield Stress versus pH behaviour of 57% Zirconia Suspension as a Function of Citrate Concentration

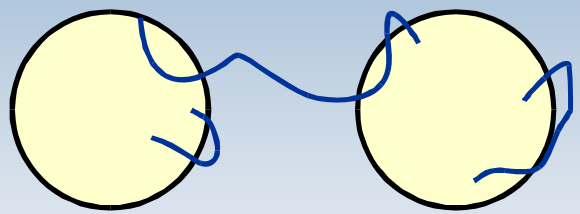



57wt% ZrO_2
%dwb phosphate
Yield Stress (Pa)
pH
0.0
0.1
0.2
0.5
0.8
500
400
300
200
100
0
2
4
6
8
10
12


57wt% m-ZrO2
Malic Acid (%dwb)
0.0
0.5
0.2
1.0
Yield Stress τy (Pa)
pH
500
400
300
200
100
0
0
2
4
6
8
10

Bridging Flocculation

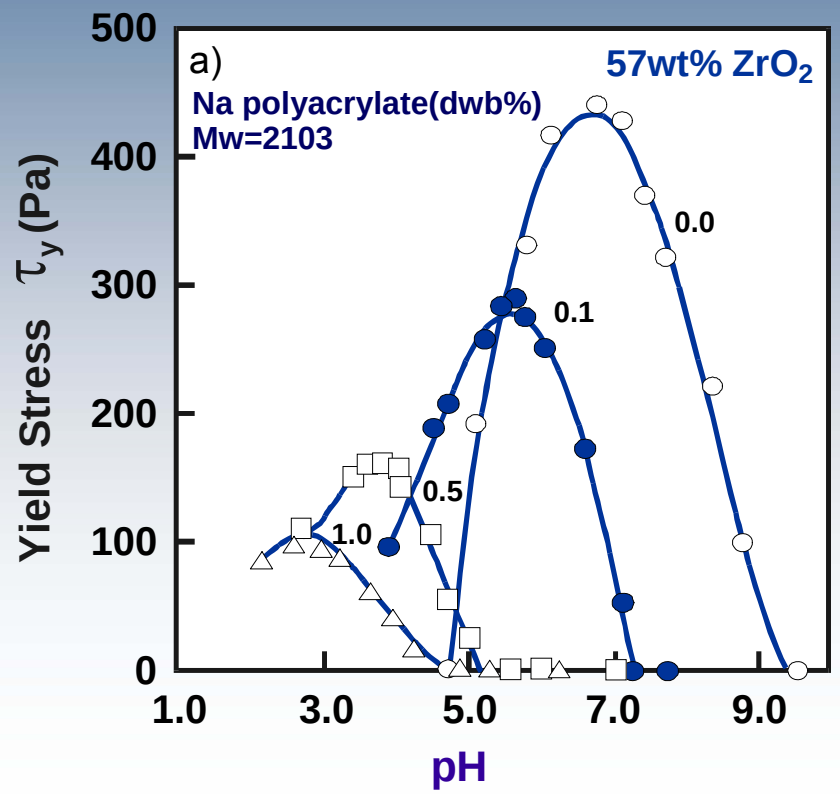
When a high Mw polymer is adsorbed onto 2 or more particles at the same time.

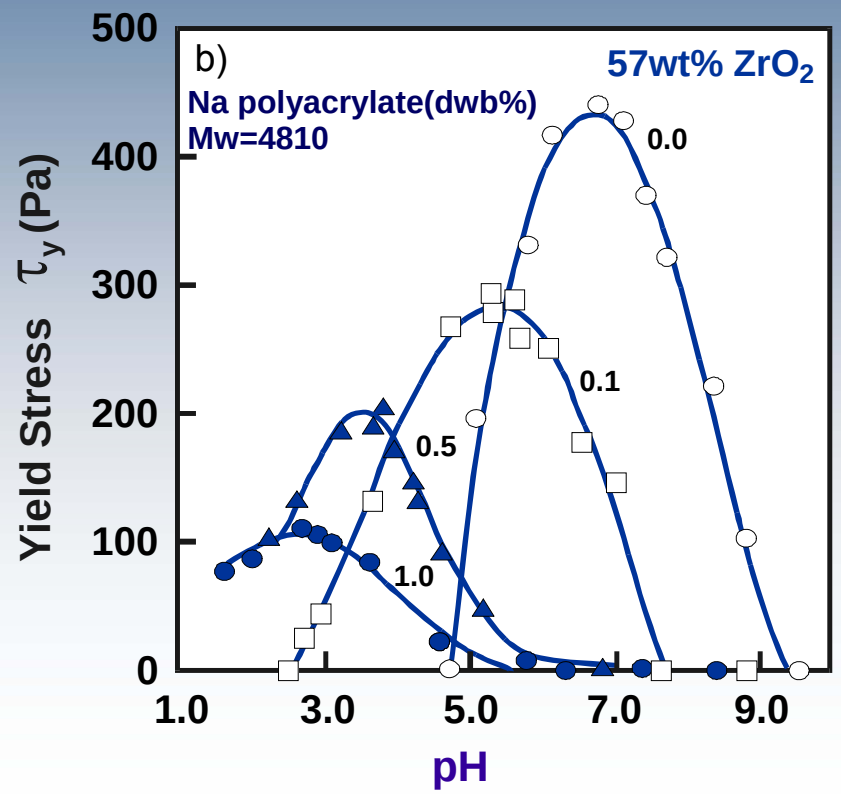


Bridging flocculation is an additional attractive force and is only important for high Mw polymer and at low additive concentration.

Z-Tech ZrO_2, S.A. = 15 m^2/g

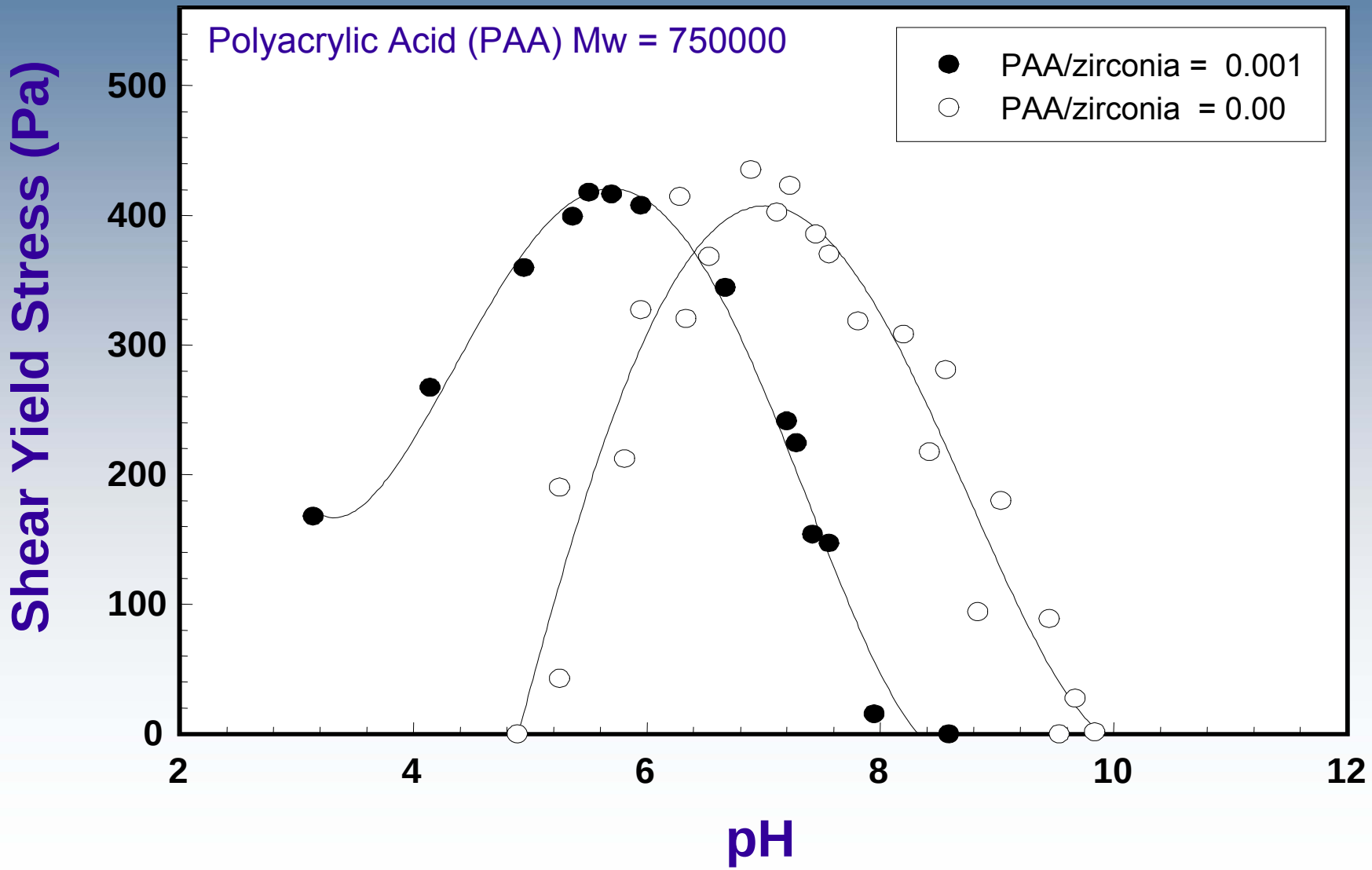
Yield stress-pH behaviour of 57wt% ZrO_2 suspensions treated with sodium polyacrylate of molecular weight: a)2103, b)4810

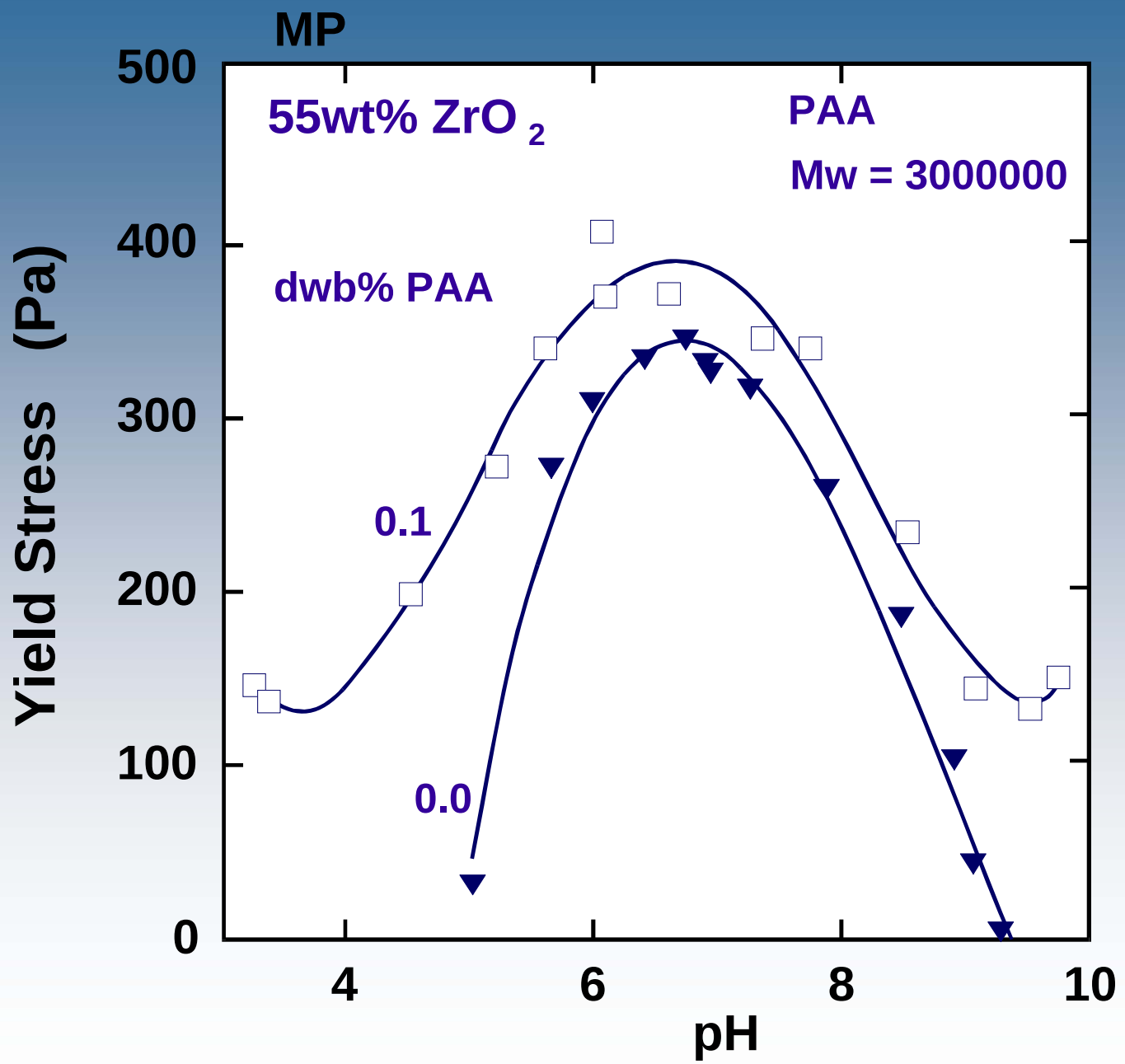




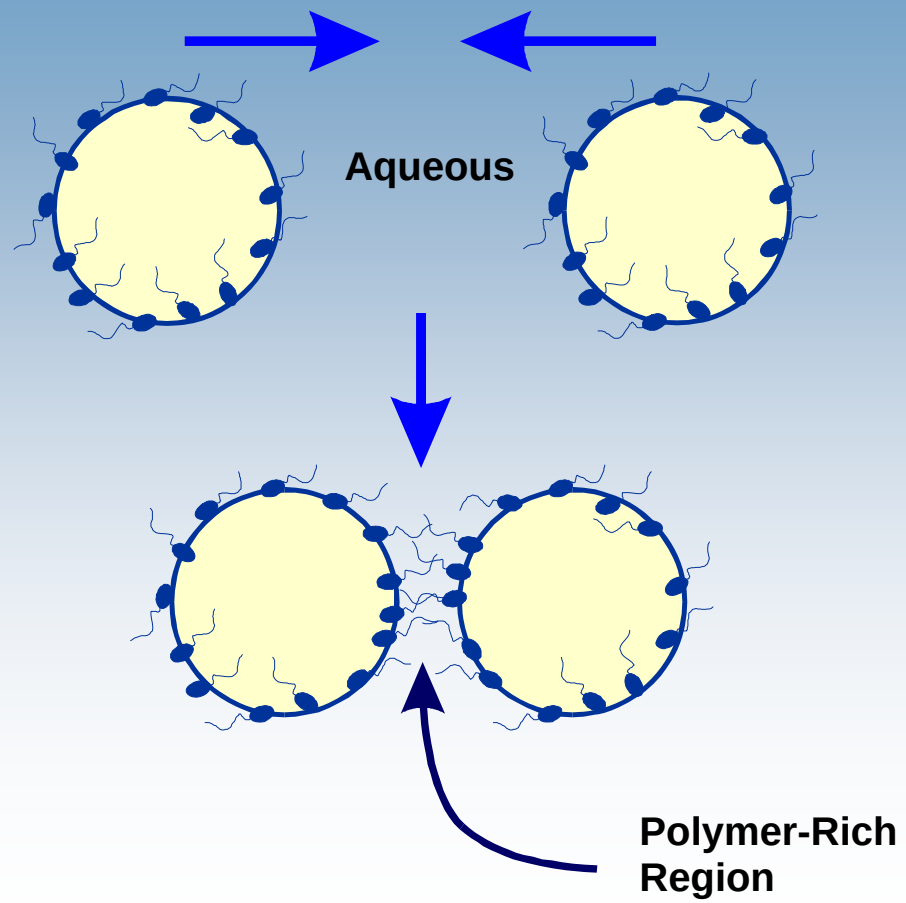
Leong, Y.K., Scales, P.J., Healy T.W. and Boger, D.V.,
94th Annual Meeting, American Ceramic Society, Minneapolis (1992)

Shear Yield Stress of Zirconia Suspension at 57wt% as a Function of pH and the Concentration of High Molecular Weight PAA




MP
55wt% ZrO 2
PAA
Mw = 3000000
dwb% PAA
0.1
0.0
Yield Stress (Pa)
500
400
300
200
100
0
4
6
8
10
pH

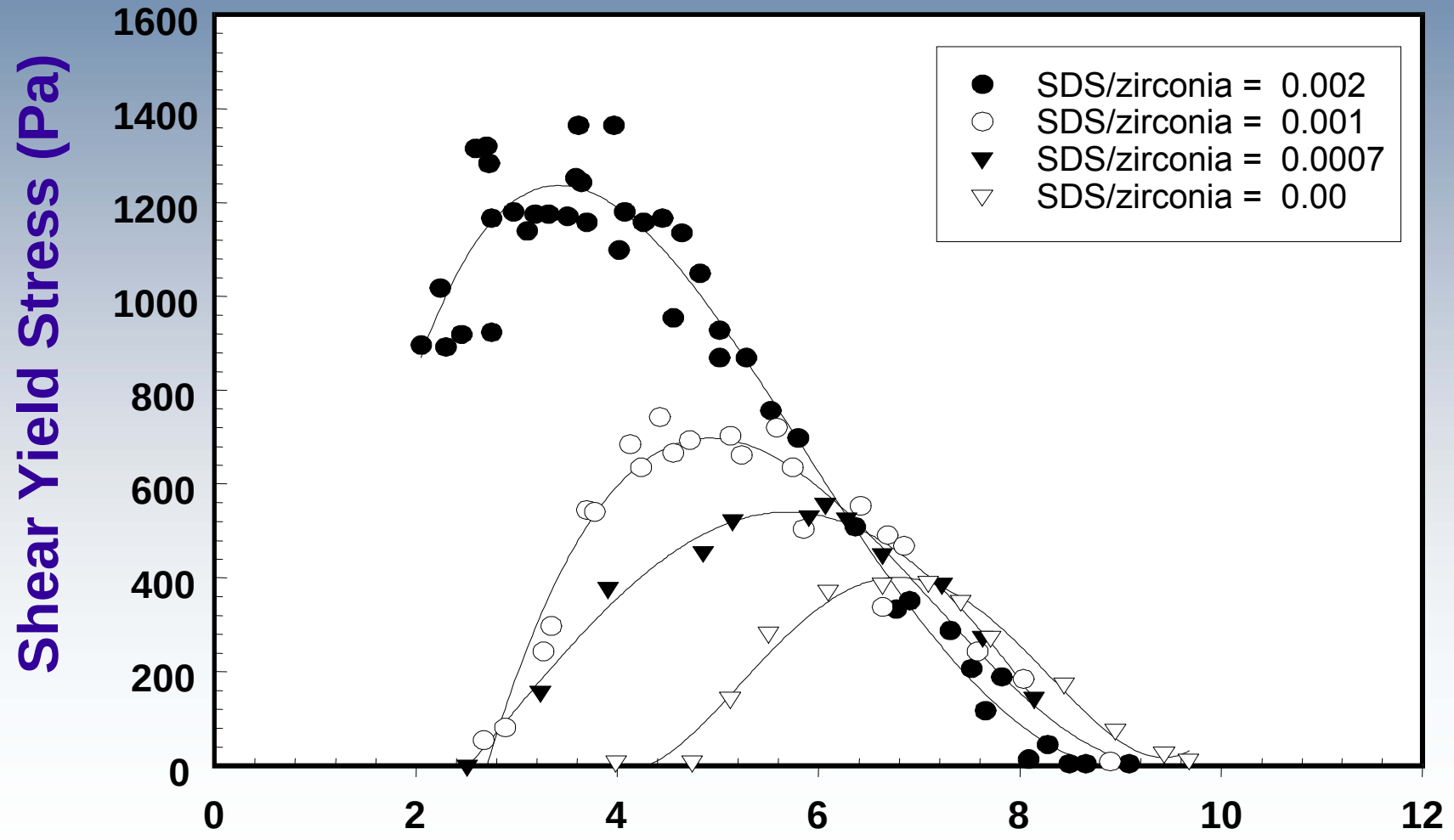
Hydrophobic Interaction



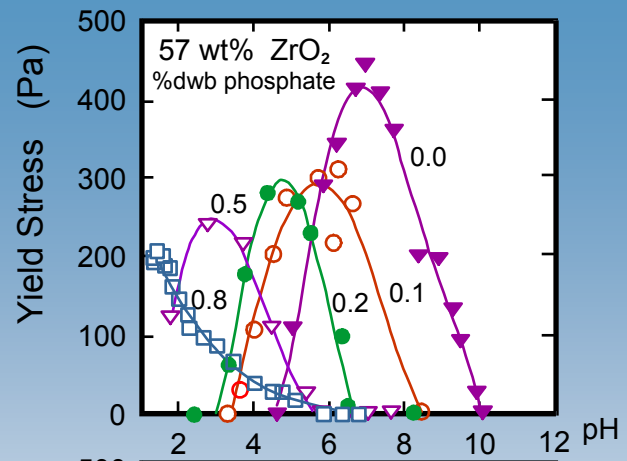
At IEP: $V_T = V_{VdW} + V_{hydrophobic}$

Hydrophobic interaction occurs when the adsorbed polymer chains have greater affinity for each other than for the aqueous medium

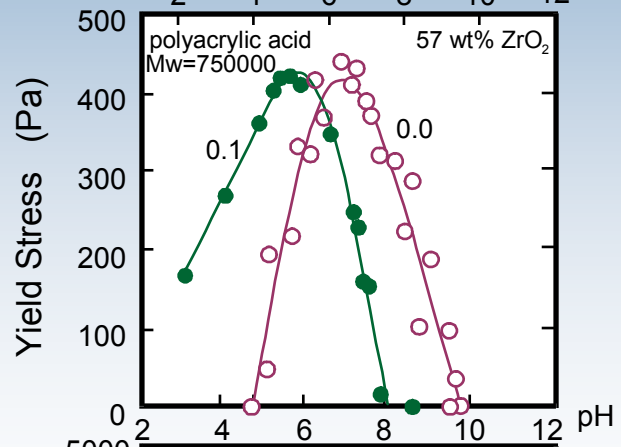
Shear Yield Stress of Zirconia Suspensions at 57% as a function of pH and Concentration of Sodium Dodecyl Sulfate (SDS)



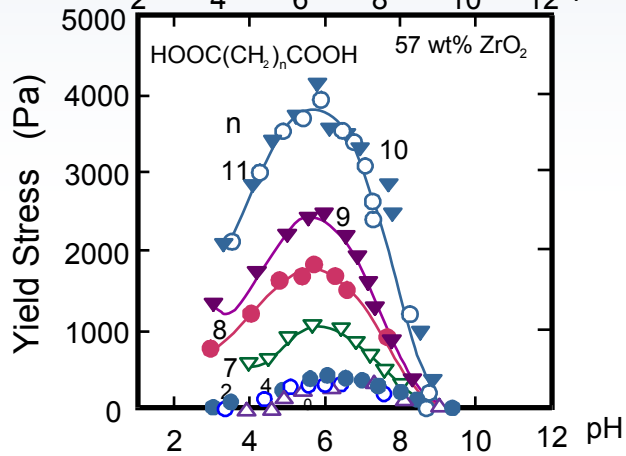
STERIC

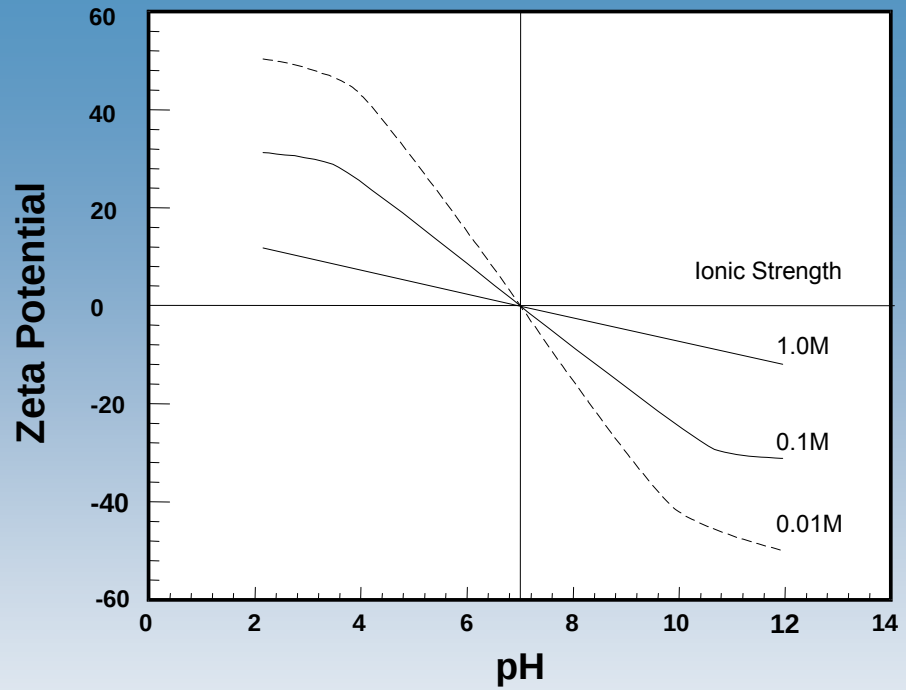


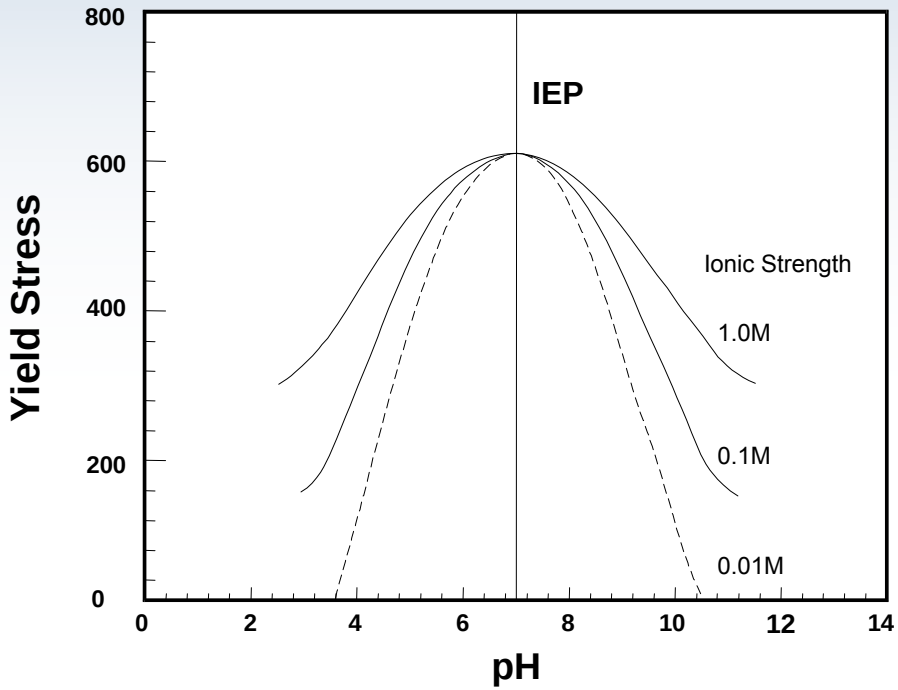
BRIDGING



HYDROPHOBIC



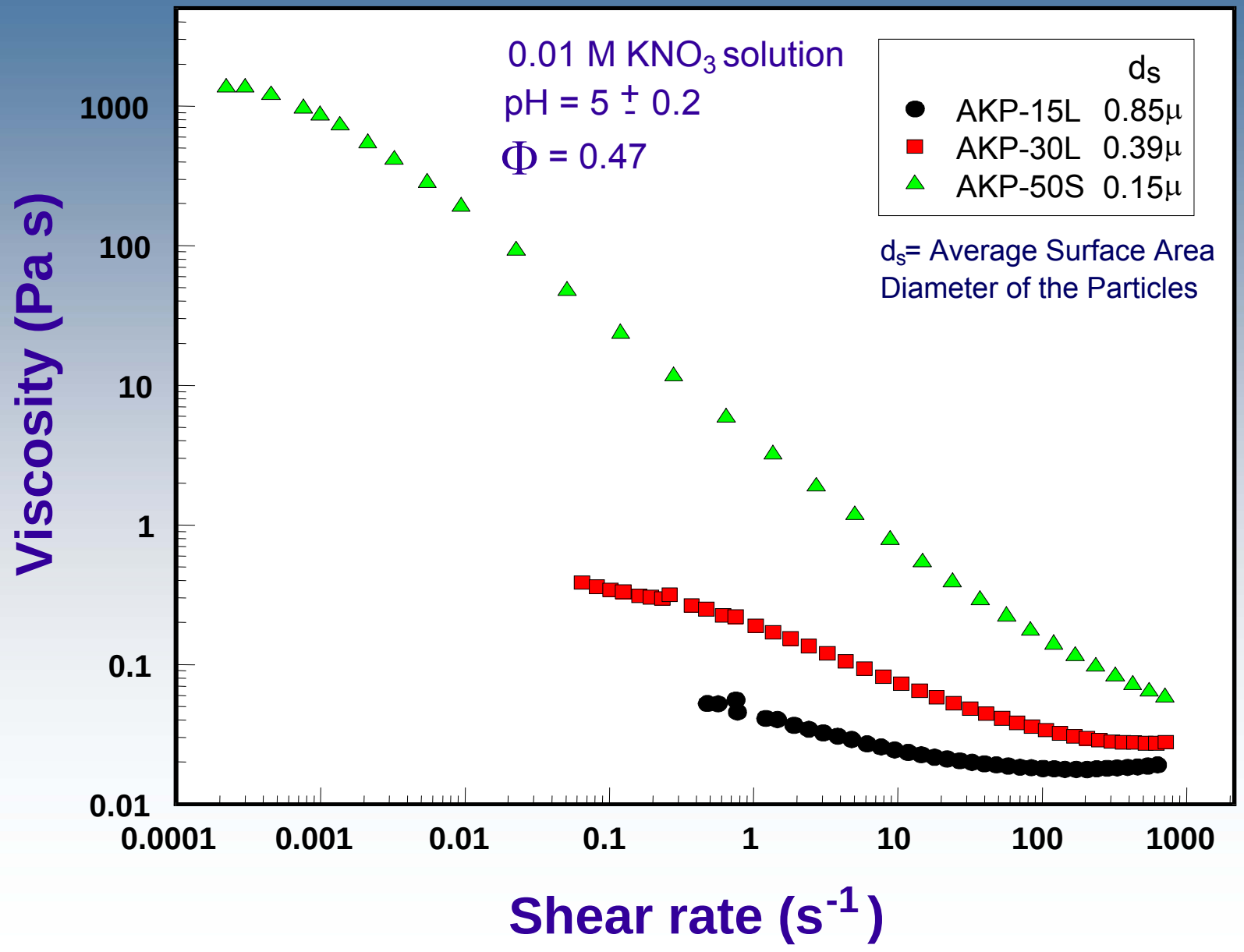

Zeta Potential
60
40
20
0
-20
-40
-60
0
2
4
6
8
10
12
14
pH
Ionic Strength
1.0M
0.1M
0.01M


Yield Stress
800
600
400
200
0
0
2
4
6
8
10
12
14
pH
IEP
Ionic Strength
1.0M
0.1M
0.01M

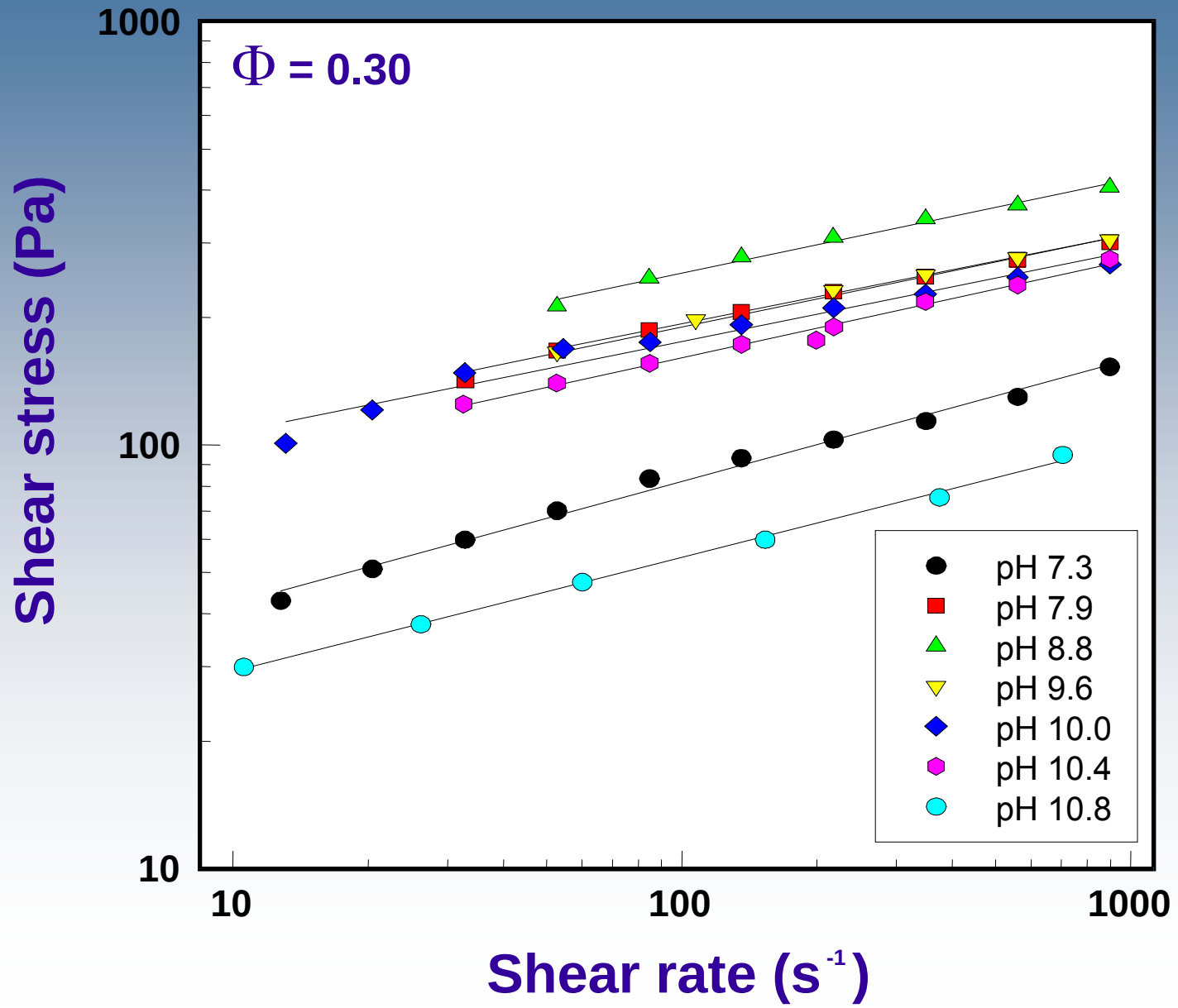
Properties of Alumina Particles

Samples	Density (g/cm^3)	BET Surface Area (m^2/g)	Mean Diameter (mm)
AKP-15	3.99	3.8	0.7
AKP-20	3.98	4.3	0.54
AKP-30	3.96	6.8	0.36
AKP-50	3.95	10.5	0.18

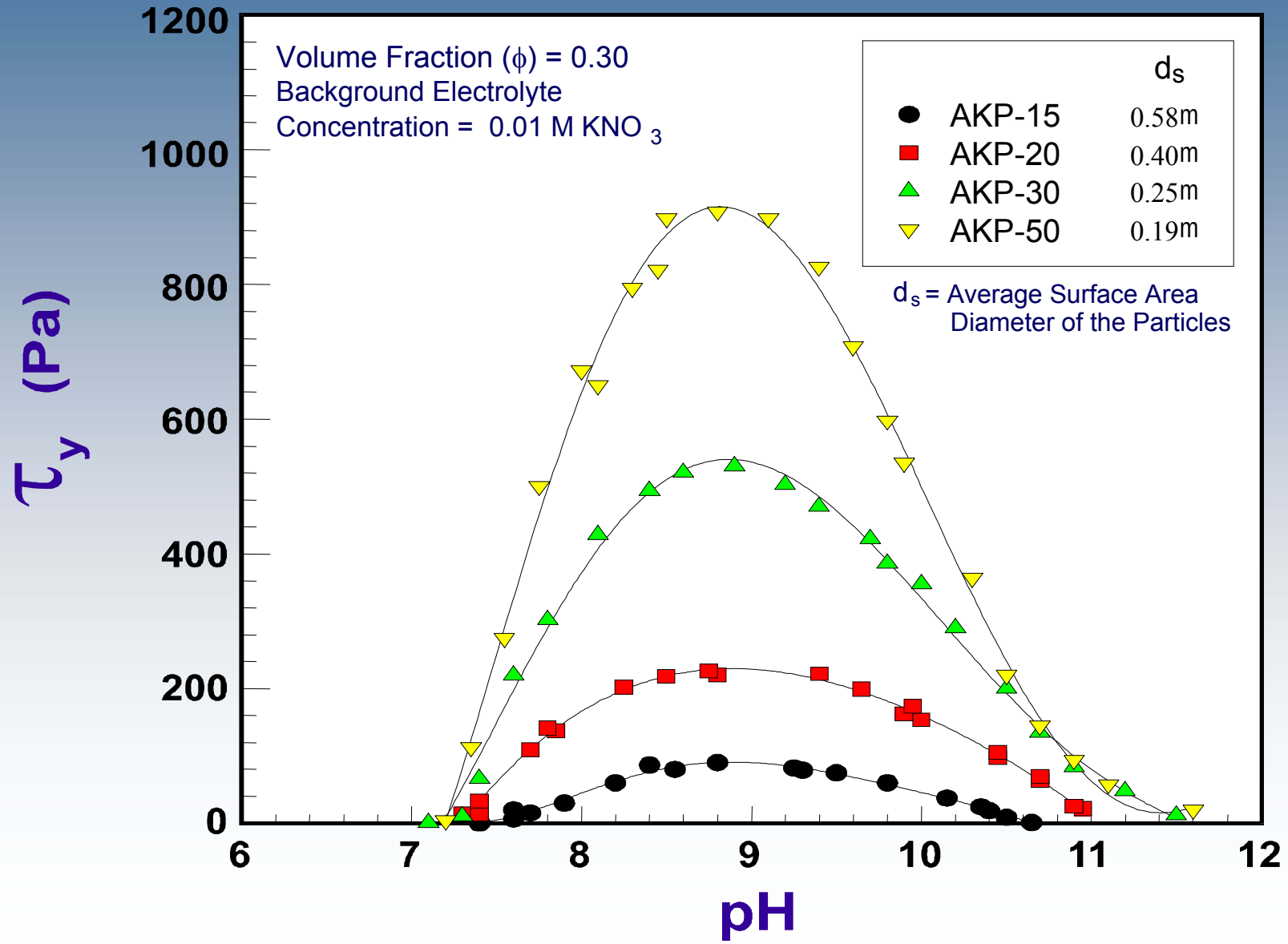
The Effect of Particle Size on the Viscosity of Alumina Suspensions



Flow Curves for AKP-20 Suspensions at various pH



Yield Stress as a Function of pH for Alumina Suspensions of Different Particle Sizes



For Monodisperse Particle Systems

At the iep

$$\tau_y = \frac{0.011\phi K(\phi,d)}{\pi}\left(\frac{A}{h_0^2}\right)\frac{1}{d}$$

where A is the Hamaker constant
h_0 is the interparticle spacing at contact
d is the particle diameter

Away from the iep

$$\tau_y = \frac{0.011\phi K(\phi,d)}{\pi}\left(\frac{A}{h_0^2} - \frac{24\pi\varepsilon\kappa\zeta^2}{(1+e^{\kappa h_0})}\right)\frac{1}{d}$$

Normalised Yield Stress

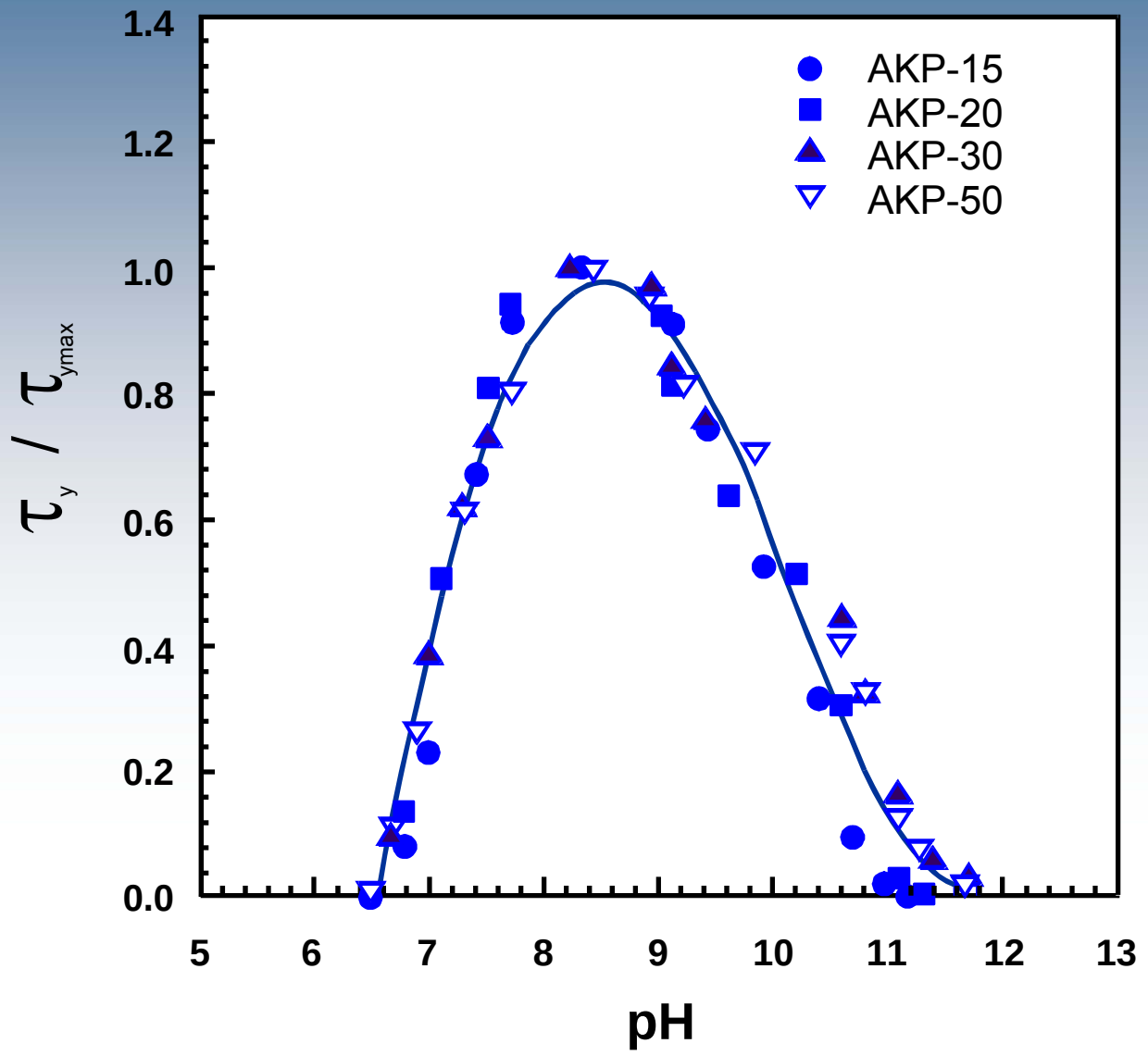
$$\frac{\tau_y}{\tau_{ymax}} = 1 - \frac{24\pi\varepsilon\kappa\zeta^2 h_0^2}{A(1+e^{\kappa h_0})}$$

No Volume Fraction Dependence

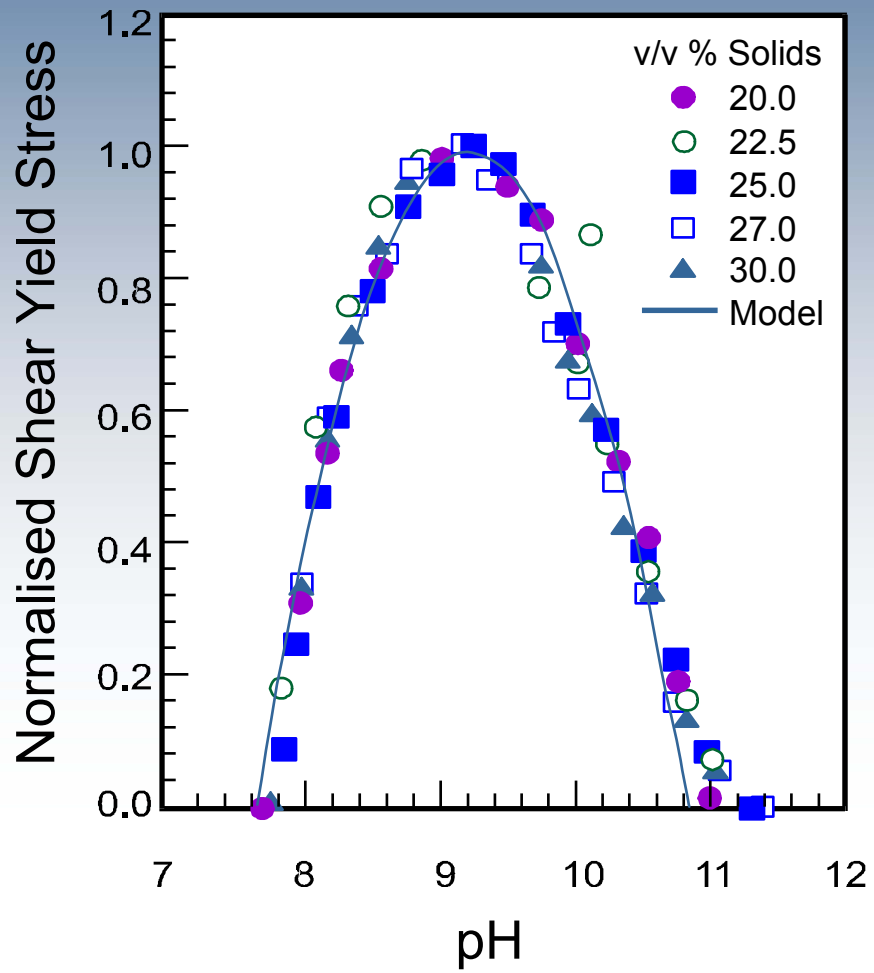
No Size Dependence

h_0 is the only Fitting Parameter

Force Comparison



Effect of Volume Fraction on Yield Stress

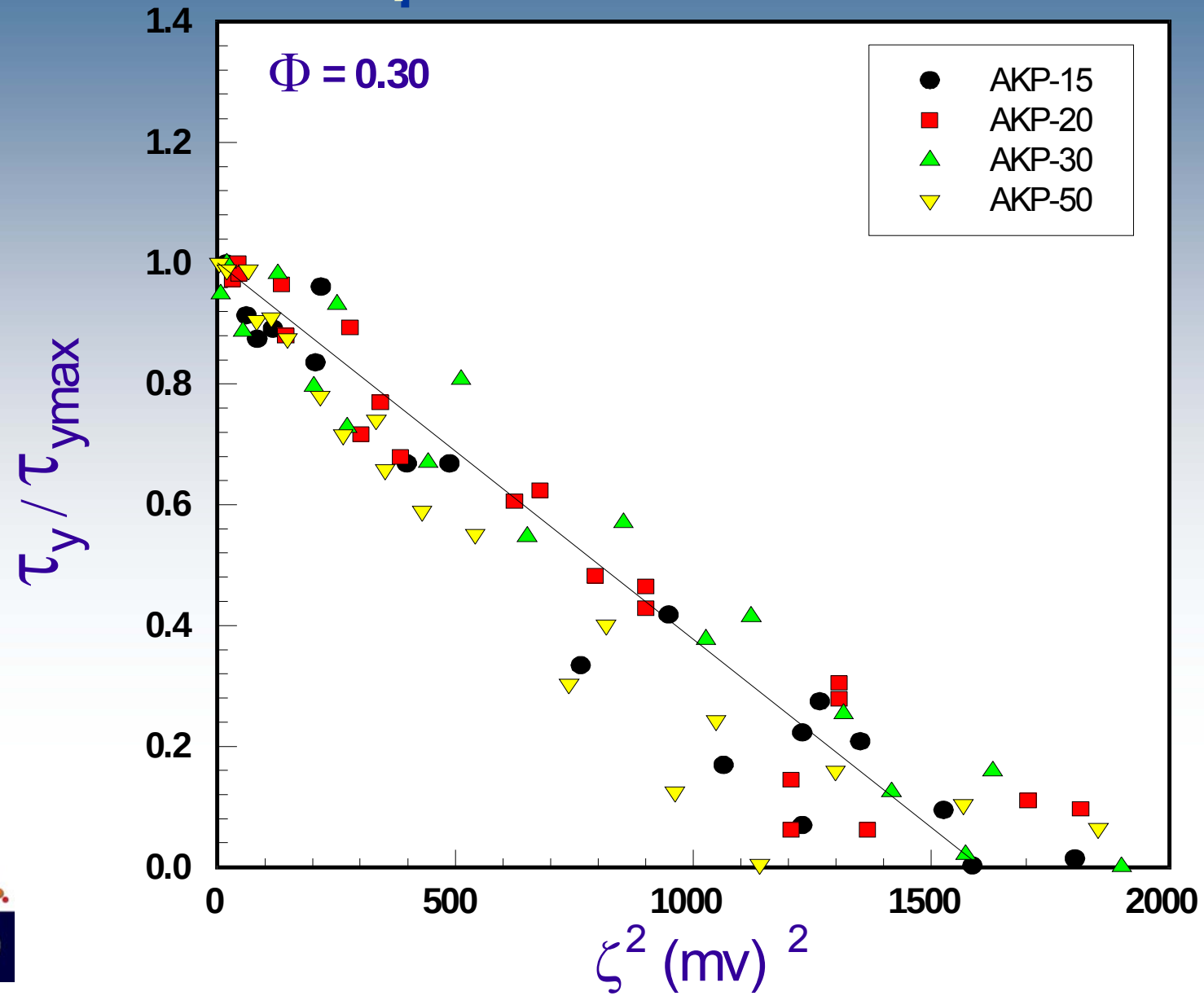


Normalised Yield Stress

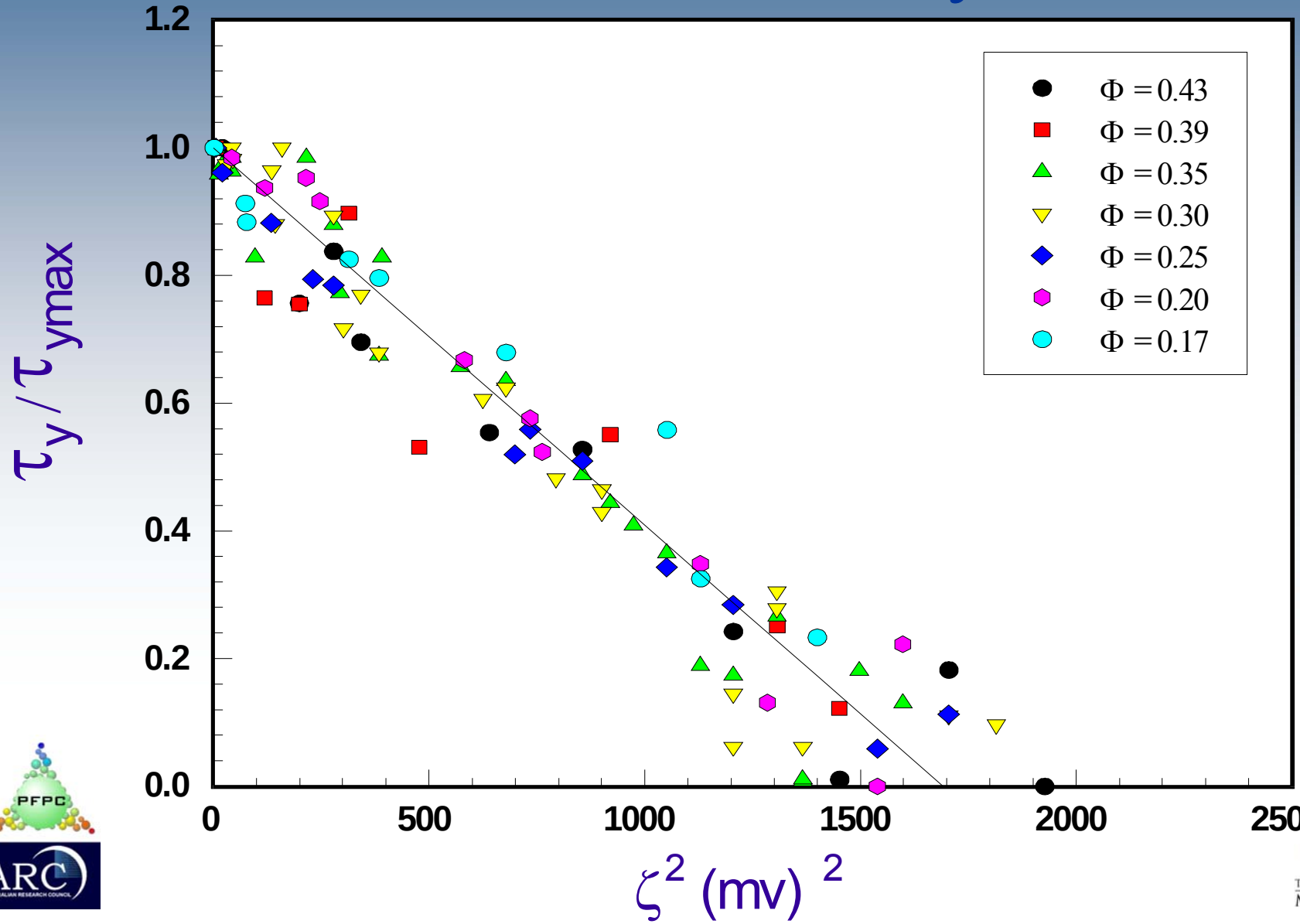
$$\frac{\tau_y}{\tau_{ymax}} = 1 - \frac{24\pi\varepsilon\kappa\zeta^2 h_0^2}{A(1+e^{\kappa h_0})}$$

- No Volume Fraction Dependence
- No Size Dependence
- h_0 is the only Fitting Parameter

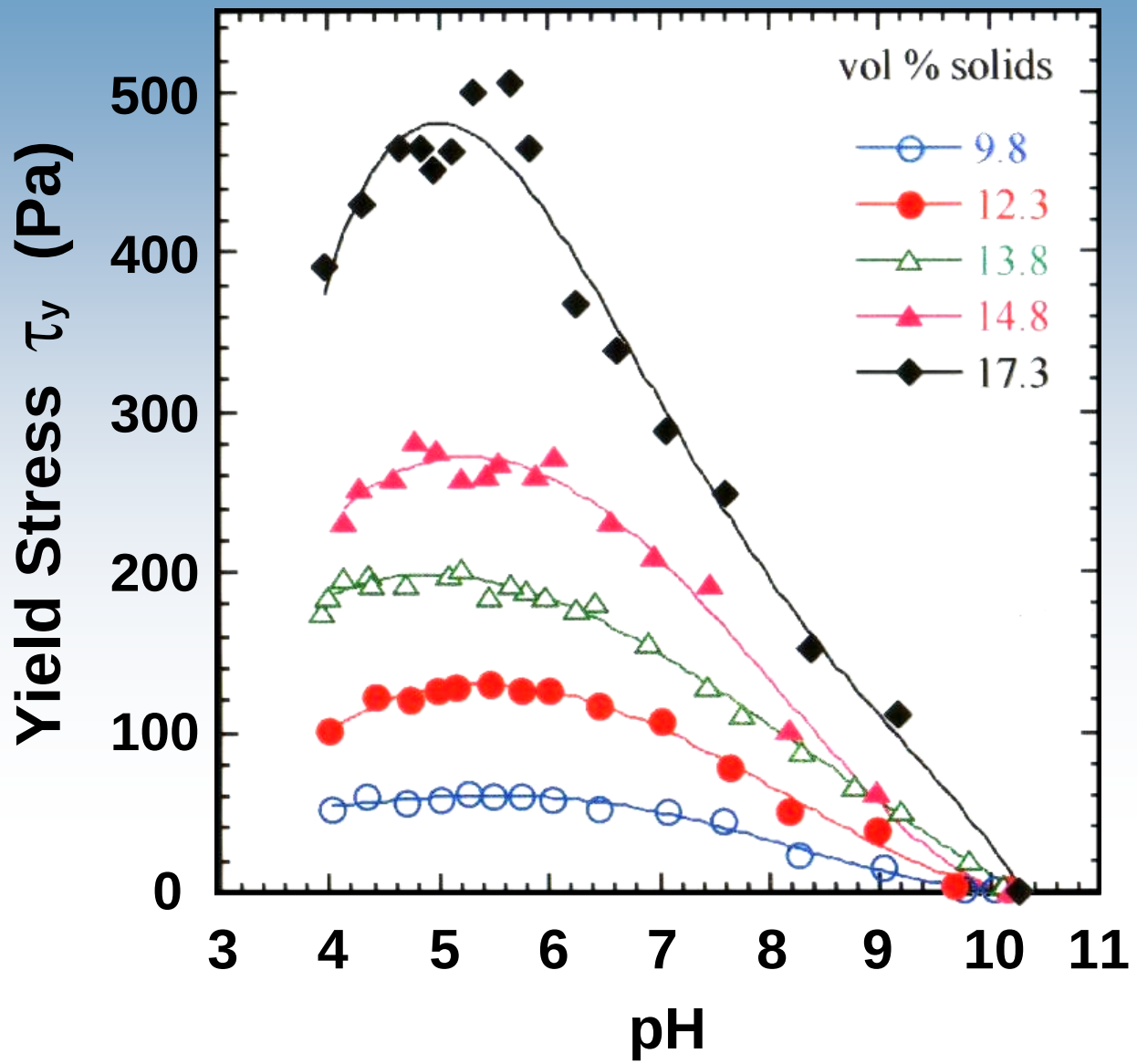
The Normalised Yield Stress as a Function of ζ^2 for Alumina Suspensions of Different Particle Sizes



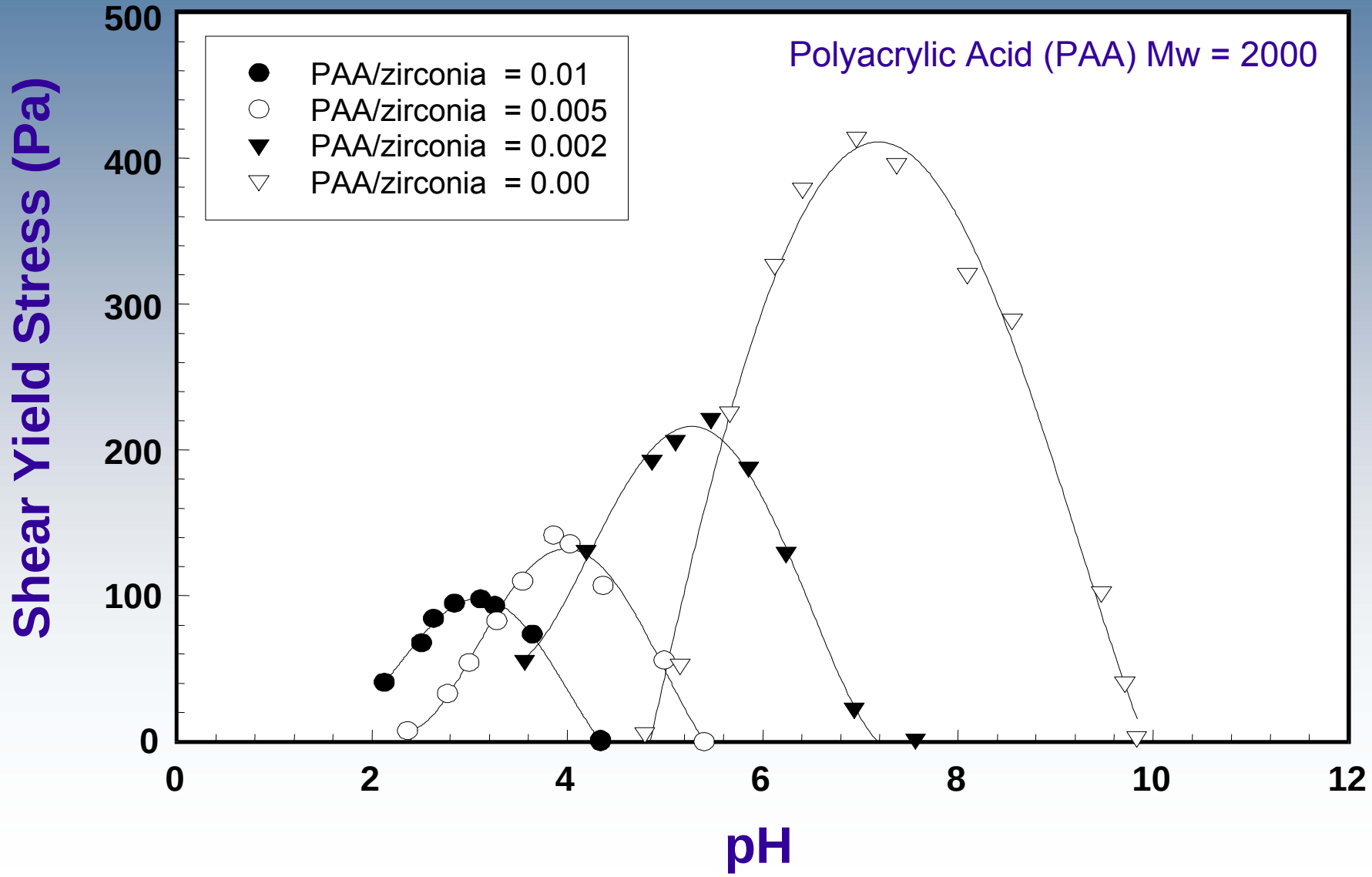
Normalised Yield Stress of Alumina Suspensions as a Function of ζ^2 for AKP-20.



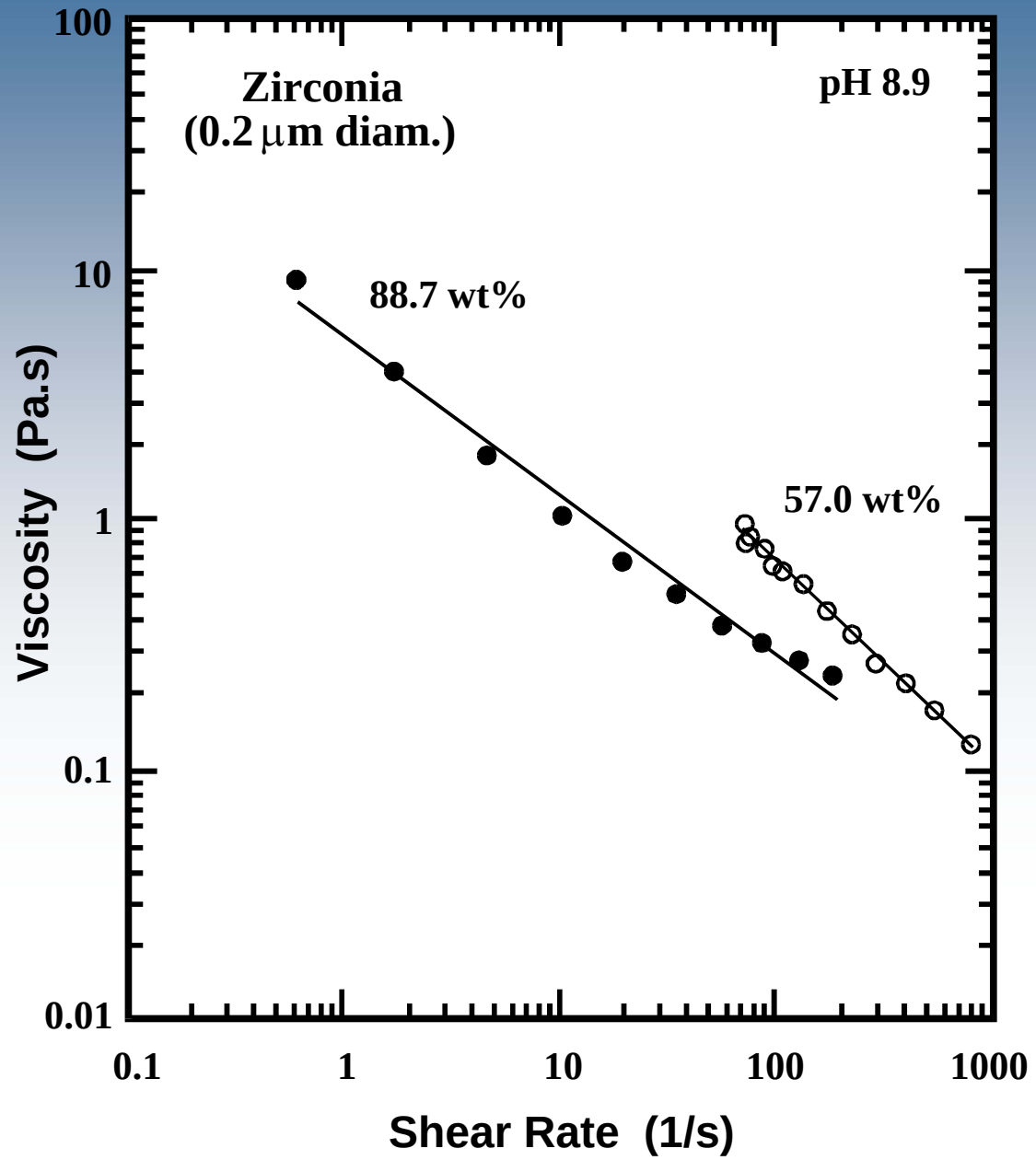
Kaolin Rheology



Effect of Low Molecular Weight Polyacrylic Acid on the Shear Yield Stress - pH behaviour for a Zirconia Suspension at 57wt%

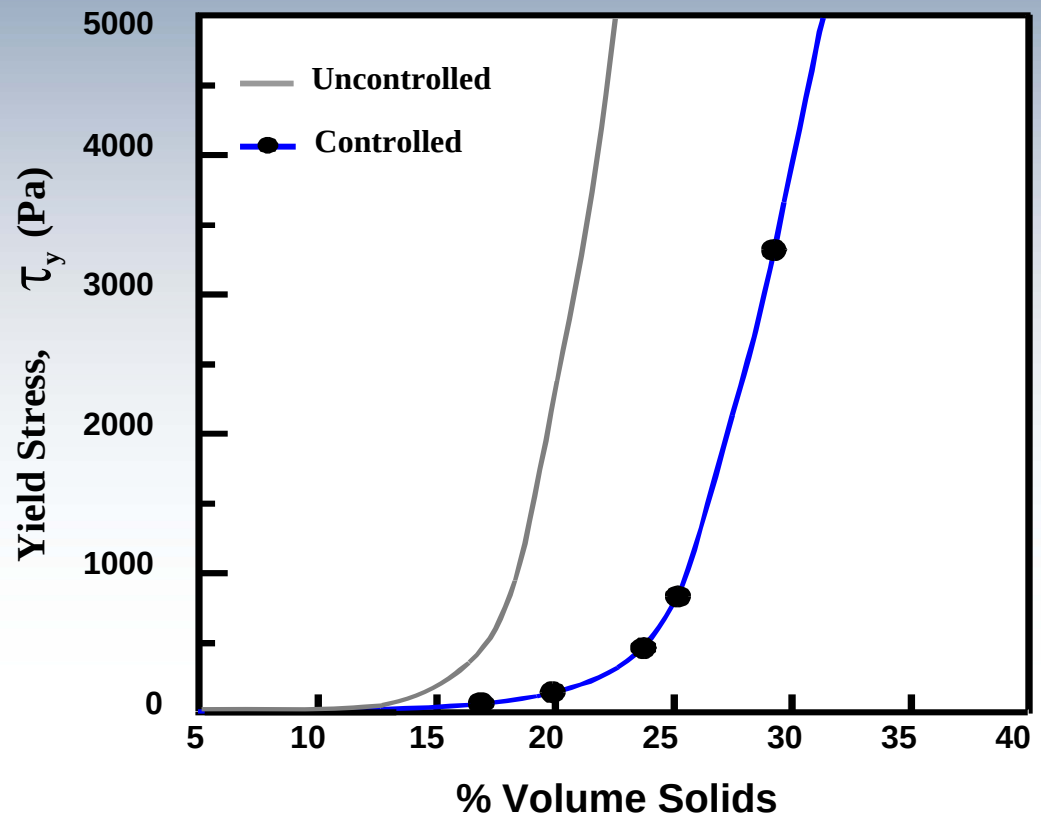


Manipulating the Flow Characteristics of Particulate Fluids

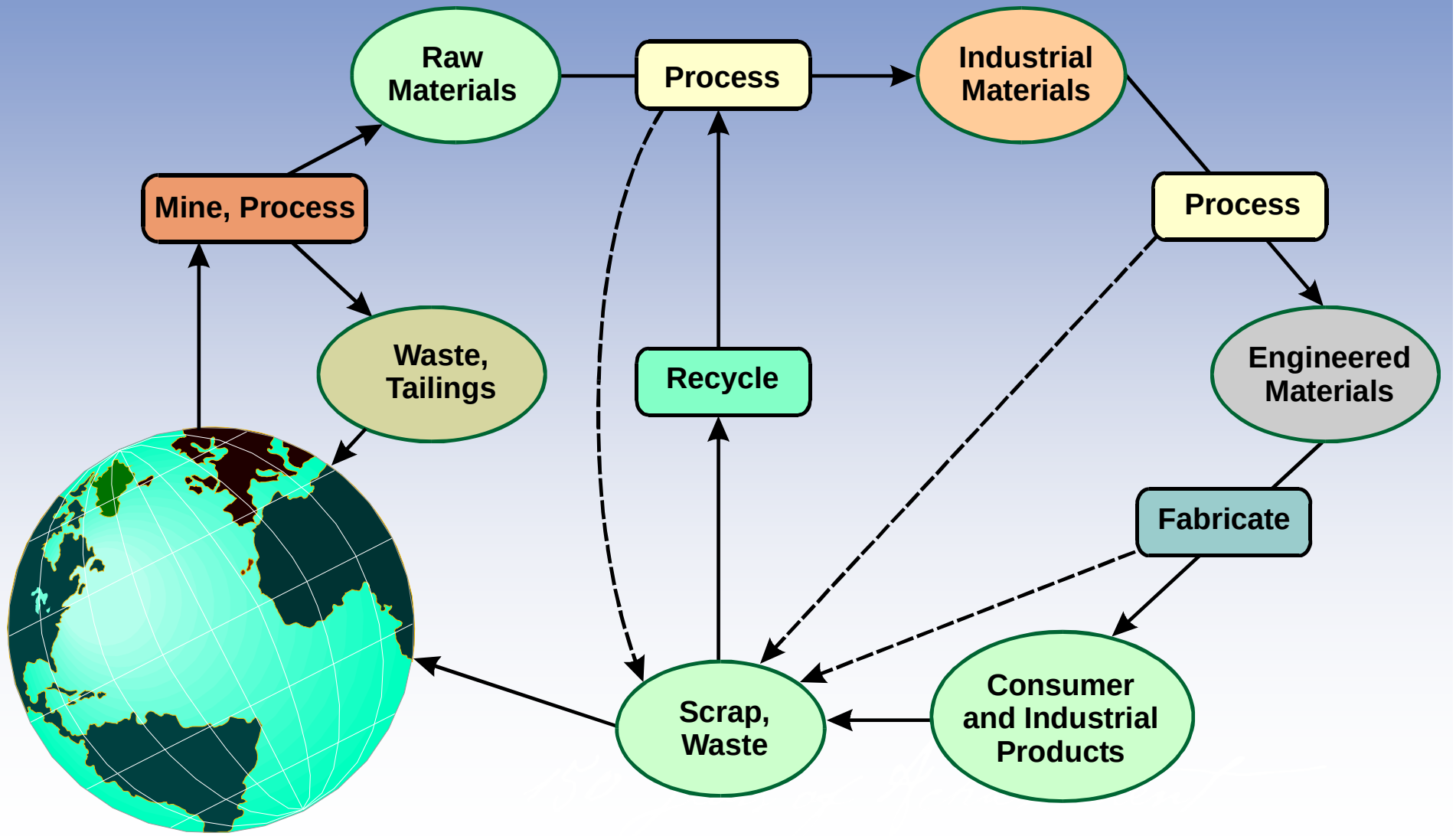


20g clay in 60g $H_2 0$	**Uncontrolled Dispersion**	**Controlled Dispersion**
25% solids	**25% solids in 1 M $CaCl_2$**	**25% solids in 1 M $CaCl_2$**

Effect on Yield Stress vs Volume Fraction Behaviour for Clay Dispersed in $CaCl_2$ *and* Clay Dispersed in Water with $CaCl_2$ added later.



Sustainability, Recycling and Environmental Modelling



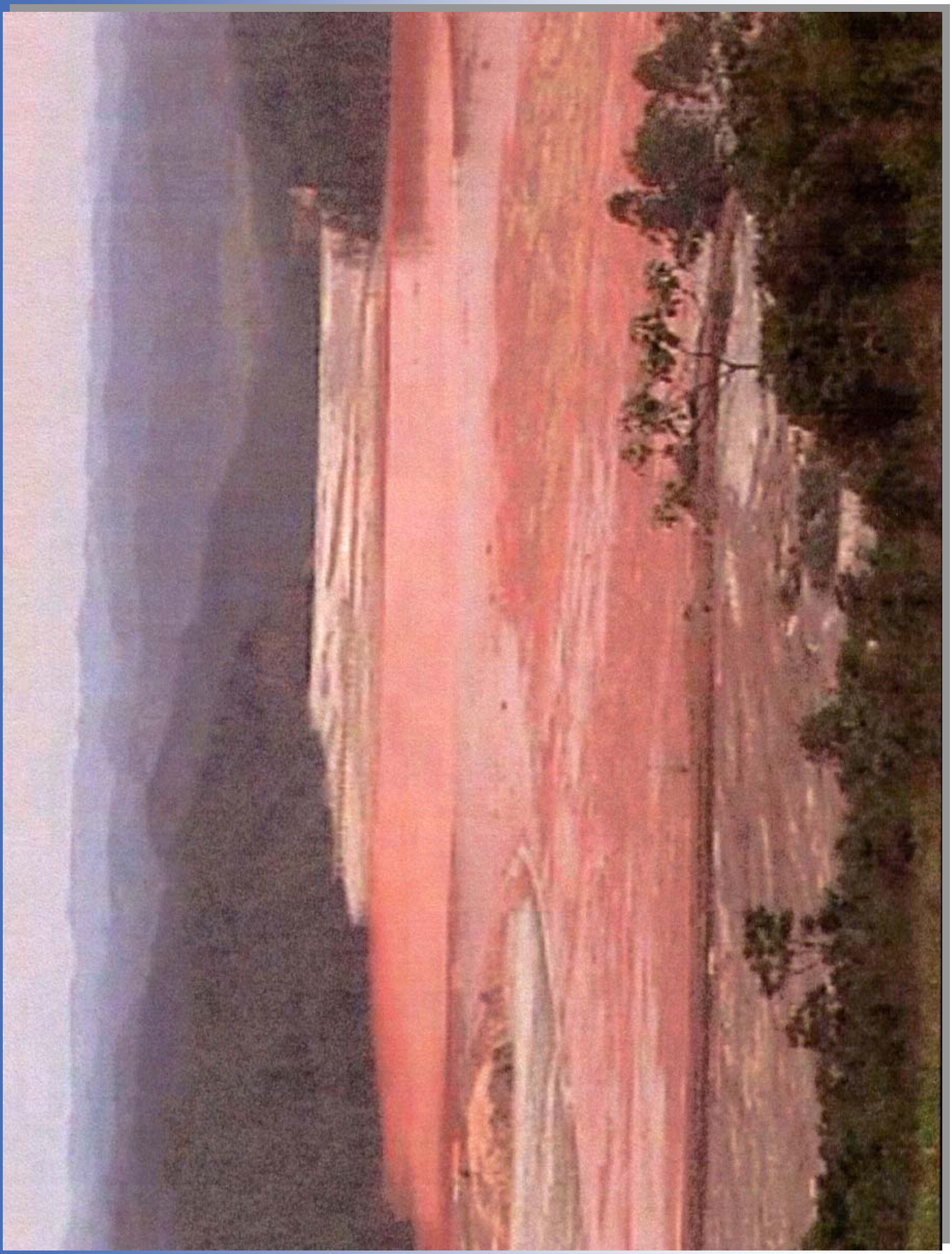


Photo by Stuart Kirsch, 1996.

This forest was killed by dieback after being smothered with mine waste.
It is on the lower Ok Tedi River near Dome Village.

*I*n-pit Fill

Environmental Policy and Tailings Disposal

Bob Watts

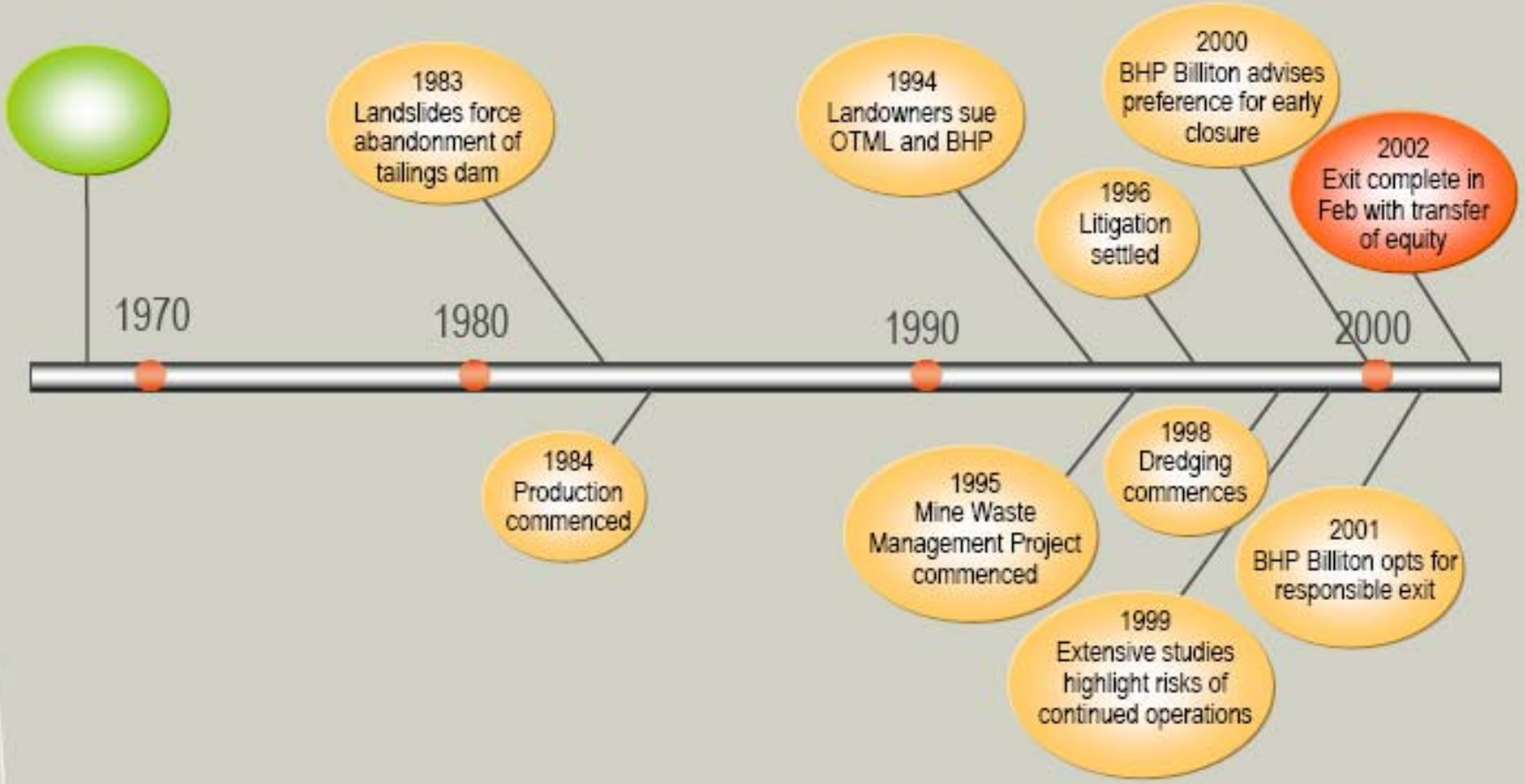
Chief Scientist and Vice President Technology

BHP Billiton

bhpbilliton

Main Menu

Ok Tedi Copper Mine in PNG



bhpbilliton

Photo by Stuart Kirsch, 1996.

This forest was killed by dieback after being smothered with mine waste.
It is on the lower Ok Tedi River near Dome Village.

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OK TEDI UPDATE

- Record profit – US$318.8 million – a 23% increase on previous year

- "…we are concerned that OTML's environmental monitoring indicated that the environmental impact of the mine may prove to be greater than previously understood."

  (Managing Director, Keith Faulkner)

- "In February 2005, areas of acid rock drainage were appearing on the levees of the Fly River….."

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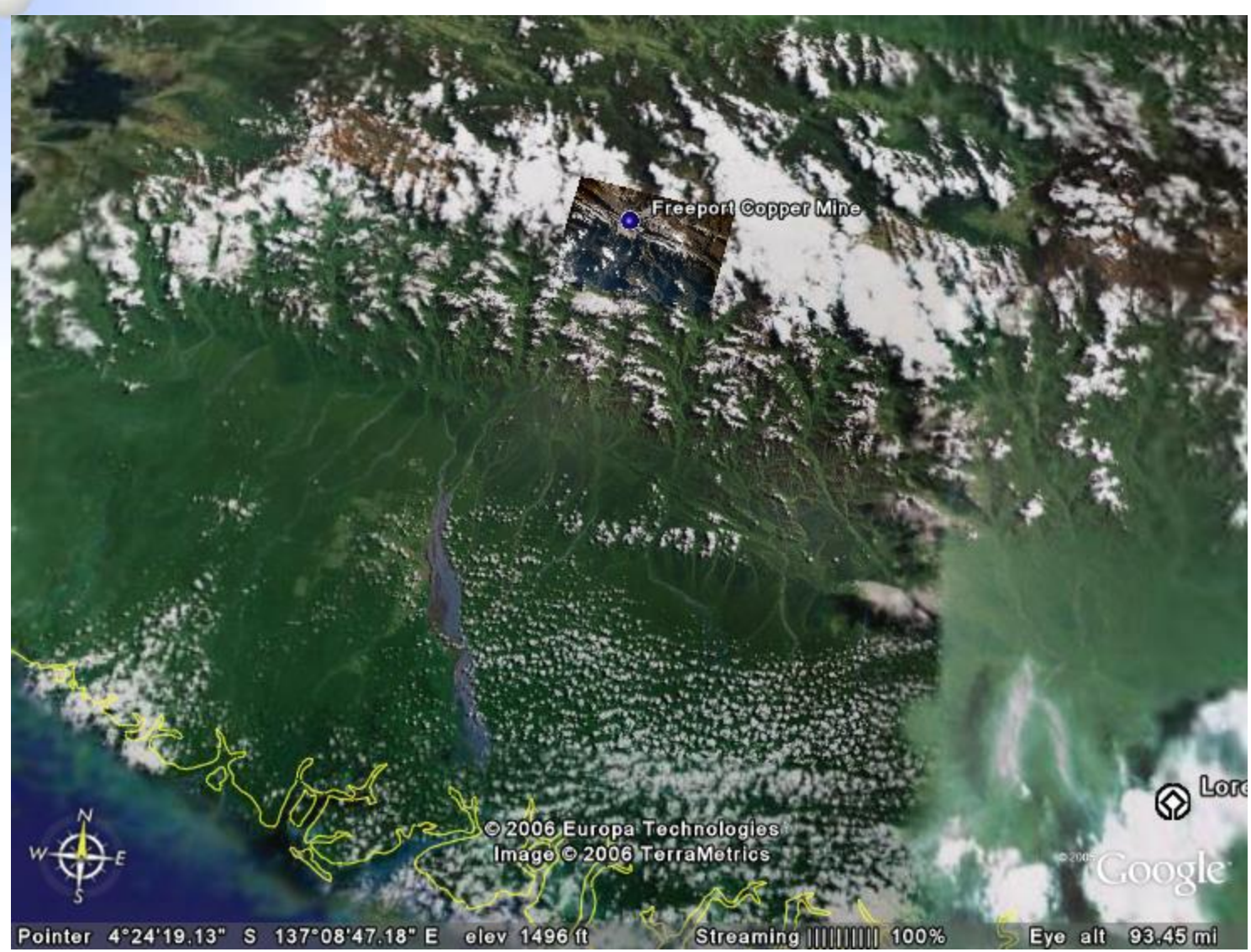

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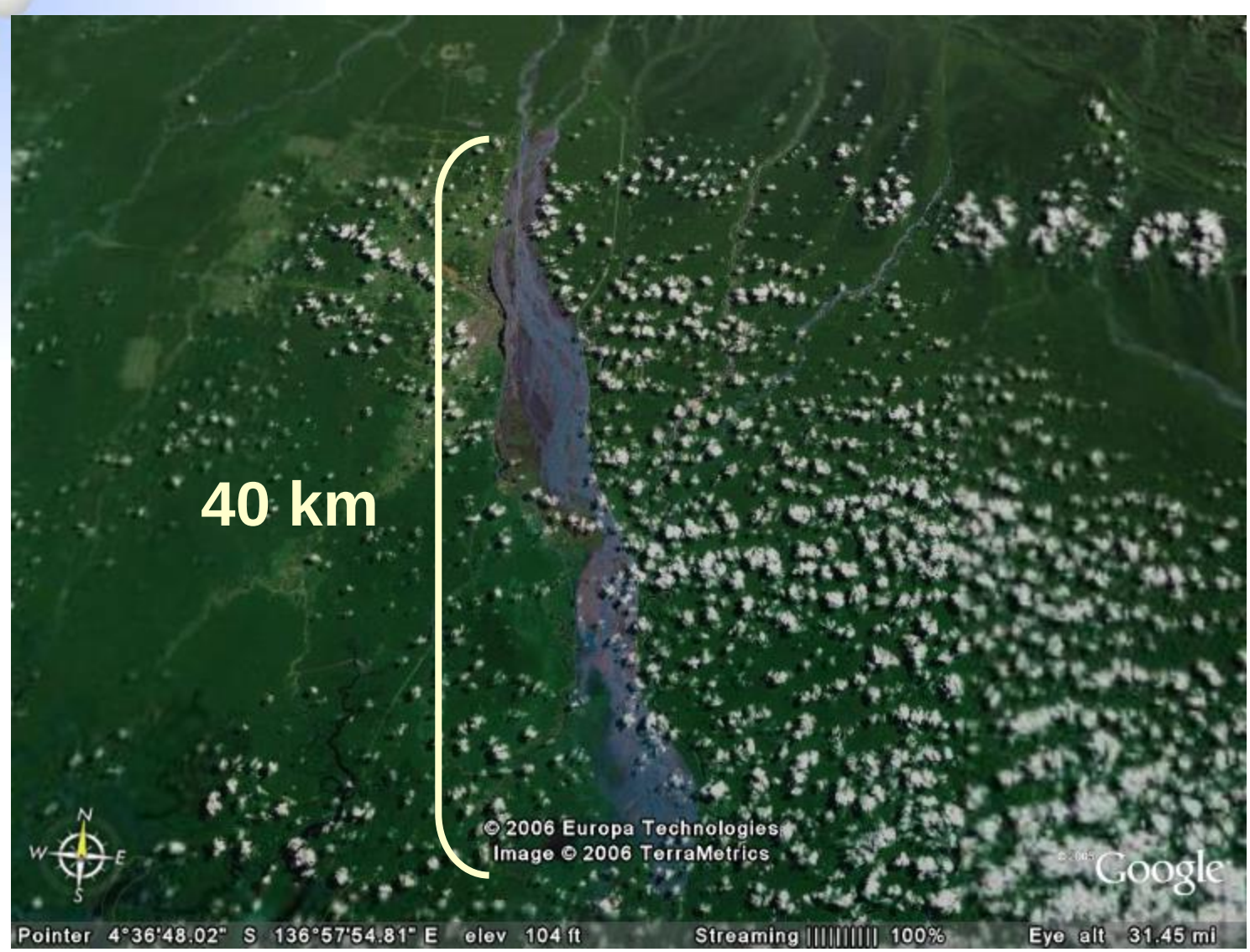

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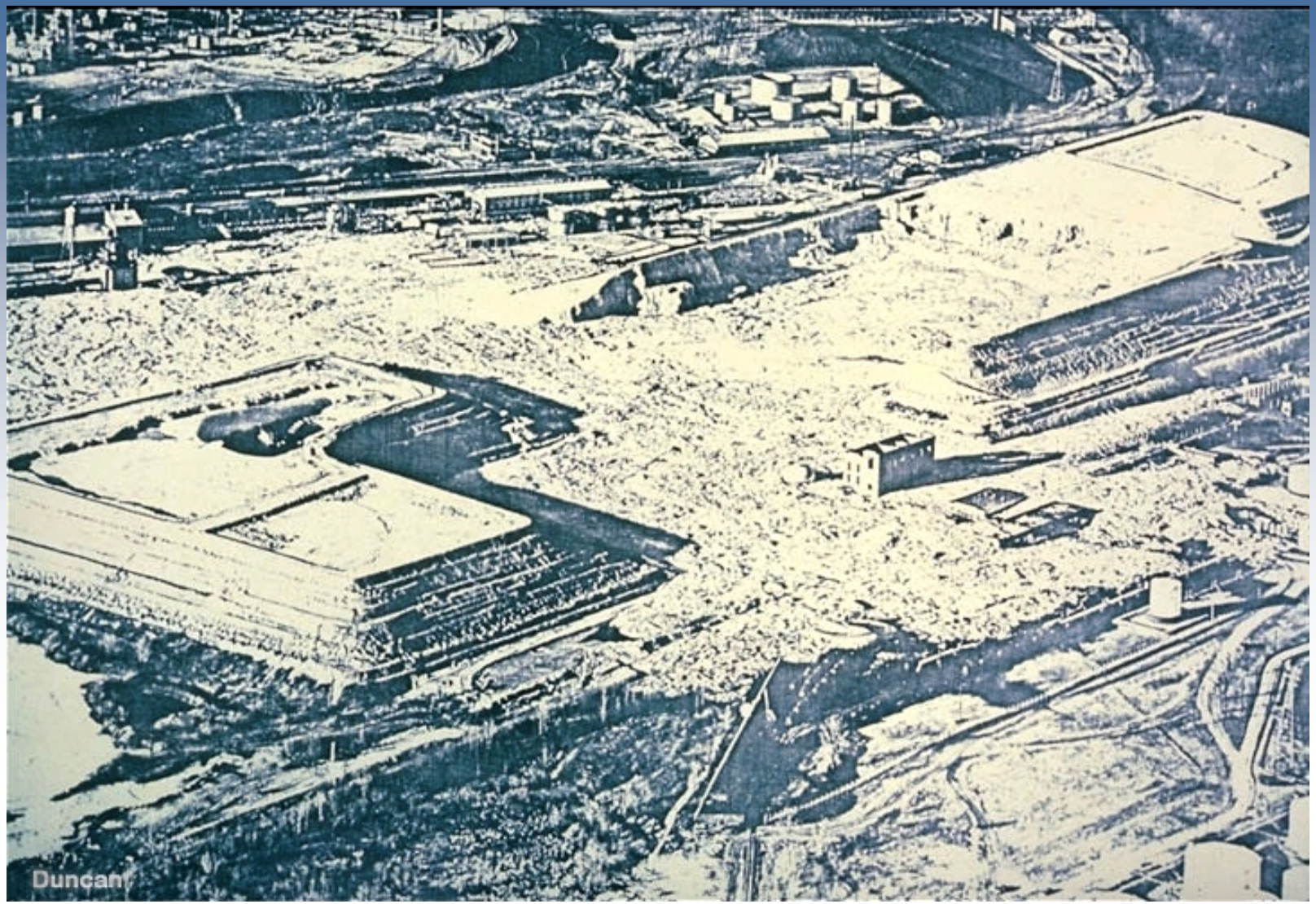
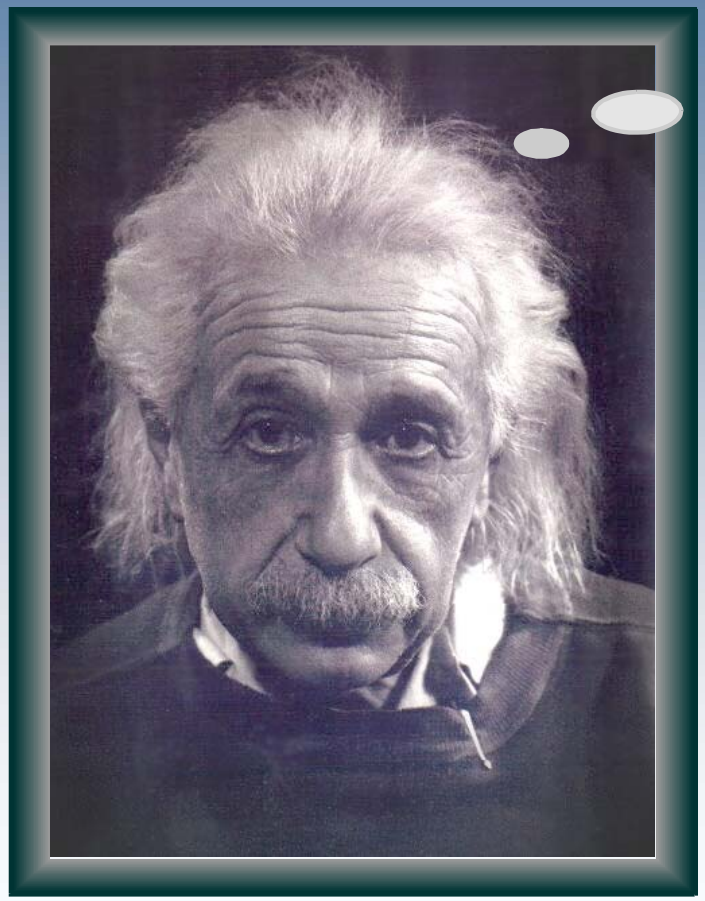
Failure of dam holding platinum tailings in South Africa



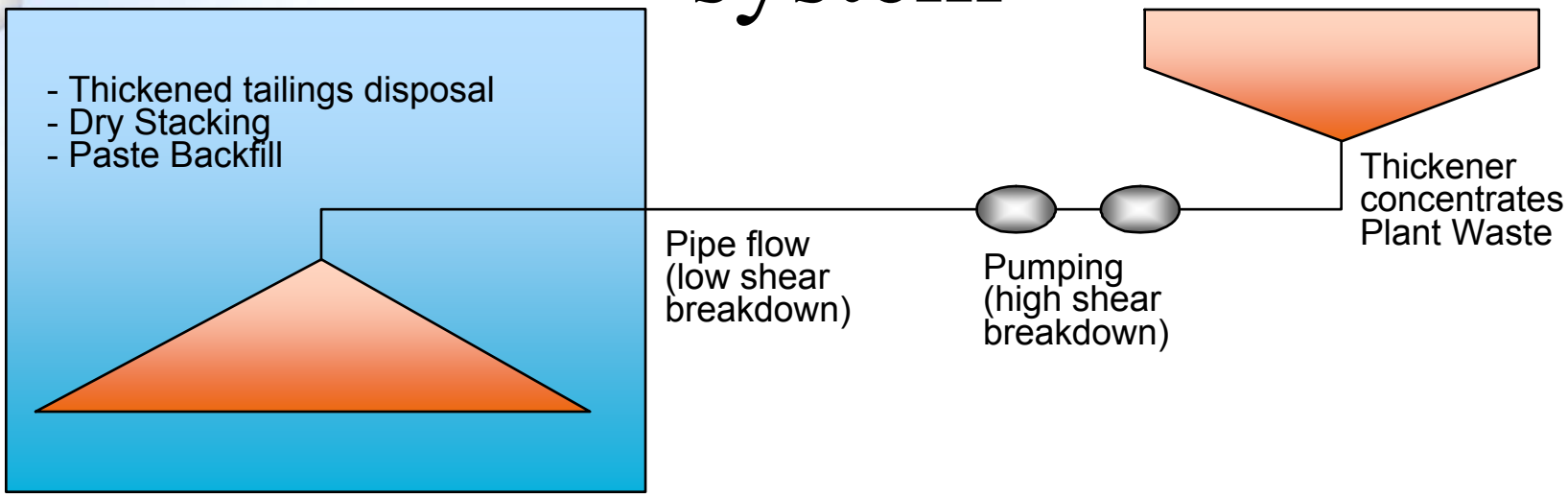
Failure of a carbide tailings pond near Louisville, Kentucky.

Failure of dam retaining gypsum tailings in Florida



A problem cannot be solved with the same kind of thinking that created it...

Suggested approach for determination of tailings disposal system



DESIGN SEQUENCE

1. Choice of disposal method, Depositional requirements.
2. Rheology requirements for pipeline transport.
3. Thickener design.

New Developments

- **Compression Rheology**
  - **Compressive Yield Stress**
  - **Permeability**

See: BSR Annual Rheology Review 2002: Compressive Rheology: An Overview, Ross G. de Kretser, David V. Boger and Peter J. Scales

Rheology – Changing thinking in the minerals industry

D V Boger
Laureate Professor
The University of Melbourne
Victoria, Australia

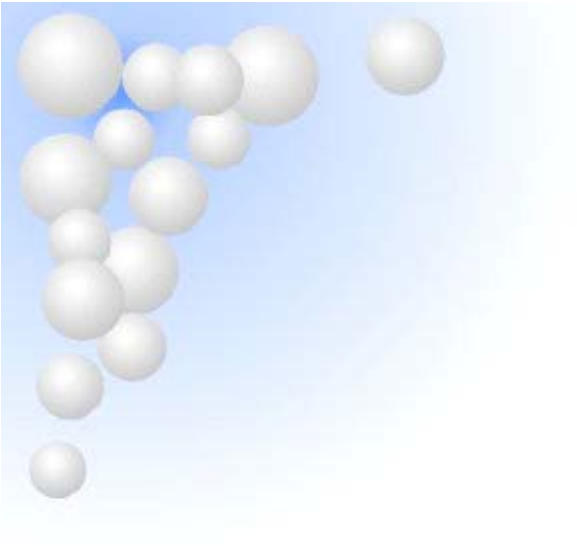
Paste and Thickened Tailings Seminars

- Edmonton, Canada, November 1999 hosted by the University of Alberta
- Perth, Australia, April 2000 hosted by the Australian Centre for Geomechanics
- Edmonton, Canada, October 2000 hosted by the University of Alberta
- Pilanesberg, South Africa, May 2001 hosted by the University of the Witwatersrand
- Santiago, Chile, April 2002 hosted by the Instituto de Ingenieros de Minas de Chile and Gecamin Ltda
- Melbourne, Australia, May 2003 hosted by the Australian Centre for Geomechanics and the University of Melbourne
- Cape Town, South Africa, March 2004 hosted by the University of the Witwatersrand and Paterson and Cooke Consulting Engineers Pty Ltd
- Santiago, Chile, April 2005 hosted by the University of Chile
- Limerick, Ireland, April 2006 hosted by Leeds University, Dorr-Oliver EIMCO and Golder Associates (UK) Ltd
- Perth, Australia, March 2007 hosted by the Australian Centre for Geomechanics
- **Rheology discovered!**

Wolf Robe (June 1909)

"Only when the last tree has died
and the last river has been poisoned
and the last fish been caught
will we realise
we cannot eat money."

Cree Indian saying



PFPC

ARC
AUSTRALIAN RESEARCH COUNCIL

THE UNIVERSITY OF
MELBOURNE

Sustainable Development is the Simultaneous Striving for Economic Prosperity, Environmental Health and Social Well-being.

"Environmental considerations dictate that we must manipulate tailings to fit a particular environment rather than manipulating the environment to contain the tailings."

Understanding and exploiting the rheology of tailings helps us do this.

The Regulatory Trend

"It is interesting to observe that many times more technical effort is devoted to ground water and toxicological studies for abandoned deposits than was ever allocated for their original design and generation.

Unfortunately much of the cost has been borne by government; should we then be surprised that responsible government is therefore imposing much greater regulatory security?"

S.G. Virk, 1990

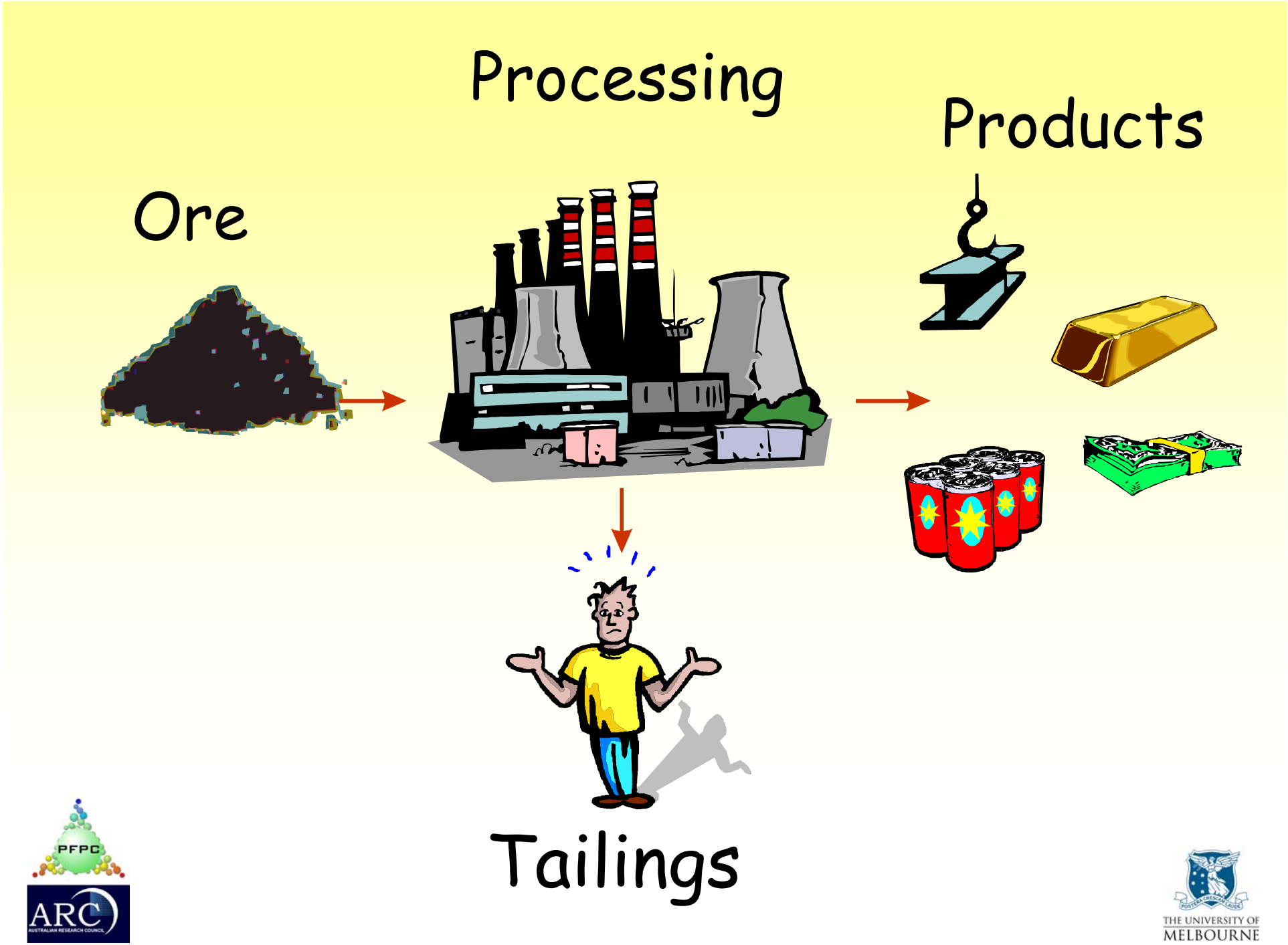
Rheological Consulting Services

Rheological Consulting Services Pty. Ltd.

- Australia
- Canada
- Chile
- Jamaica
- New Guinea
- South Africa
- USA
- New Caledonia
- Indonesia

The evidence is getting stronger that what is good for sustainable development can also be good for business, in sometimes surprising ways.

Business, society and the environment can "succeed" simultaneously.


Processing
Products
Ore
Tailings

Slurry Transportation
An Overview

Robert Cooke

PhD, Member of the Society of Mining, Metallurgy and Exploration
Fellow of the Southern African Institute of Mining and Metallurgy

Paterson & Cooke, Denver Colorado

RobertC@PatersonCooke.com, Tel 303 867 2264
1580 Lincoln Street, Suite 1000, Denver CO 80203

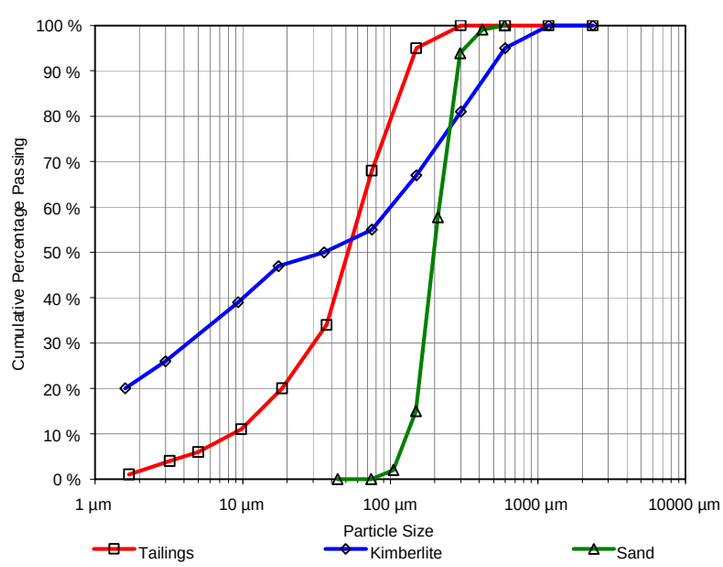
DOE Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop
January 14-18, 2008
Orlando, Florida

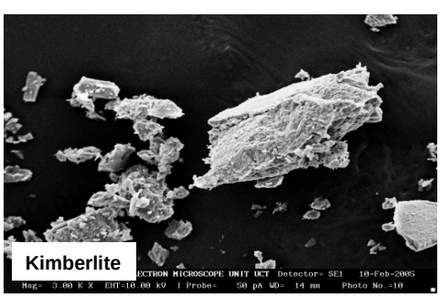
Presentation Outline

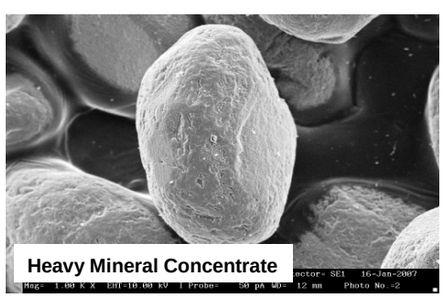
- **Slurry properties**
- **System applications**
- **Design considerations for yield stress slurries**
- **Engineering slurry systems**

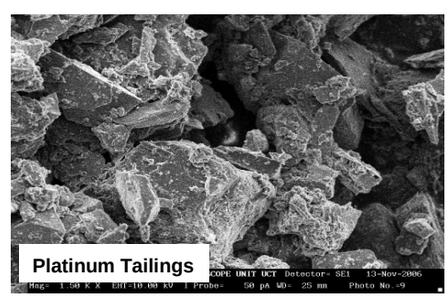
Slurry Properties

PATERSON & COOKE









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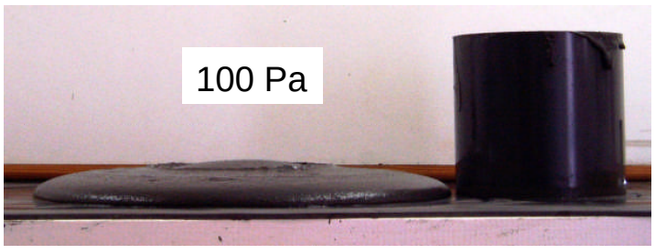
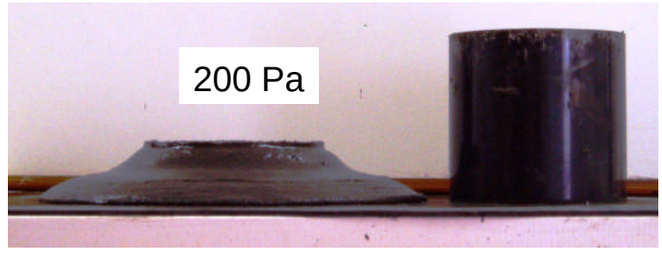
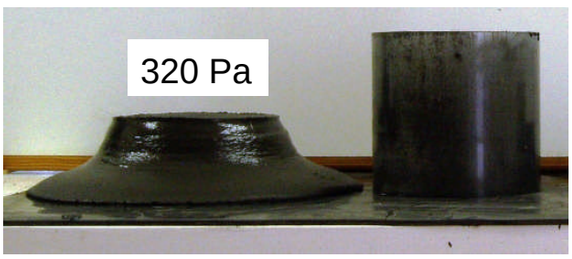
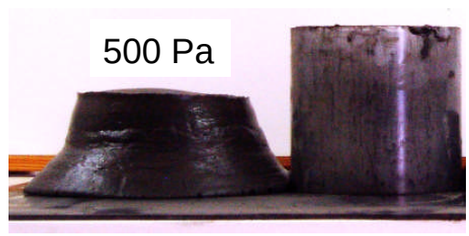
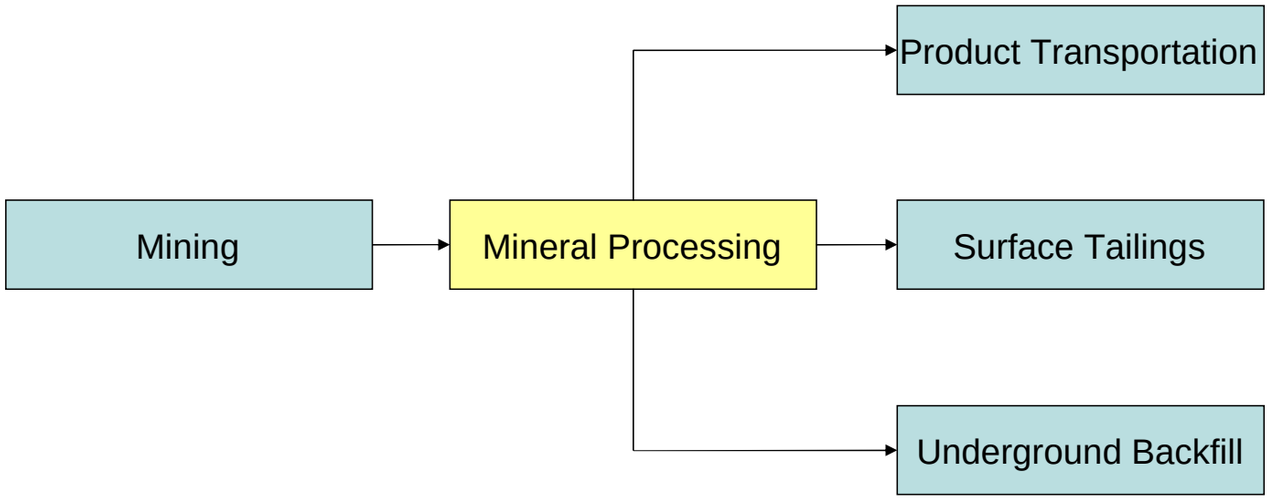

100 Pa


200 Pa


320 Pa


500 Pa

System Applications: Mineral Industry



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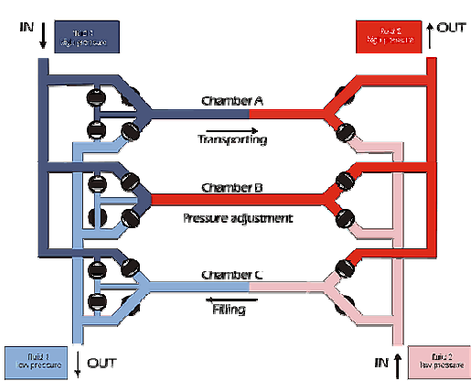
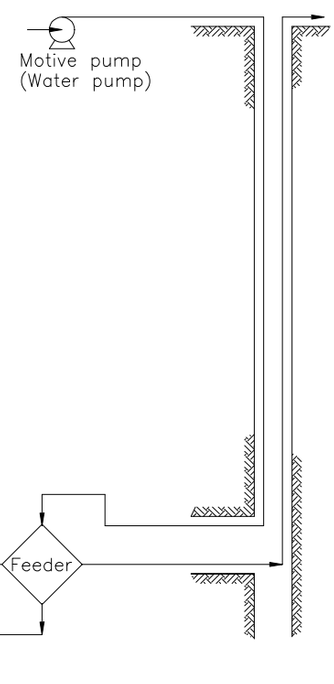
Hydraulic Ore Hoisting





2” rocks
250 t/h
3300 feet vertical
3625 PSI (250 bar)

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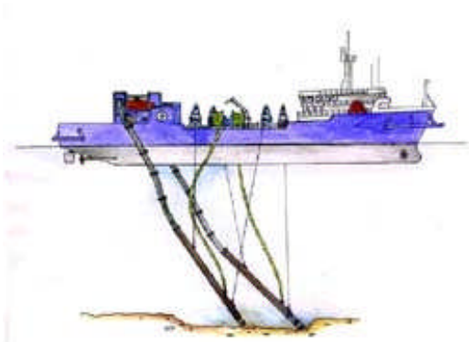
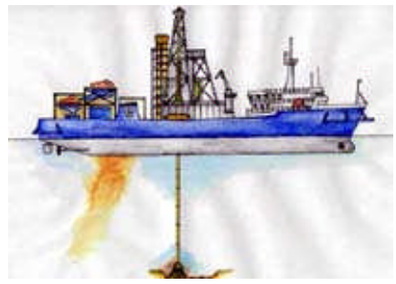
Marine Mining





16” rocks
1000 t/h
600 feet vertical
53 000 GPM

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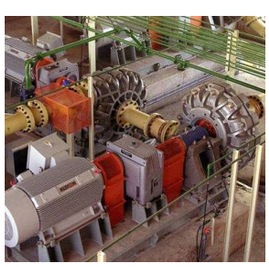
Mineral Processing



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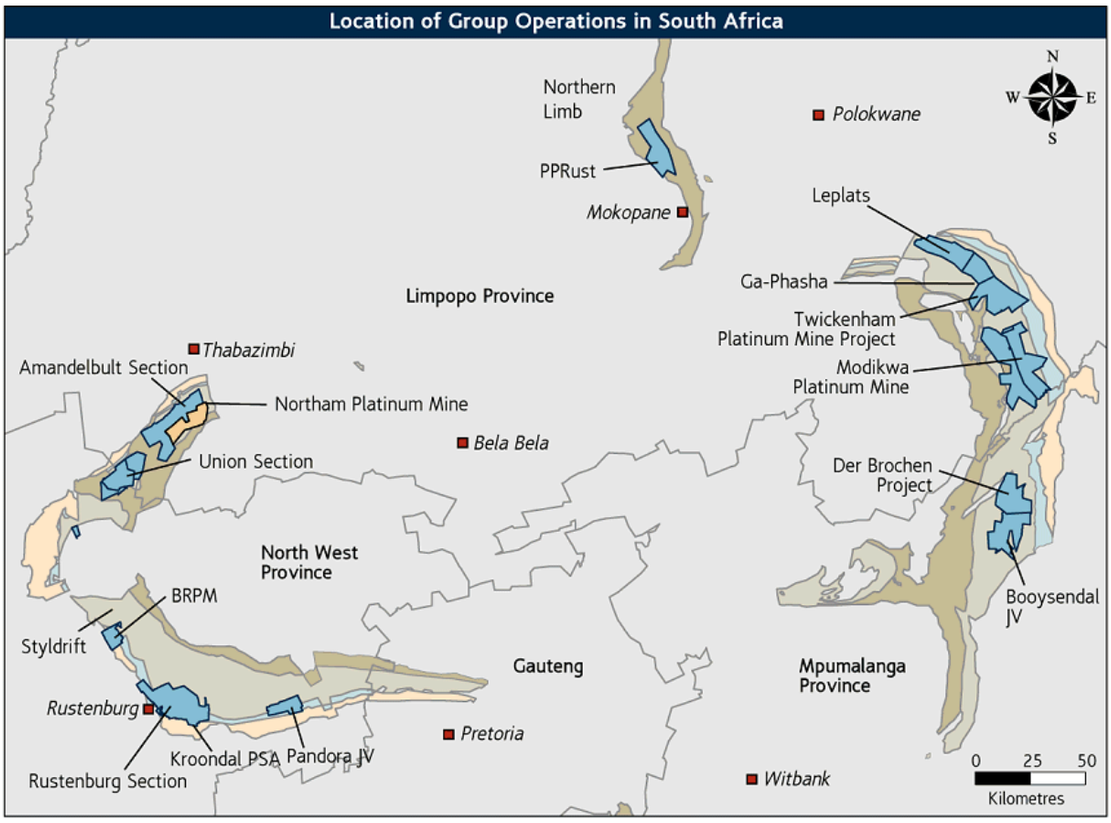
Mineral Processing



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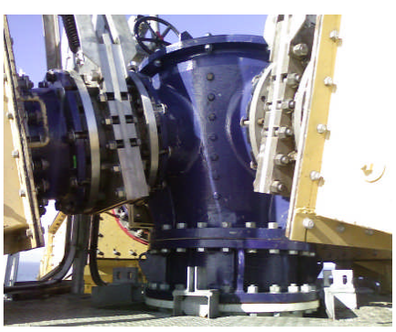
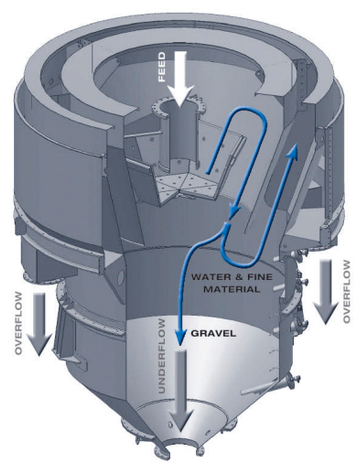
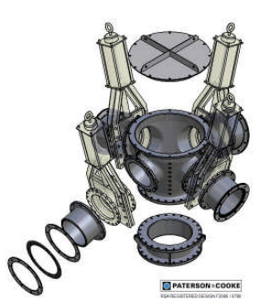
Mineral Processing







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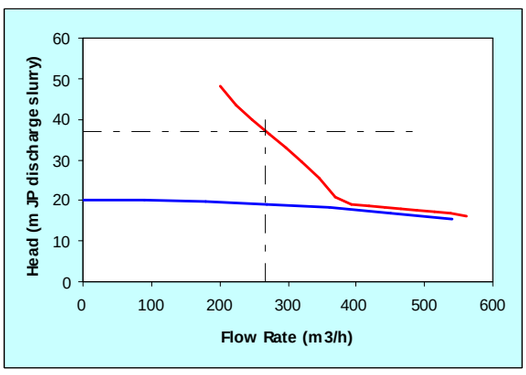
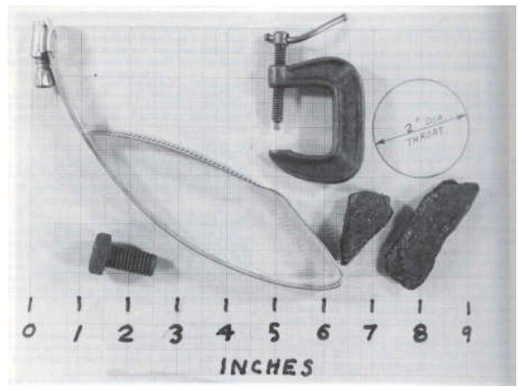
Mineral Processing





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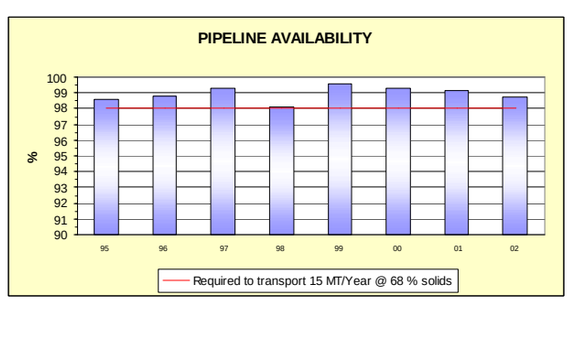
Concentrate Transport



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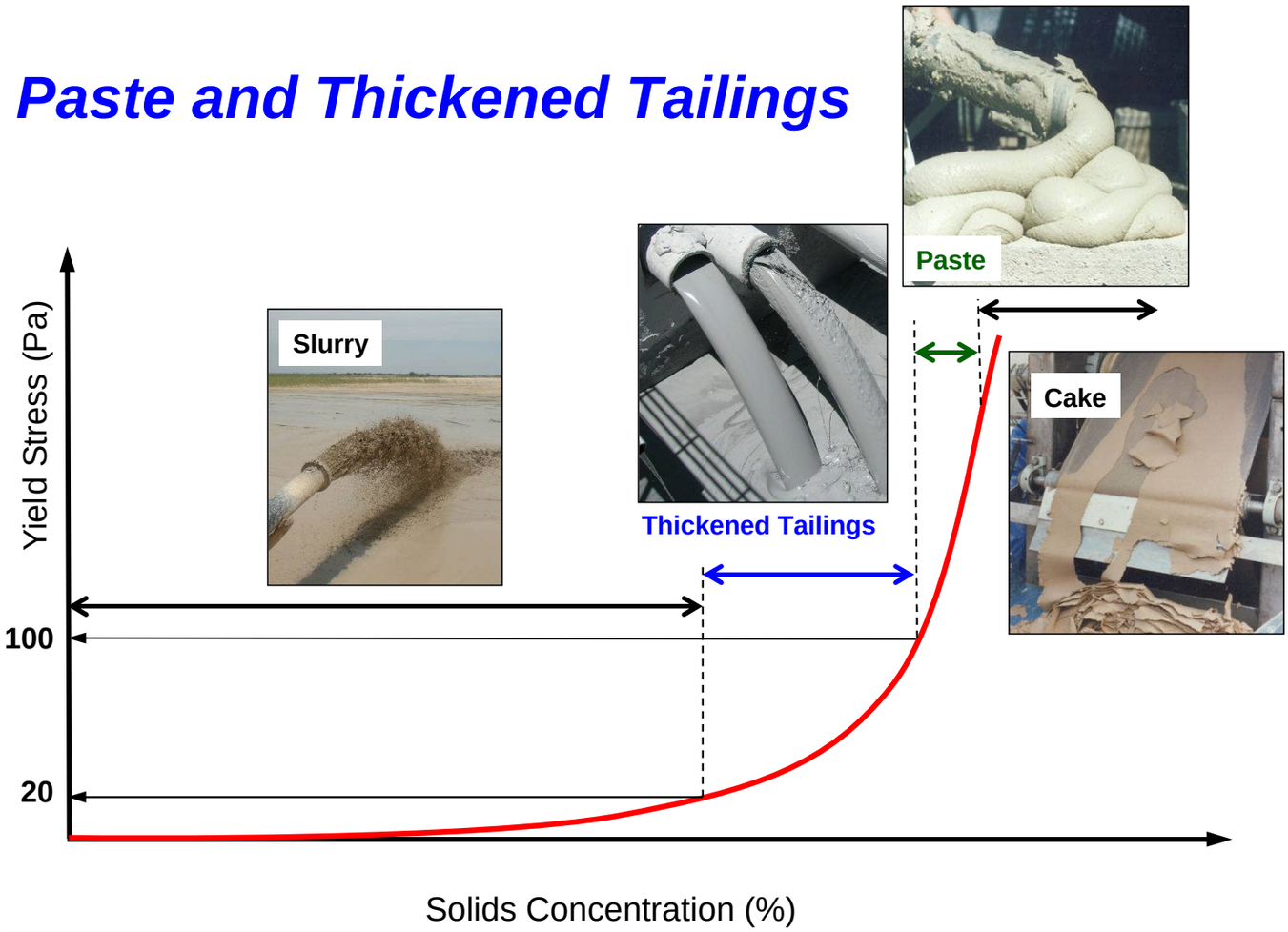
Paste and Thickened Tailings



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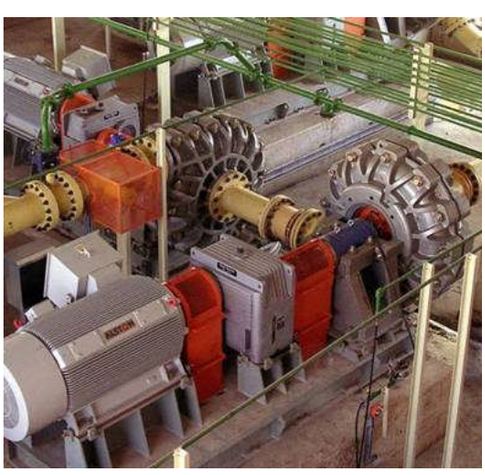
Surface Tailings



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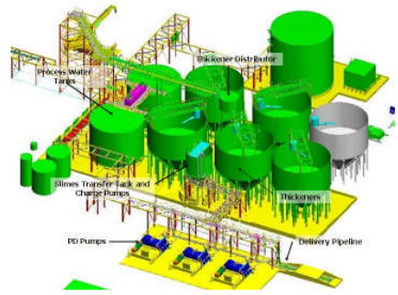
Surface Tailings



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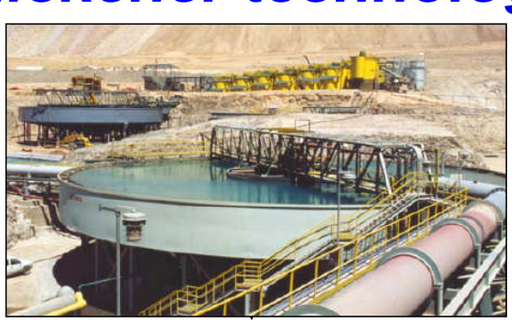
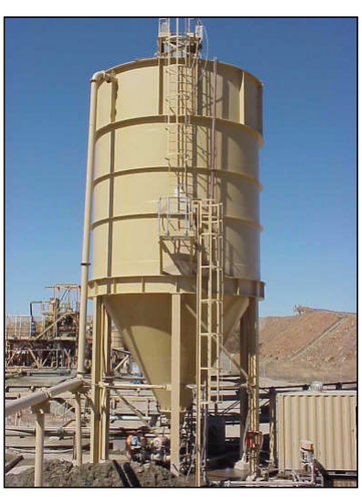
Thickener technology

Conventional



High Rate



Ultra High Rate

Paste

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Underground Backfill

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Yield Stress Slurries

- **Operating velocity**
  - Laminar or turbulent flow
- **Laminar flow operation**
- **Residual pressure in pipeline**

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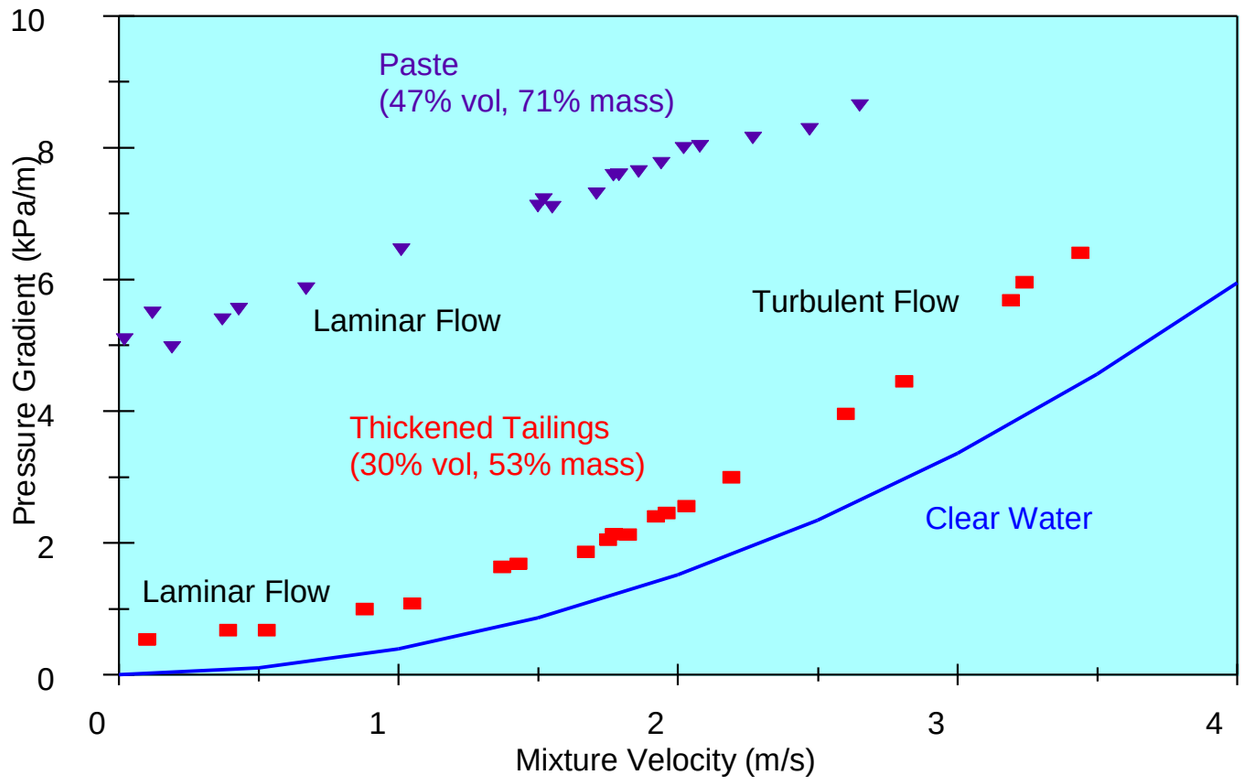

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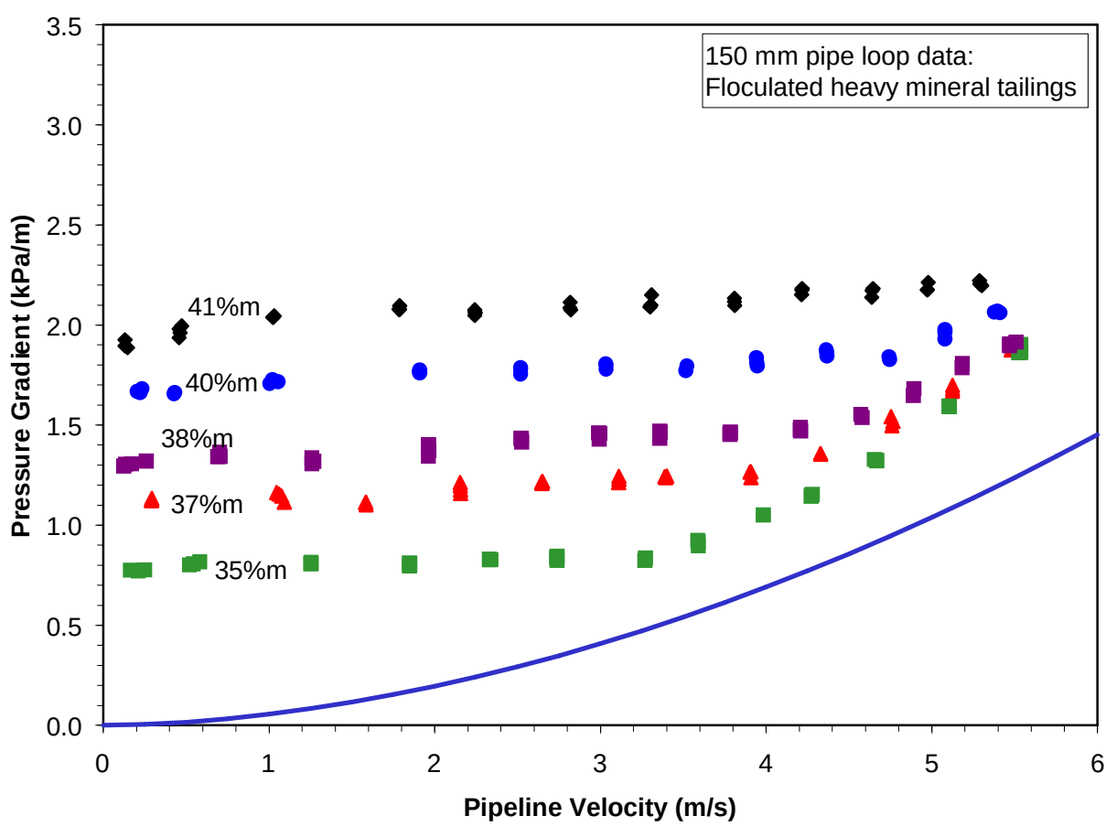

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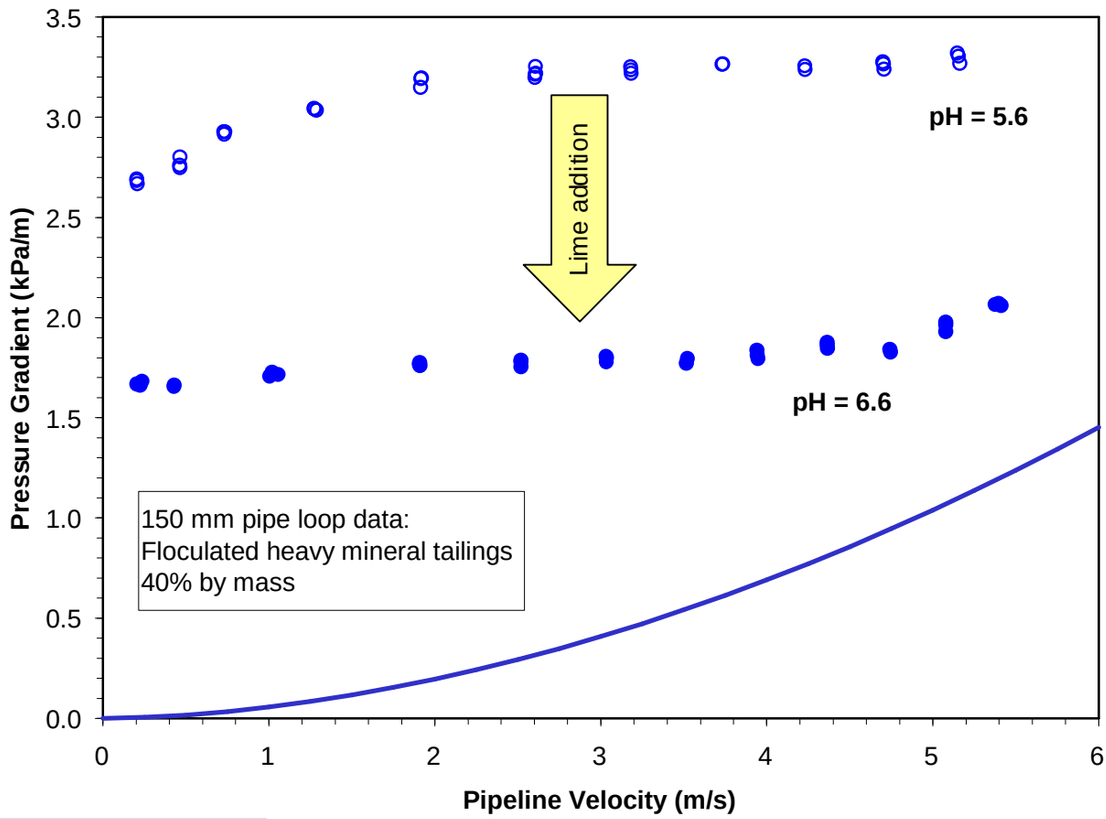
Design Considerations: Friction Loss



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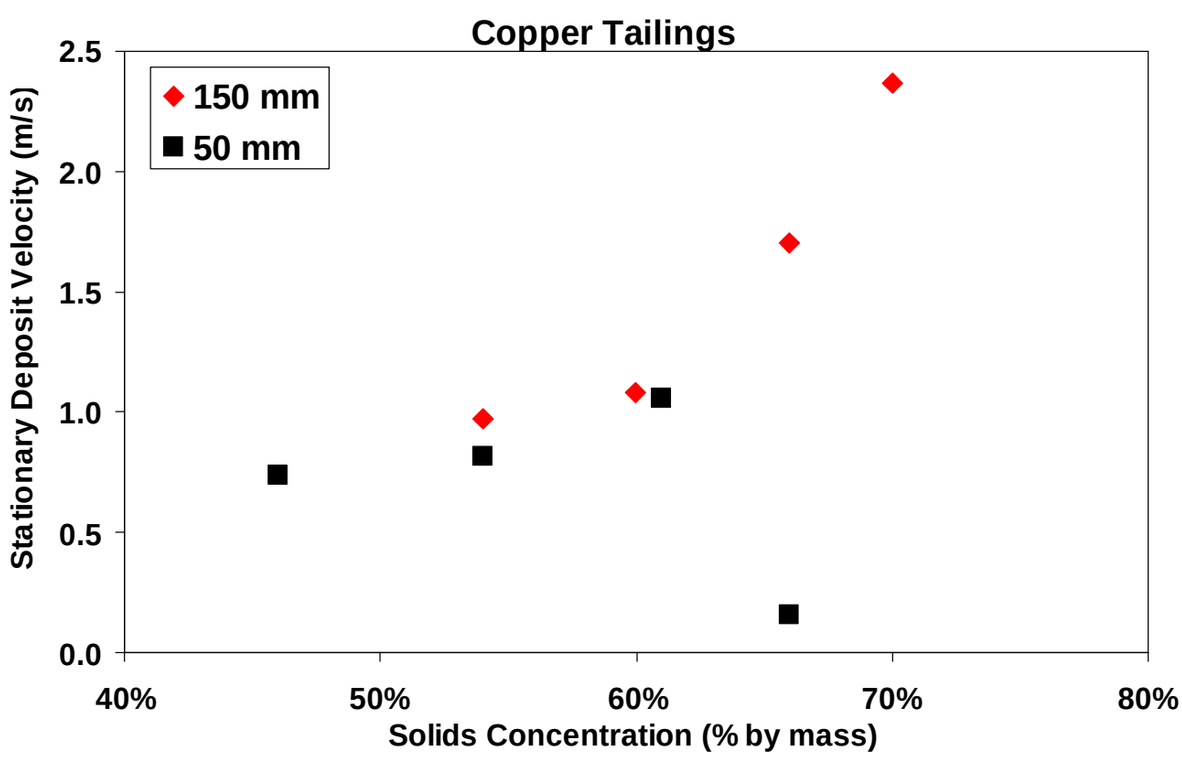

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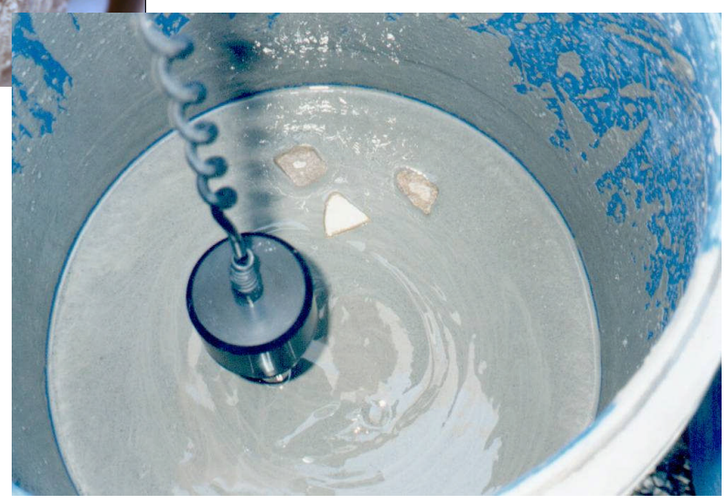

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Laminar Flow: Particle Settlement

- **Rugby Limestone pipeline**
  - Laminar flow operation
  - 92 km long, 250 mm diameter
  - Over 36 hours, 0.106 kPa/m to 0.121 kPa/m

Feed	Discharge
56.5%m	54.5%m
1.1% > 300 µm	trace
150 µm > 2.8% > 300 µm	trace

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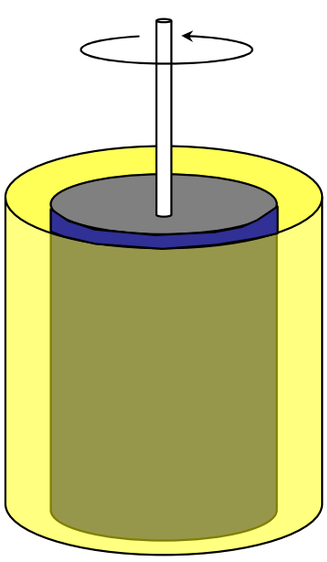
Laminar Flow: Particle Settlement

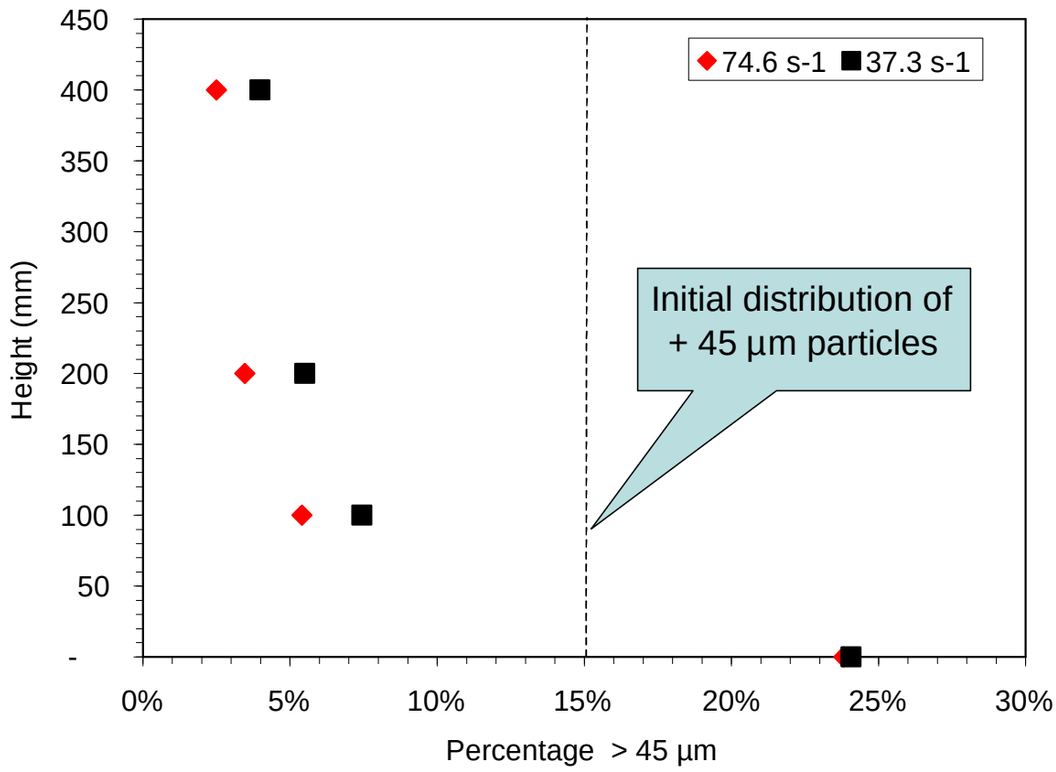
- **Belovo-Novosibirsk pipeline (Siberia)**
  - Laminar flow operation (stabilized coal)
  - 262 km (164 mile) long, 530 mm diameter
  - Pipeline blocked during commissioning
  - Pressure gradient increased with time
  - Stationary deposit on pipe invert
  - Instabilities were not observed during loop tests using a 200 mm pipeline

Laminar Flow: Particle Settlement

- **Will particles settle?**
  - Small-scale tests under sheared conditions



Laminar Flow: Particle Settlement



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Laminar Flow: Particle Settlement

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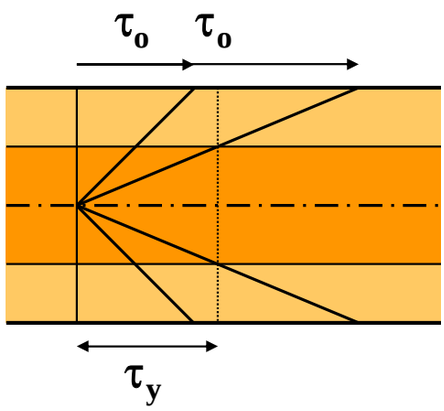
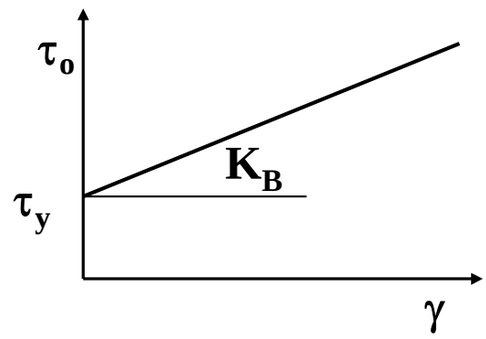
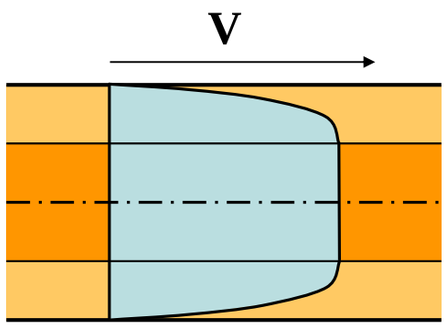
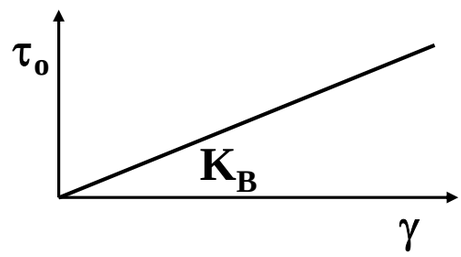
Laminar Flow: Particle Settlement









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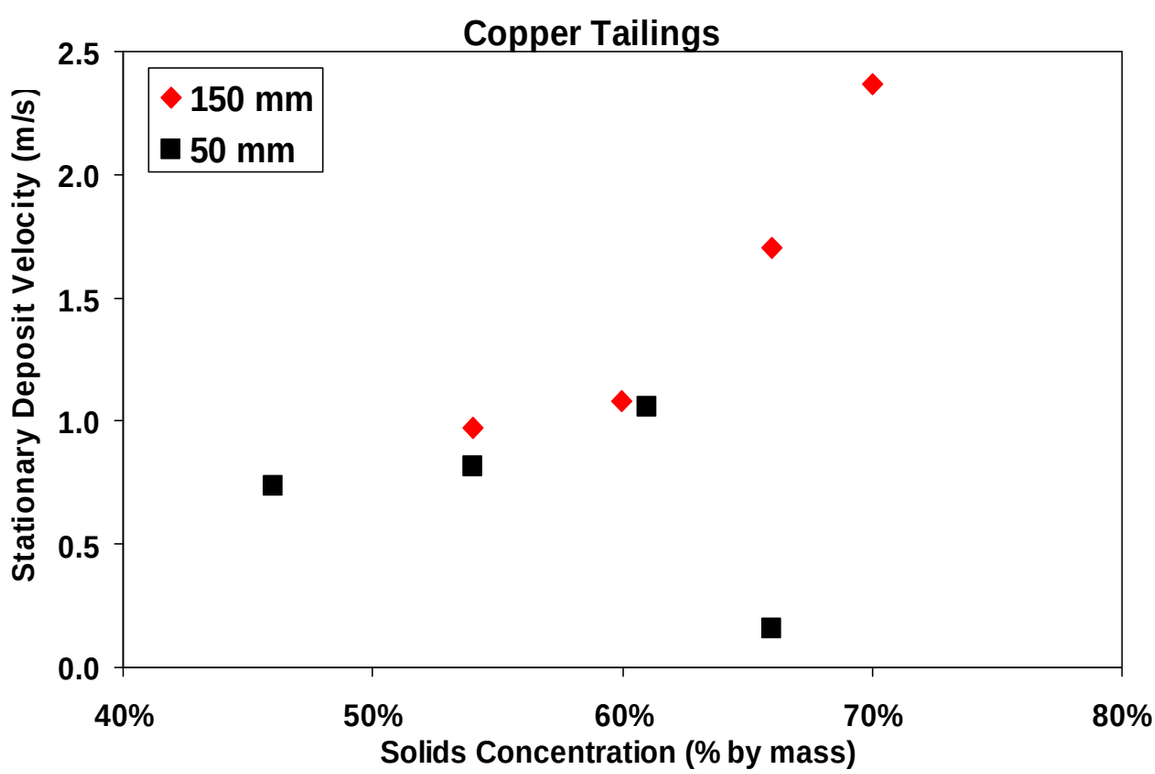
Laminar Flow: Particle Settlement



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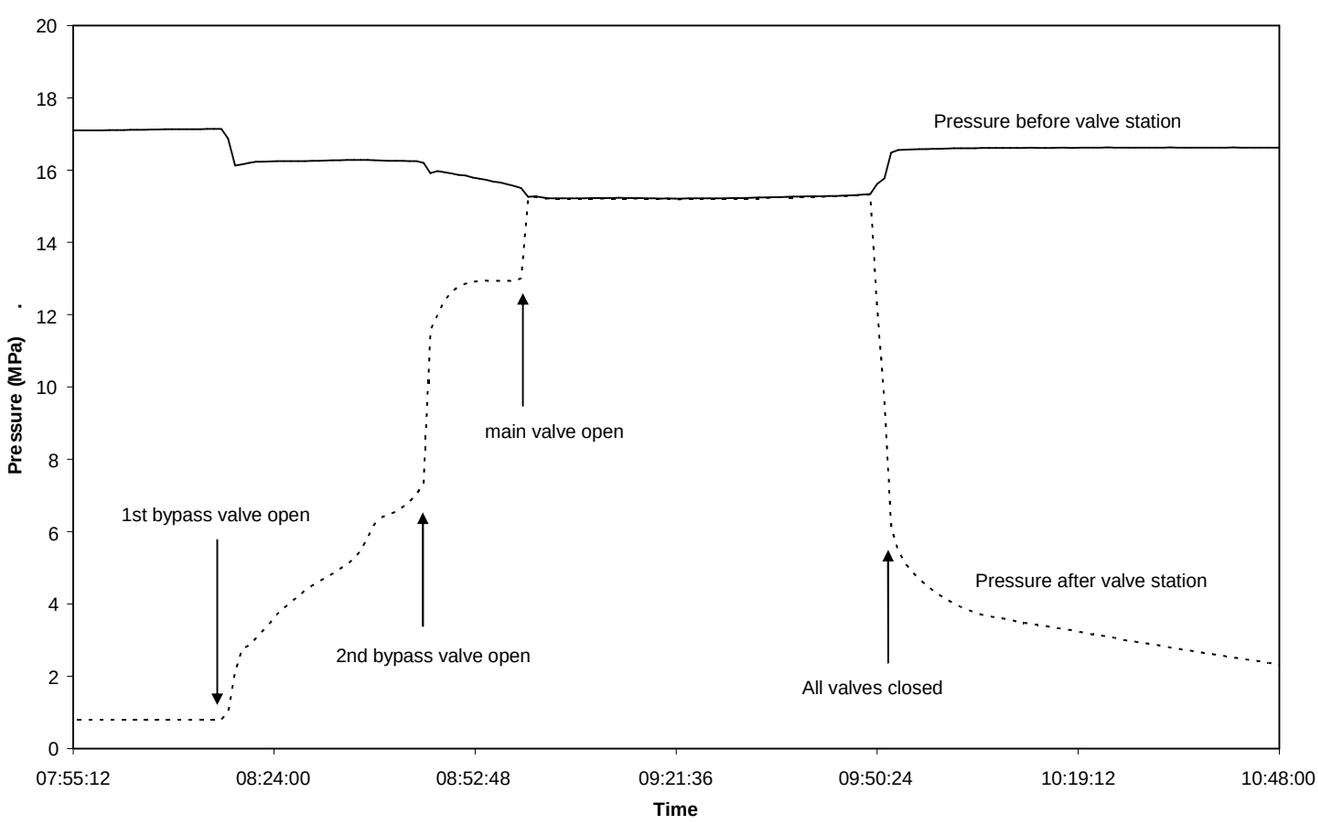
Laminar Flow: Particle Settlement

- **Under what conditions will the particles be transported?**
- **Thomas (1977)**
  - Deposition occurs at a constant pressure gradient regardless of pipe size.
- **Gillies *et al* (1999)**
  - Pressure gradient of about 2 kPa/m required to transport sand particles in a viscous Newtonian oil.
- **Gillies *et al* (2007)**
  - Propose that the criterion for transport is based on the ratio of the mean wall shear stress to the mean surficial particle stress.
- **This is an area of ongoing research**

Residual Pressure



Engineering Slurry Systems

- Design criteria / basis
- Slurry test requirements
- Minor losses
- Pump performance
- Hydraulic tools
  - System curve
  - Hydraulic gradeline
- Transient conditions
- P&ID Review
- Pigging
- Instrumentation

Design Criteria / Basis

- Client/owner requirements
- Site conditions
- Material properties
- Design methodology
- Standard and codes

Slurry Test Requirements

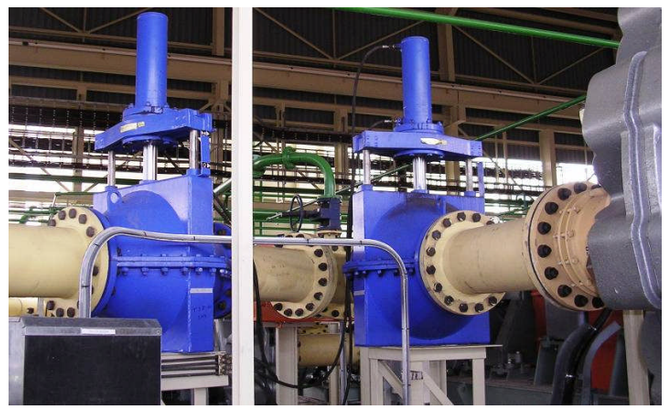
- **Information regarding the design of a slurry system is based on knowledge of the slurry flow behavior.**
- **The sources of information include:**
  - Practical experience
  - Empirical correlations or information
  - Two layer predictive models
  - Historical test data
  - Specially commissioned project specific test work
- **Yield stress slurries:**
  - There is no method for predicting the rheology of high concentration slurries.
  - The behavior of flocculated slurries is complex.

When is Test Work Required?

- **Test work is expensive:**
  - Sample collection (for green fields projects this may require that samples are generated from ore).
  - Time delays to project.
  - Actual test work costs.
- **Test work reduces risk:**
  - Reduced design factors (over design)
  - Reduced potential for design failures or extensive post commissioning modifications
- **It is the responsibility of the Designer to balance**

Information Required versus Risk

Minor Losses



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Pump Peformance

- Performance derating
- Pump suction conditions
- Pump blockage

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Centrifugal Pump Explosions

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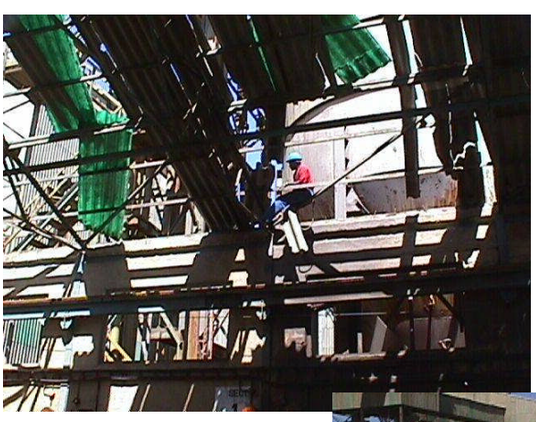
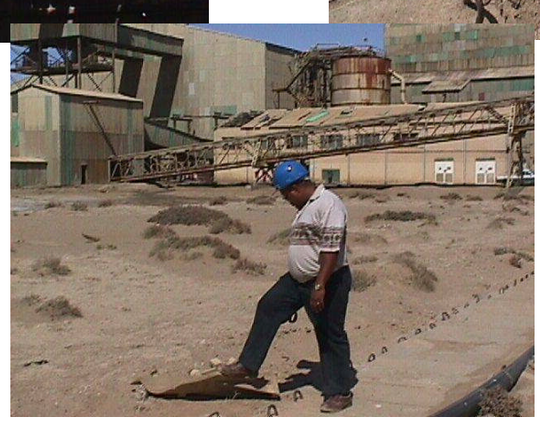
Centrifugal Pump Explosions





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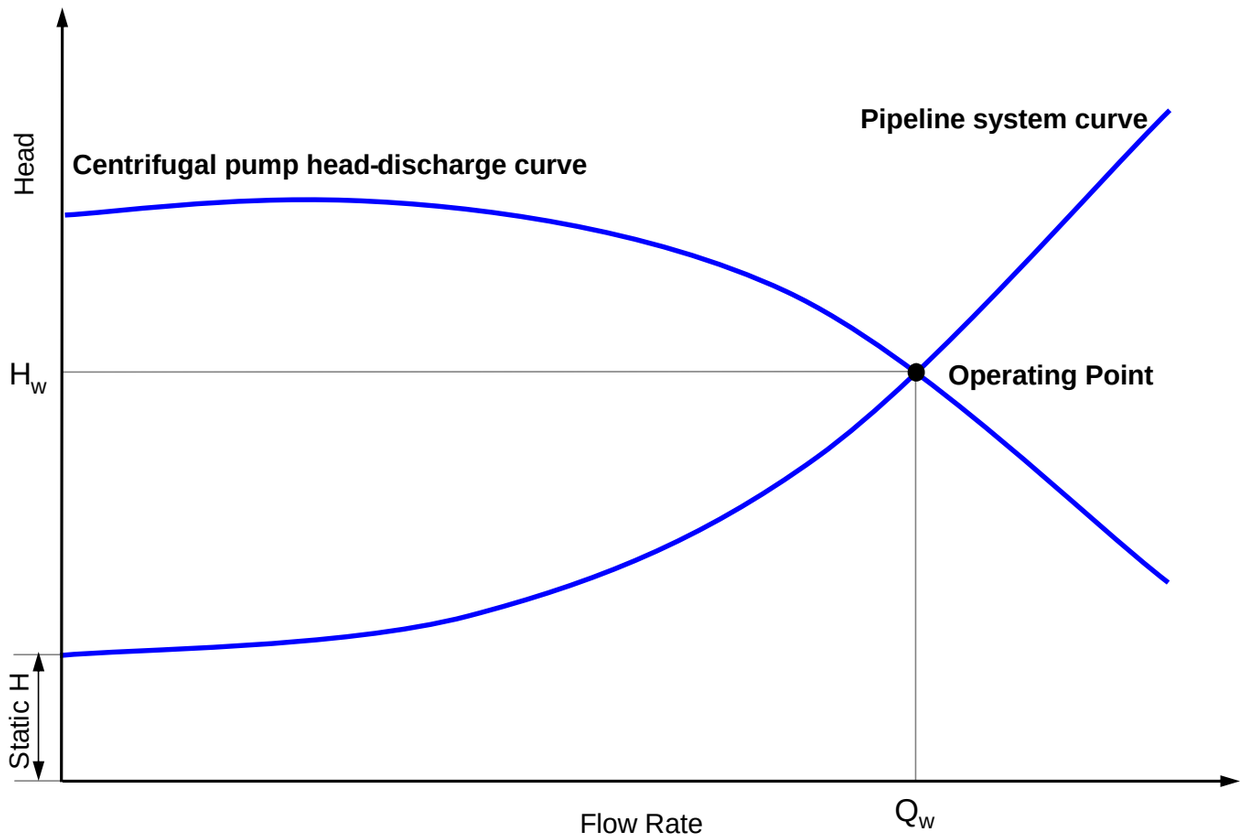
System Curve



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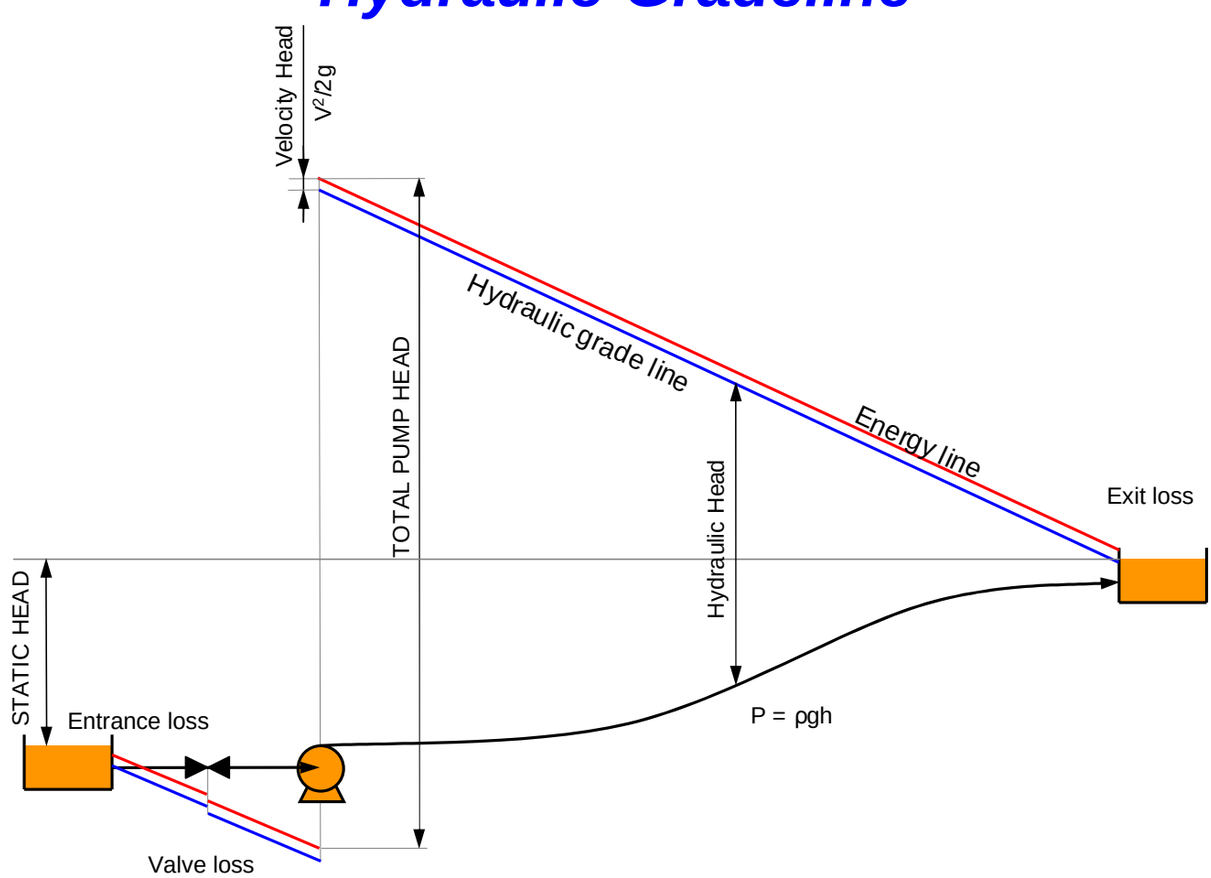
Hydraulic Gradeline



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Choke Station

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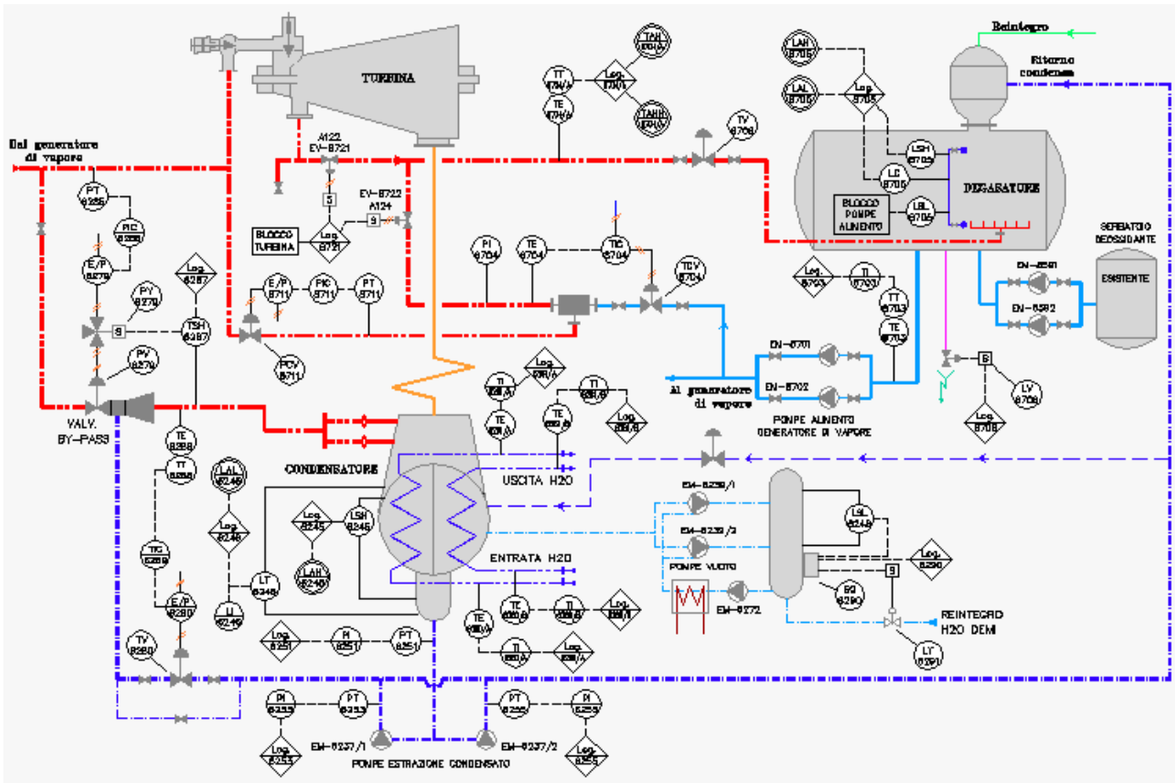
P&ID Review



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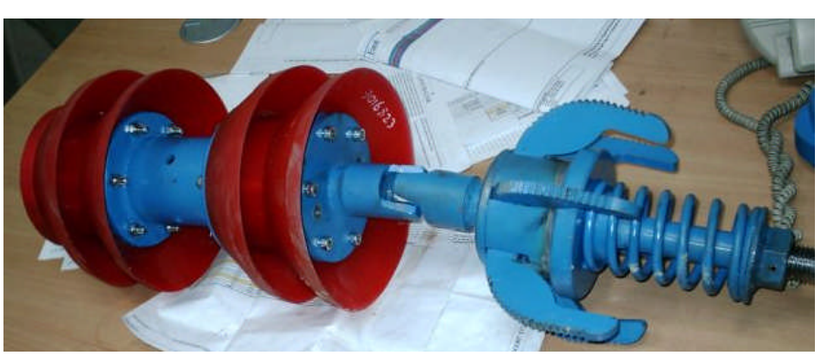
Pigs



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Instrumentation

- Simple
- Control and Measure

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Conclusion

"The conviction was that the key to the design of slurry systems which would operate reliably lay, not in the selection of exotic materials or the design of special equipment, but in the *understanding* and control of the slurry environment"

EJ Wasp

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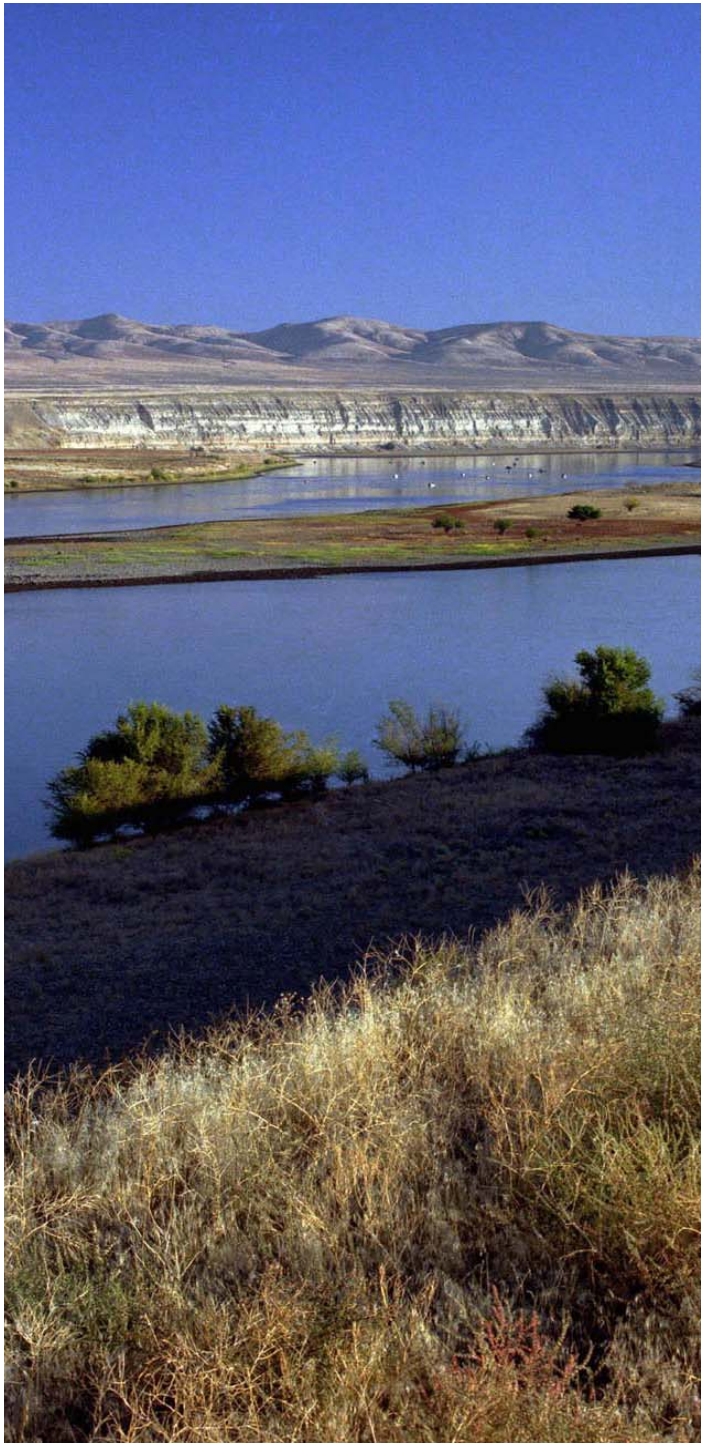
Appendix A.5

Other Special Topic Presentations



Slurry Retrieval, Pipeline Transport & Plugging, and Mixing Workshop

Gravity Flow Systems

River Protection Project

WASTE TREATMENT PLANT

MN Hall
1-16-98

Department of Energy

Office of River Protection

Bechtel National, Inc.

Examples of WTP Gravity Flow Systems

- Internal Process Lines (pitch from high point to both source and receiver vessels after transfer)
  - 3-inch to 24-inch lines
  - Feed Receipt lines
- Breakpots (Steam ejectors to high point, vents steam, then gravity flow to receiver vessel)
- Flush to Drain (change of fluid, solids build up, plug removal)
- Floor and Fire water drains (C2, C3 and C5 areas)

Chemical Plugging

- BNI Design Guide

24590-WTP-GPG-M-0059, Rev 0
Avoiding Chemical Line Plugging - Plant Design Considerations
Effective Date: 15 November 2007

R10862386

Approved By:

Prepared By: MN Hall

MW Hoffmann, M&PE — Date 11/14/07
Deputy Discipline Production Engineering Manager

Design Guide:

Avoiding Chemical Line Plugging - Plant Design Considerations

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WTP Gravity Flow

- 1 to 21 cP , Newtonian Slurries with less than 5% solids
- Fill factors 50 to 70% (vented flow)
- Typical slopes
  - 1/100
  - 1/50
  - 1/20

Gravity Flow Methodology

Section 1 - Critical Flow

$g := 32.2$

Fill Factor is equal to: $F = \frac{y}{D} = \%$ Defined Fill Factor $F := \frac{70}{100}$

Schedule 40 S Pipe

1 inch
1 1/2 inch
2 inch
3 inch
4 inch
6 inch
8 inch
10 inch
12 inch

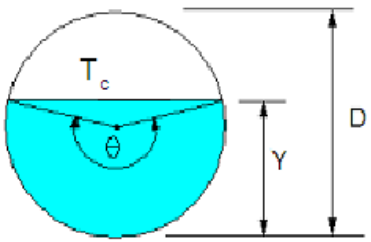
$d = 7.981$ in

$D := \frac{d}{12}$

$\theta := 2 \cdot acos(1 - F \cdot 2)$ $\theta = 3.965$ rads

$A_c := \frac{(D)^2}{8} \cdot (\theta - \sin(\theta))$ $A_c = 0.26$ ft^2

$T_c := D \cdot \sin\left(\frac{\theta}{2}\right)$ $T_c = 0.6096$ ft



$Q_c := 1$

Given $\frac{A_c^3}{T_c} = \frac{Q_c^2}{g}$ $Q_{gpm} := Find(Q_c) \cdot 448.83$ $Q_{gpm} = 431.9$ gpm

Pipe Throat Velocity $V_c := \frac{\frac{Q_{gpm}}{448.83}}{A_c}$ $V_c = 3.7$ $\frac{ft}{s}$

Liquid Surface Height $E := \left(D \cdot F + \frac{V_c^2}{2 \cdot g}\right) \cdot 12$ $E = 8.1435$ in

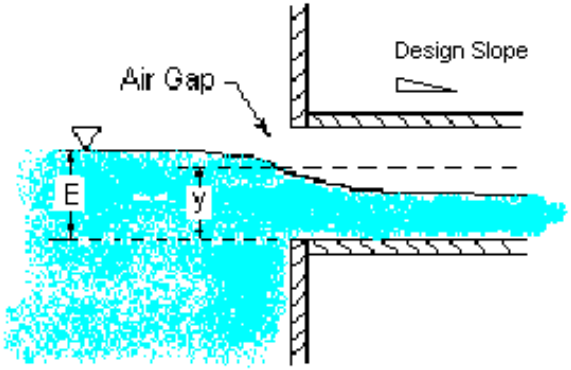


Figure Overflow Inlet

Gravity Flow Systems

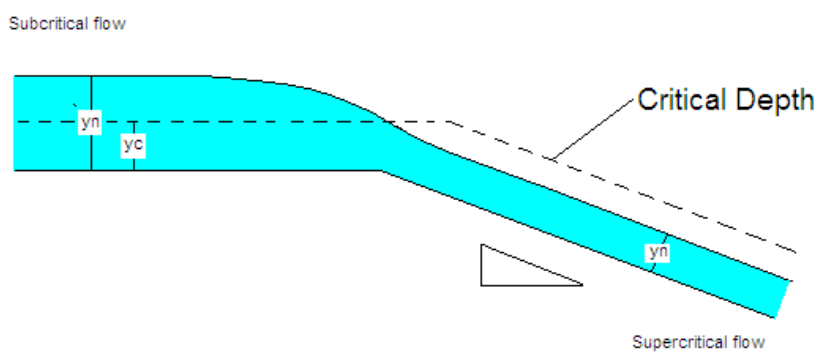
Critical Slope

Assumed Manning's n $n := .011$ $Q_{cfs} := \frac{Q_{gpm}}{(448.83)}$

Hydraulic Radius: $R_h := \frac{2 \cdot A_c}{\theta \cdot D}$ $R_h = 0.19702\ ft$ $S_c := 1$

Manning Equation: Given $Q_{cfs} = \frac{1.49}{n} \cdot A_c \cdot R_h^{\frac{2}{3}} \cdot S_c^{\frac{1}{2}}$ $S_c := Find(S_c)$ Uses Critical Flow and Area as input - Result is critical slop

$S_c = 0.00652$ **Critical Slope:** $\frac{1}{S_c} = 153$ The Critical Slope is the slope that the pipe would have to be at if the surface level anc the bottom of the pipe were equal. (Lquid surface was at the hydraulic gradiant.)



Gravity Flow Systems

Section 3 - Supercritical Flow and Fill Factor

The Manning equation, substituting equations from Section 1 for A_c, and R_h from Section 2. Design Slope: $S_d := \frac{1}{20}$

Given $Q_{cfs} = \frac{1.49}{n} \cdot \left[\frac{D^2}{8} \cdot (\theta - \sin(\theta))\right] \cdot \left[\frac{2 \cdot \left[\frac{D^2}{8} \cdot (\theta - \sin(\theta))\right]}{\theta \cdot D}\right]^{\frac{2}{3}} \cdot S_d^{\frac{1}{2}}$ $\theta := \mathrm{Find}(\theta)$

$A_c := \frac{D^2}{8} \cdot (\theta - \sin(\theta))$

Velocity At Supercritical Flow : $V_{c2} := \frac{Q_{cfs}}{A_c}$ $V_{c2} = 8.02 \frac{ft}{s}$

Given $\theta = 2 \cdot \mathrm{acos}(1 - F \cdot 2)$ $F_{sc} := \mathrm{Find}(F) \cdot 100$

Fill Factor % at Supercritical Flow : $F_{sc} = 37.7$ %

New Wetted Perimeter: $\theta \cdot D = 1.759$ ft

given $\theta \cdot D = \pi \cdot D_2$ $D_2 := \mathrm{find}(D_2) \cdot 12$ $D_2 = 6.72$ in

Stationary Bed Formation

Initial guess for critical velocity: $Vcr := 1\,\frac{ft}{sec}$

Given

$$Vcr = \left| \begin{array}{l} Cs\mu \leftarrow \dfrac{Cwt}{\dfrac{\rho s}{\rho L} - \left(\dfrac{\rho s}{\rho L} - 1\right)\cdot Cwt}\cdot\left(\eta_{homo}\right)\cdot\eta 1 \\ Cs \leftarrow \dfrac{Cwt}{\dfrac{\rho s}{\rho L} - \left(\dfrac{\rho s}{\rho L} - 1\right)\cdot Cwt}\cdot\left(1 - \eta_{homo}\right)\cdot\eta 1 \\ \mu M \leftarrow 2\cdot\left[1 + 2.5\cdot Cs\mu + 10.05\cdot Cs\mu^{2} + 1.3\cdot\left(\exp\left(17\cdot Cs\mu\right) - 1\right)\right]\cdot\left(\dfrac{8\cdot Vcr\cdot\frac{sec}{m}}{\frac{D}{m}}\right)^{-0.06} \\ F \leftarrow \sqrt{9.81\,\frac{dp}{m}\cdot\left(\frac{\rho s}{\rho L} - 1\right)} \\ (F)\cdot 1.85\,Cs^{0.1536}\cdot(1 - Cs)^{0.3564}\cdot\left(\dfrac{\frac{dp}{m}}{\frac{D}{m}}\right)^{-0.378}\cdot\left[\dfrac{\frac{D}{m}\cdot\rho L\cdot\frac{m^{3}}{kg}\cdot(F)}{\frac{\mu M}{1000}}\right]^{0.09}\cdot\chi^{0.3}\cdot(1 + M)\cdot\frac{m}{sec} \end{array} \right.$$

Homogeneous Solids Fraction

Heterogeneous Solids Fraction

Viscosity Adjustment (RPP-9805, Section 6)

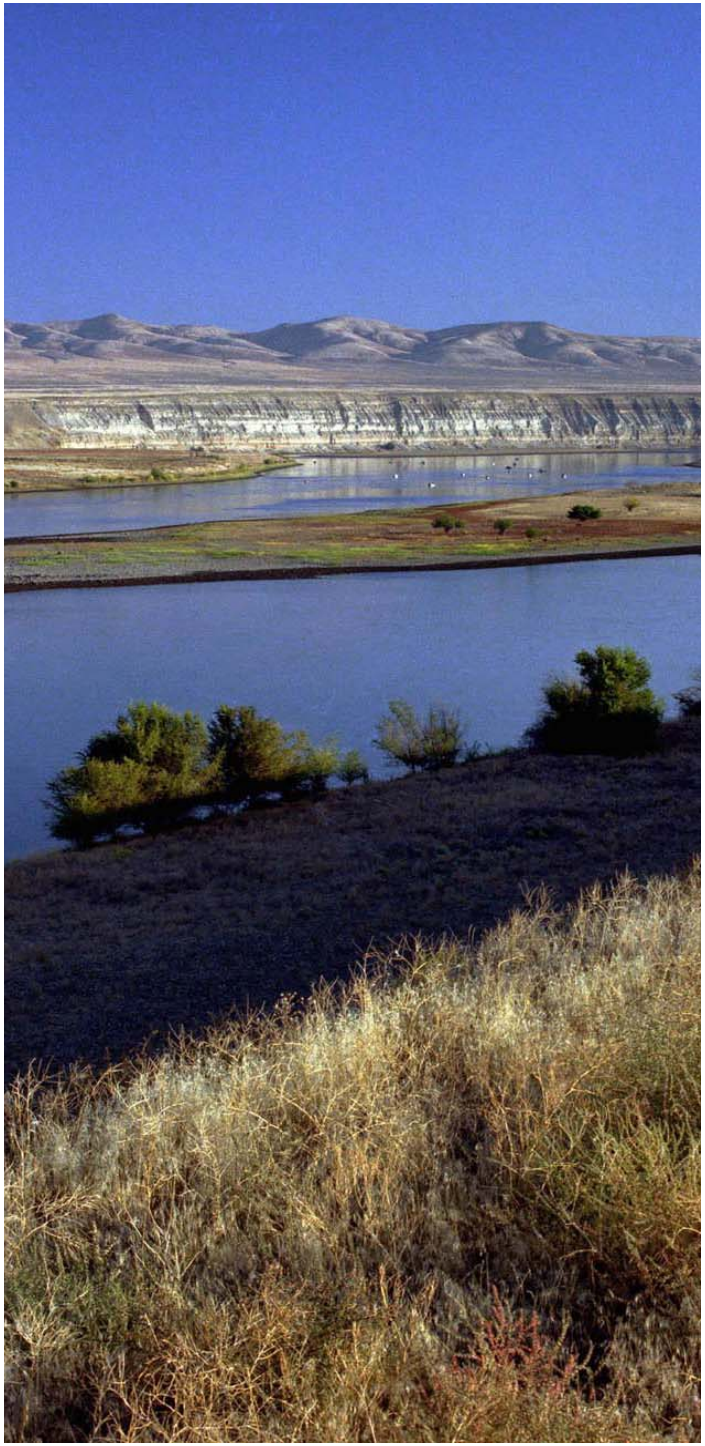
OT Equation

$$Vcr\left(D, dp, \rho s, \rho L, Cwt, \chi, M, \eta_{homo}, \eta 1\right) := \mathrm{Find}(Vcr)$$

Velocity Check

Affective Pipe diameter:	$D := 6.72in$	From Critical Flow Estimate
Particle diameter:	$dp := 210 \times 10^{-6} m$	
Solids density:	$\rho s := 2180 \frac{kg}{m^3}$	
Liquid density:	$\rho L := 1100 \frac{kg}{m^3}$	
Solids weight percent:	$Cwt := 16.7\%$	
Fraction of eddies with velocities exceeding the hindered settling velocity of solids:	$\chi := 0.95$	
Design margin:	$M := 0\%$	
Solids Homogenous Fraction:	$\eta_{homo} := 75\%$	74 micron fines/coarse
Solids in pipe at start of flow:	$\eta 1 := 100\%$	$Vcr(D, dp, \rho s, \rho L, Cwt, \chi, M, \eta_{homo}, \eta 1) = 3.584 \frac{ft}{sec}$

Critical Flow = 8.2 ft/s or 128% margin



Slurry Retrieval, Pipeline Transport & Plugging, and Mixing Workshop

Critical Velocity w/ Viscosity Adjustment

River Protection Project

WASTE TREATMENT PLANT

MN Hall
1-16-98

Department of Energy

Office of River Protection

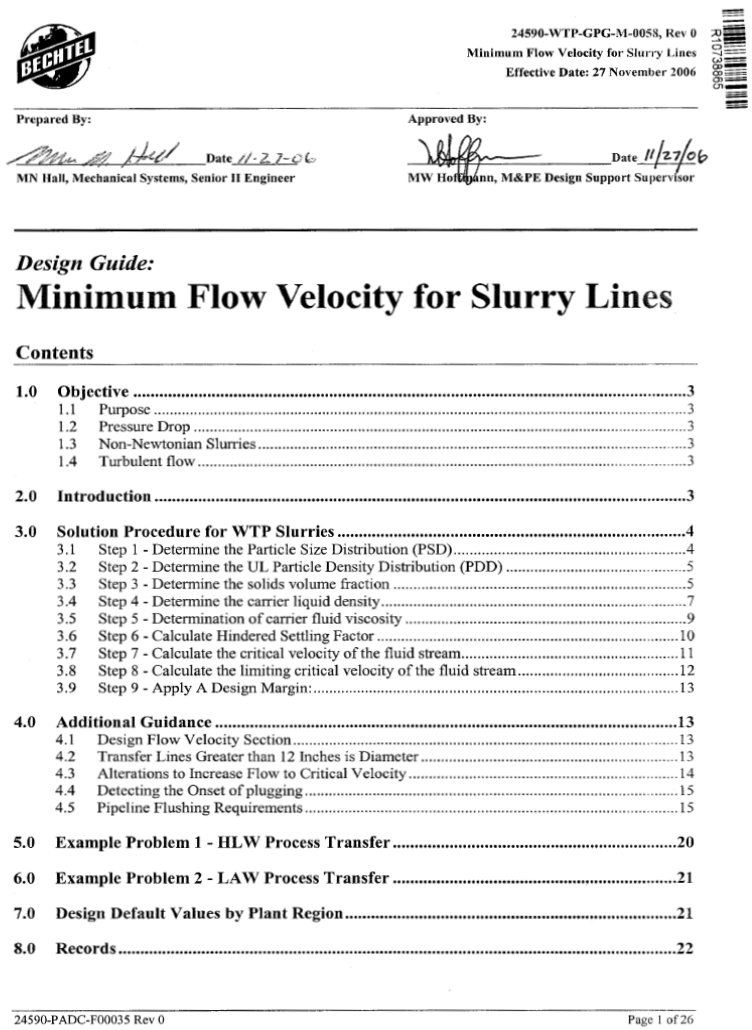
Bechtel National, Inc.

Hanford Waste Treatment Plant (WTP)

Newtonian Fluids

Introduction

- Largest nuclear facility under construction in the world.
- Will process 120 Million gal of defense nuclear waste over 30-40 yrs.
- 70 Mgal of High Level Waste (HLW) and 50 Mgal Low level waste (LAW)



24590-WTP-GPG-M-0058, Rev 0
Minimum Flow Velocity for Slurry Lines
Effective Date: 27 November 2006

Prepared By: Date 11-27-06
MN Hall, Mechanical Systems, Senior II Engineer

Approved By: Date 11/27/06
MW Hoffmann, M&PE Design Support Supervisor

Design Guide:
Minimum Flow Velocity for Slurry Lines

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Non-Newtonian

Non-Newtonian Fluids

Chemical Plugs

24590-WTP-GPG-M-0059, Rev 0
Avoiding Chemical Line Plugging - Plant Design Considerations
Effective Date: 15 November 2007

Prepared By: MN Hall
Approved By: MW Hoffmann, M&PE Deputy Discipline Production Engineering Manager — Date 11/14/07

Design Guide:

Avoiding Chemical Line Plugging - Plant Design Considerations

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Bingham Plastics

R10764685

24590-WTP-GPG-M-016, Rev 2
Pipe Sizing for Lines with Liquids Containing Solids - Bingham Plastic Model
Effective Date: 8 August 2007

Prepared By: MN Hall
Approved By: MW Hoffmann, M&PE Design Support Supervisor — Date 8/6/07

Design Guide:

Pipe Sizing for Lines with Liquids Containing Solids - Bingham Plastic Model

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Power Law Fluids

24590-WTP-GPG-M-039, Rev 2
Determination of Pressure Drop for Lines with Liquids Containing Solids - Power Law Fluids
Effective Date: 7 June, 2007

Prepared By: MN Hall
Approved By: MW Hoffmann, M&PE Design Support Supervisor — Date 6/7/07

Design Guide:

Determination of Pressure Drop for Lines with Liquids Containing Solids - Power Law Fluids

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Table 1 Darby's 3-K Constants for Loss Coefficients, Calculation of Pressure Drop for Valves and Fittings 6

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Risk Management

- Due to unknown rheology
  - Lack of comprehensive characterization
  - Blending issues
  - Changing waste forms (hydrates, reactions, concentrations)
- High dilution rates
  - to cause Newtonian like behavior – add water
  - Force waste to behave like model – can predict
- Evaporation at destination
  - Usually storage tank
  - Unknown rheology
- Interface Control Documents (ICD-19 – Waste Feed Criteria)

Slurry Flow In Pipelines

- Data Conversion is Important Issue
- wt% solids measured many different ways (25 C, 105 C, 1200 C)
- Bulk density reconciliation

$$\rho_s = \frac{\rho_b - \rho_L + \rho_L C_{vSludge}}{C_{vSludge}}$$

where
ρ_s = Density of Solids, (g/mL)
ρ_L = Density of Liquid, (g/mL)
ρ_b = Bulk density of sludge, (g/mL) (Note: Not Slurry, sludge only)
$C_{vSludge}$ = Solids fraction of sludge, vol% (Note: Not slurry, sludge only)

$$C_v = \frac{(0.001)C}{\rho_{solid}}$$

where
C_v = Solids fraction of Slurry, vol%
C = Grams of dry solids per liter of slurry, g
ρ_{solid} = Dry-base solids density, g/mL

Newtonian Mixture Viscosity

- As the fluid increase in solids concentration, or the shear rate, the viscosity changes: Thomas (1965)

$$\mu_M = \mu_L \left[1 + 2.5C_V + 10.05C_V^2 + 0.00273\exp(16.6C_V)\right]$$

where μ_M = Slurry viscosity, cP
C_v = Solids fraction of Slurry, vol%

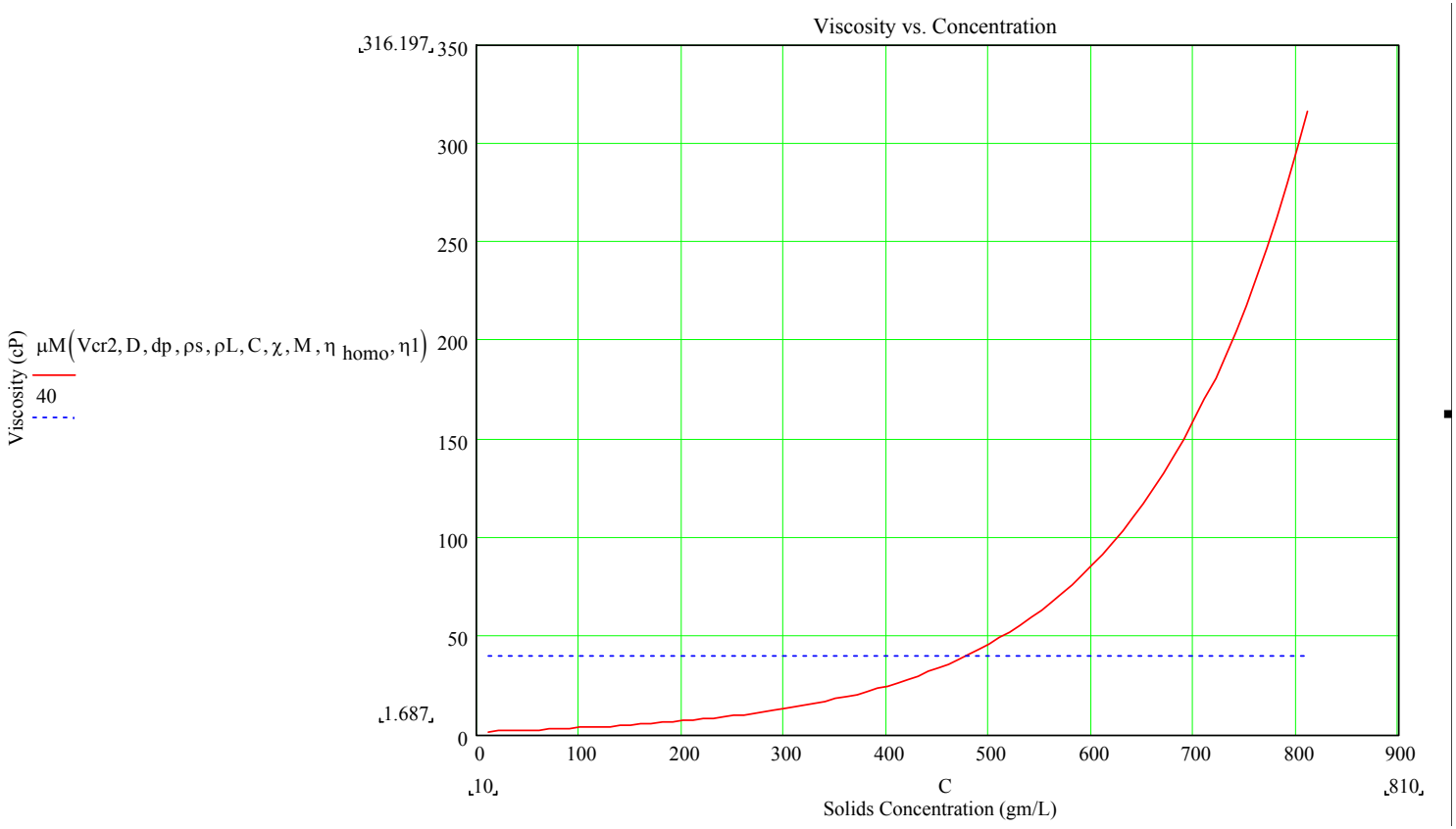
$$\mu_L = \mu_{water}\left[x_{salt}\left(1 + 1.071\left(\frac{\rho_L}{\rho_{water}}\right)\right) + x_{caustic}\exp\left(\left(7.143\left(\frac{\rho_L}{\rho_{water}} - 1\right)\right)^{1.15}\right)\right]$$

If the strain rate is expressed by $\gamma = 8\frac{V}{D}$

$$\mu_M = 2.0(1 + 2.5C_V + 10.05C_V^2 + 1.3(\exp(17C_V) - 1))\gamma^{-0.6}$$

Viscosity Results

Generally goes up with solids concentation



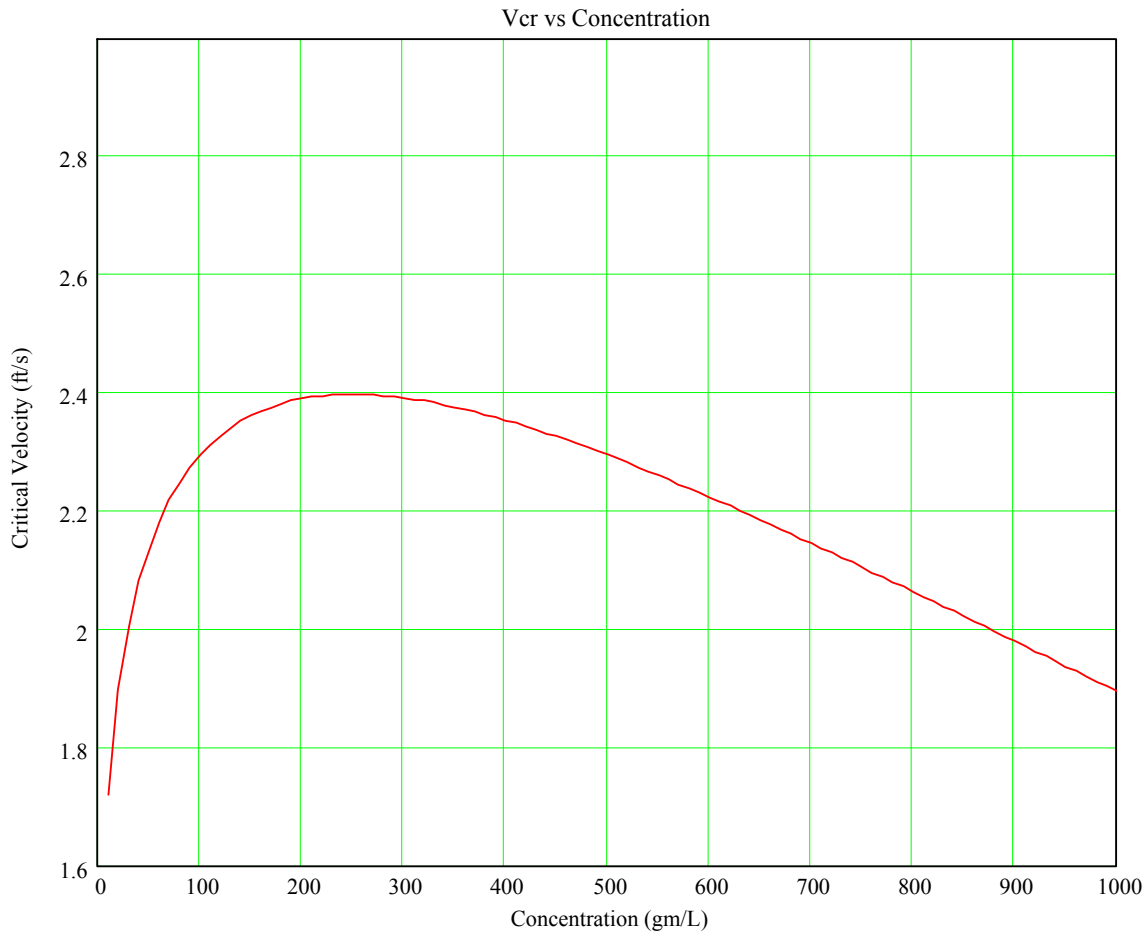
Newtonian - Critical Velocity in Pipe Lines

- Oroskar & Turian (1980)
- Function of 7 waste parameters (most descriptive)

$$V_{crOT} = \sqrt{gd_p\left(\frac{\rho_s}{\rho_L}-1\right)}\,1.85C_s^{0.1536}C_L^{0.3564}\left(\frac{d_p}{D}\right)^{-0.378}\left[\frac{D\rho_L\sqrt{gd_p\left(\frac{\rho_s}{\rho_L}-1\right)}}{\mu_L}\right]^{0.09}\chi^{0.30}$$

Combined Correlations

- Combination of OT Correlation w/ Jewett's Viscosity (reconciled with Hanford waste)



Mixing Sludges & Slurries with Pulsed Jets: Some mixing theory & Test Results

Slurry Retrieval, Pipeline Transport & Plugging & Mixing Workshop
January 14 - 18, 2008, Orlando, Florida.

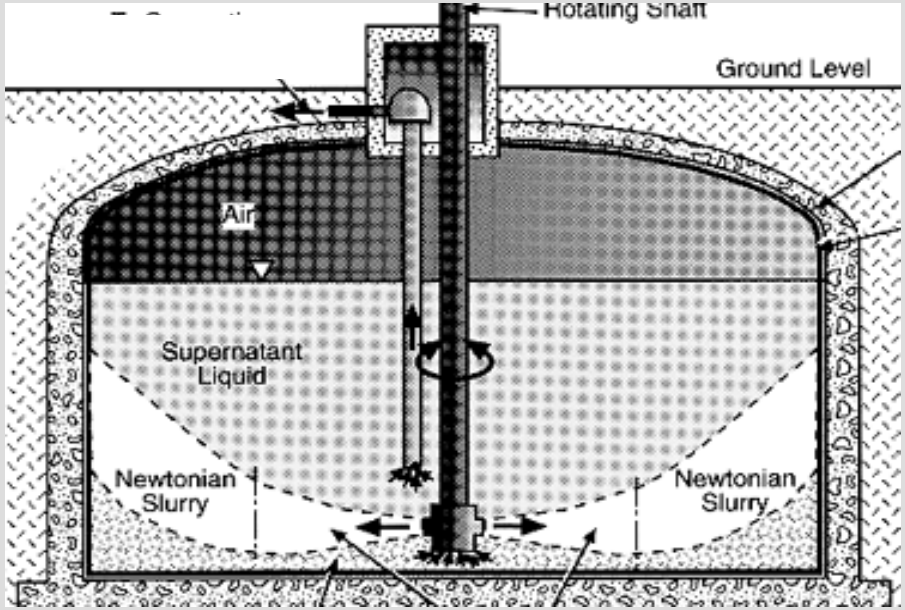
Perry A. Meyer
Pacific Northwest National Laboratory

Battelle

Pacific Northwest National Laboratory
Operated by Battelle for the U.S. Department of Energy

Unsteady Jet Mixers at Hanford

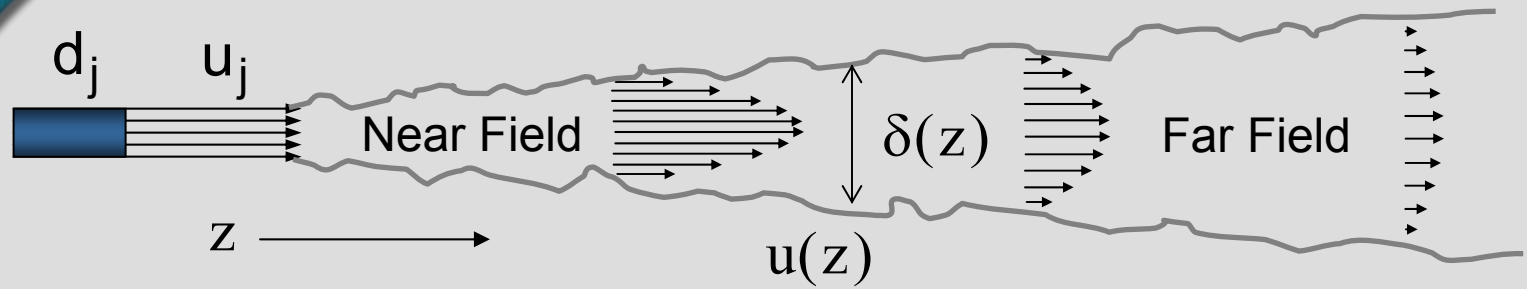
- Retrieving from storage
  - Underground, 1 - 2ft risers
  - Limited access for equipment
  - 2 - 300hp mixer pumps (baseline)
- Treating & vitrifying waste
  - Closed "black" cells
  - No maintenance for 40 years



Rotating horizontal opposed jets

Pneumatic pulsed jets

Turbulent Jets

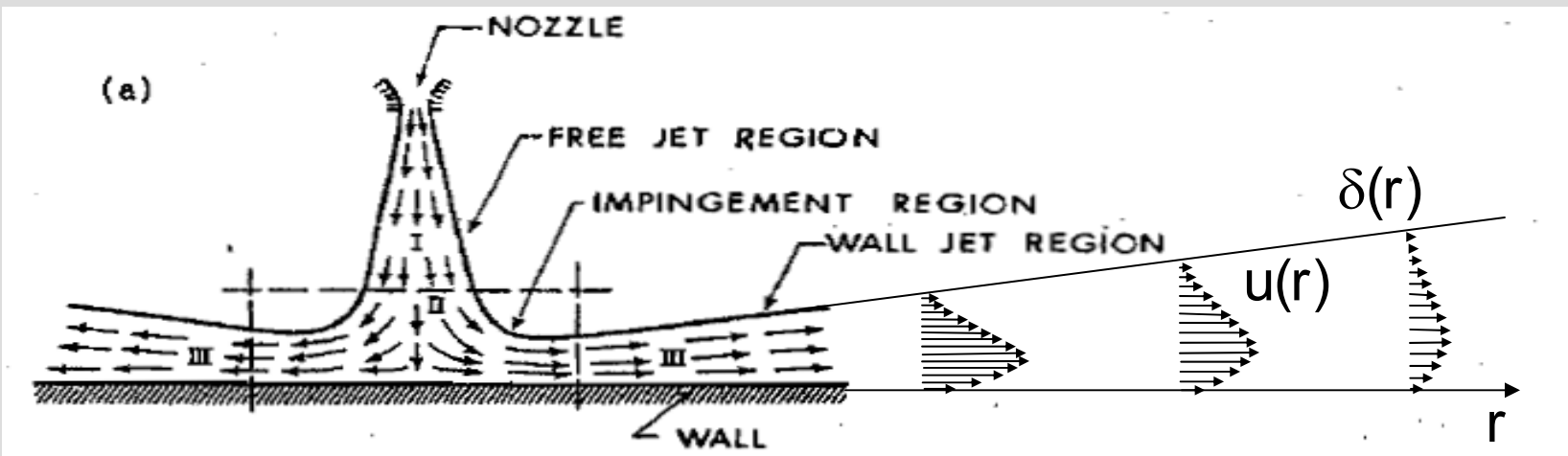


- High Reynolds number far field
  - Constant spread angle $\delta(z) = \theta z$
  - Peak & ave. velocity decrease $u(z) = c_j u_j d_j / z$ $u_d A$
  - Thrust/force is constant $F(z) = F_j$
  - Flow rate increases (entrainment) $q(z) / q_j \sim z / d$
  - Energy decreases $e(z) / e_j = d / z$
  - Constant Reynolds number $Re_\delta(z) = Re_d$

A.5.21

Turbulent Jets, cont.

- Same results for impinging & attaching jets
  - Different constants
  - Wall shear stress $\tau_w(r) \sim \rho u_j^2 (d_j / r)^2$
- True independent of nozzle cross-sectional area
- Approximately true in near-far-field transition $z / d_j,\ r / d_j = 15 - 30$
- Allows one to approximately obtain flow fields, fluxes, forces, etc
- Similar relations for dense jets



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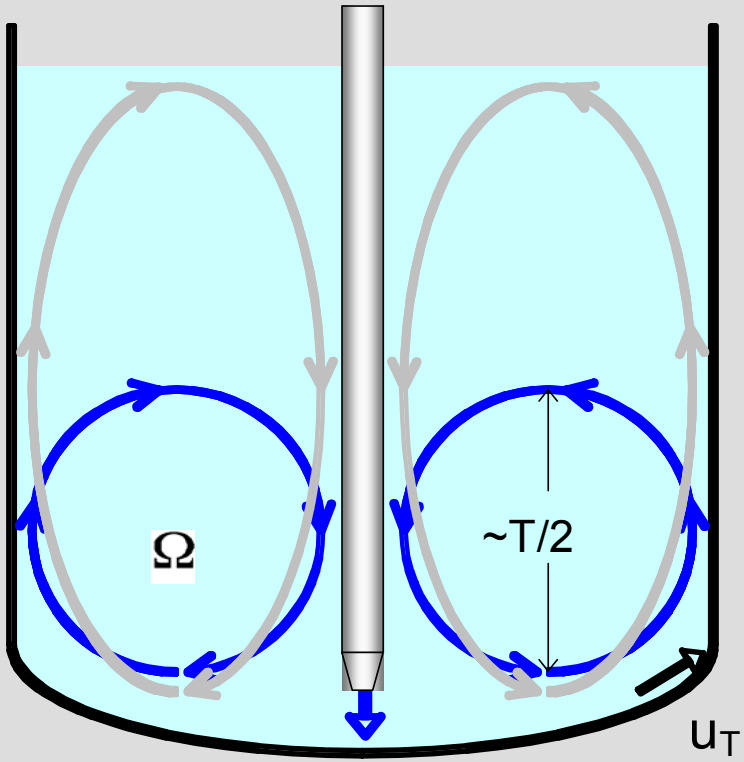
Jets as mixers

- Axial flow impeller: ND ~ uj
  - dj/T <<1 (careful about blindly applying agitator results)
  - Power, thrust, and flow numbers = ~ 1
    - Much higher power than agitators for same thrust
    - Lower flow, higher head
- Highly directional
  - point them where you want them
  - Must design for thrust reaction
- Return placement
  - Can be important

Downward vertical jet mixers

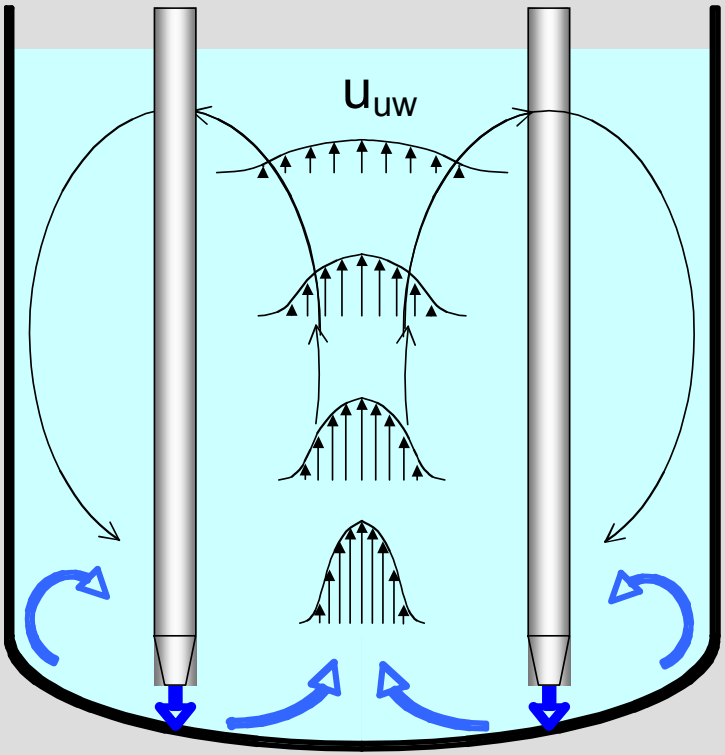
Centered Jet(s)



$$u_T \sim u_j (d_j / T)$$

$$\Omega \sim u_j d_j / T^2$$

Jet rings(s)



$$u_{uw} \sim u_j \sqrt{N_j} (d_j / T) \times f(H/T)$$

$$t_{uw} \sim T^2 / u_j d_j \sqrt{N_j} \times f^2(H/T)$$

Geometry

- Nozzle geometry
  - Cross-section: No effect in far field- only area counts
  - Convergence: extra thrust from pressure
- Stand-off
  - No effect for h/dj < 6, little effect for h/T<<1
- Number of jets
  - $N^{1/2}$dj momentum/thrust effect
  - $T/N^{1/2}$ ZOI geometric effect
- Return location
  - Can be important- Avoid short-circuiting
- Dish shape
  - Impingement angle- flow distribution
- Other internals
  - Wakes/blockages

Intermittent Jets

- Dimensionless pulse time determines regime

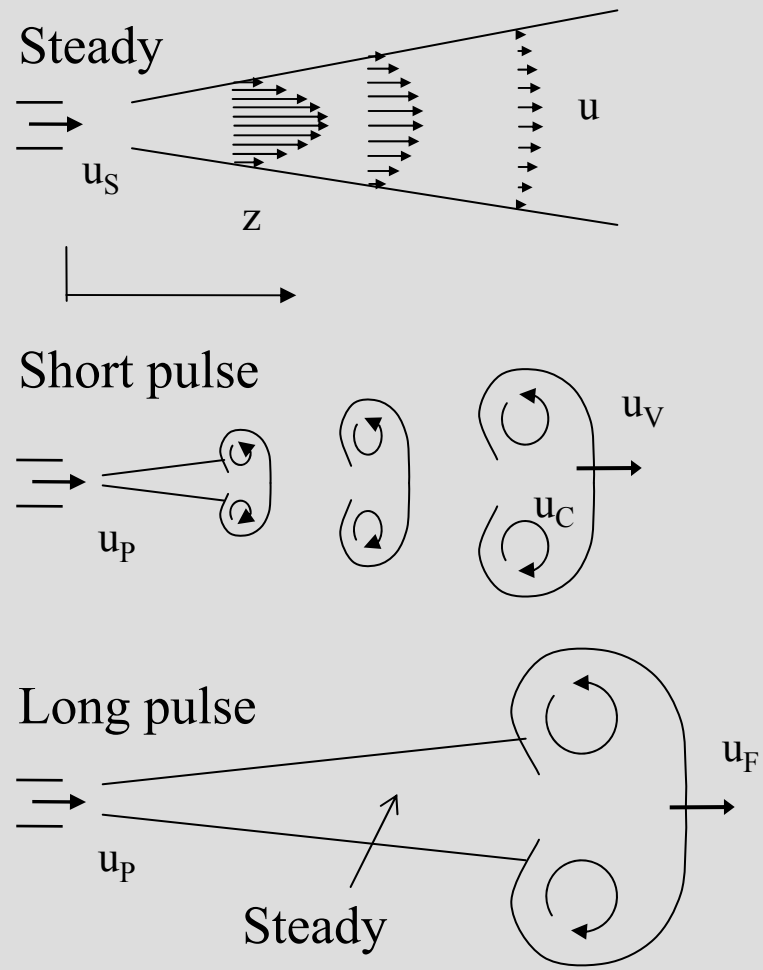
$$N_p = t_p u_d / d$$

$N_P < 4$ vortex ring

$4 < N_P$ vortex ring with tail

$4 << N_P$ developing steady

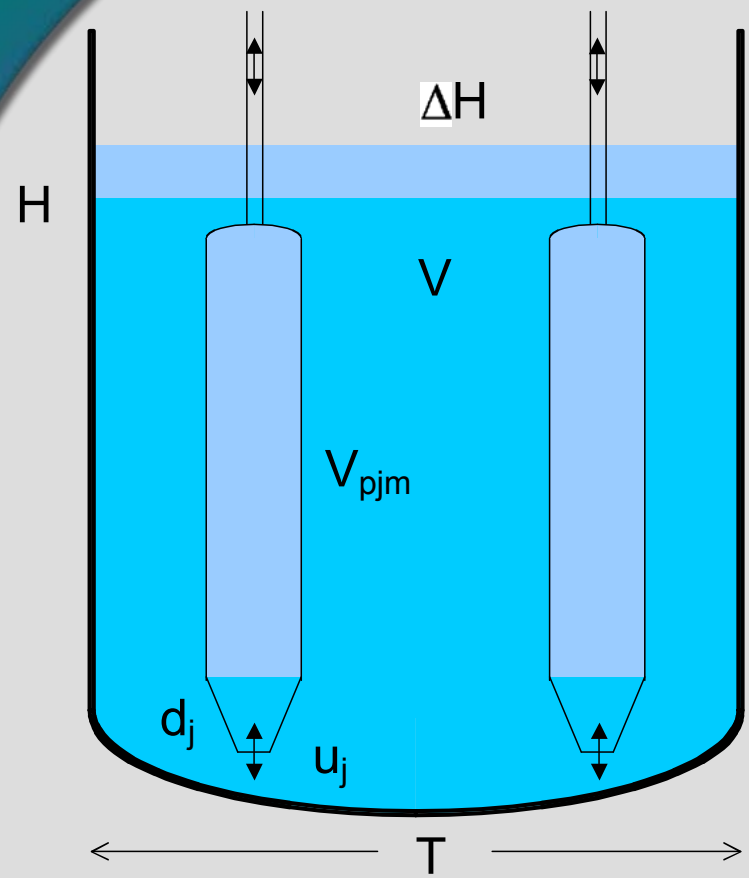
PJMs: N_P = 80 - 500

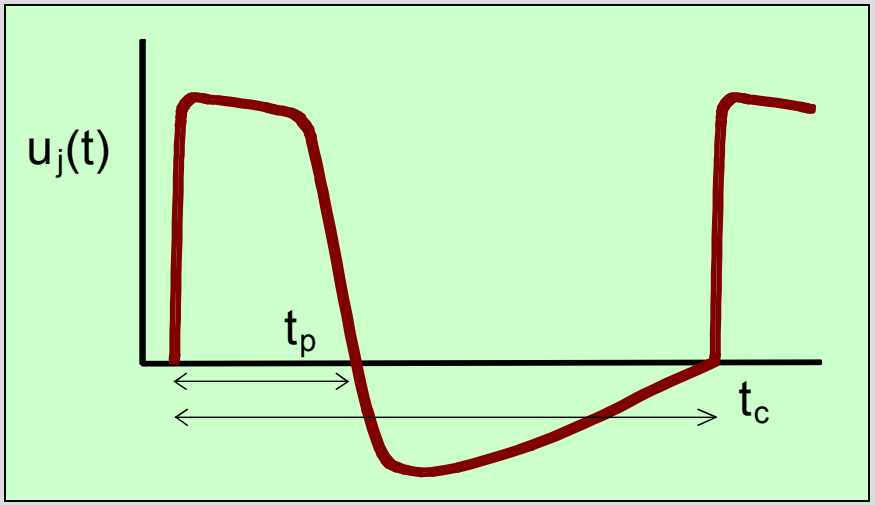


Unsteady effects on mixing/mobilization

- Would like to utilize steady mixing knowledge base
  - Can we find simple corrections for unsteady effects or are we dealing with fundamentally new phenomena?
- Must consider relative time scales
  - Flow establishment/mixing times compared with pulse time
  - Duty cycle
    - What happens when the jet is off?
  - Other time scales
    - Erosion rates
    - Settling rates
    - Etc.
- Two new parameters are introduced
  - Relative pulse volume
  - Duty cycle

Pulse jet mixers





- Mixing modes
  - drive
  - refill

- PJMs in the WTP
  - V (range)
  - N (range)
  - Pvf (range)
  - DC (range)
  - Dpjm (range)

A.5.28

Important parameters

- Geometry

N	number jets
Uj	jet velocity (peak)
d_j/T	nozzle diam.
$\Phi_p = V_p/V$	pulse size
$DC = t_p/t_c$	duty cycle
geometry	

- Operational

- Waste physical configuration
  - Normal/off-normal operations
  - Uniform
  - Settled layers

- Physical & rheological properties

Pulse Jet Mixing Studies at Battelle/PNNL

- Physical regimes
  - Transitional flow
  - unsteady
  - Non-settling/non-Newtonian
  - Settling- wide particle size & density range, agglomerates
  - Heels- cohesive/non-cohesive
  - In situ gas generation
- Mixing requirements
  - Stagnation/caverns
  - Off-bottom suspension- V_{JS}
  - Vertical distribution
  - Gas hold-up & release behavior
- Scaled testing program
  - Simulant development
    - Physical/chemical
    - Transparent/opaque
    - 1/2/3 phase
  - Testing
    - Bench scale - 40m^3
    - Single & multi jets
    - simplified & prototypic geometries
  - Scale up
    - Rating, not designing
    - Similarity, physical, empirical
  - Instrumentation

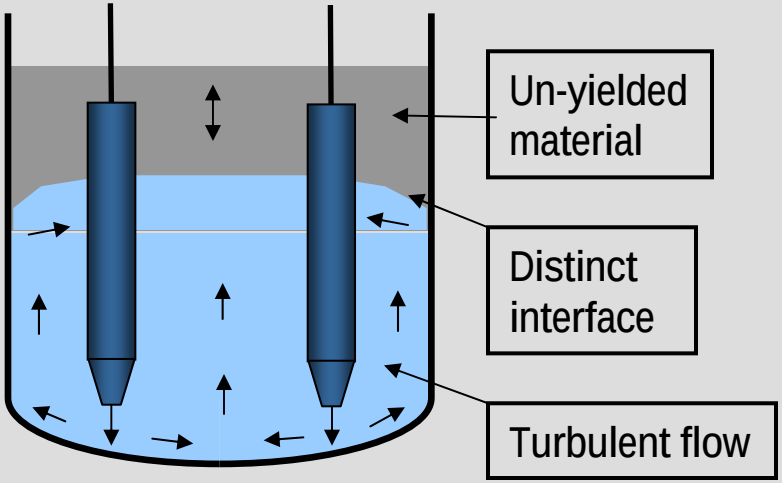
Non-Newtonian PJM Test Program

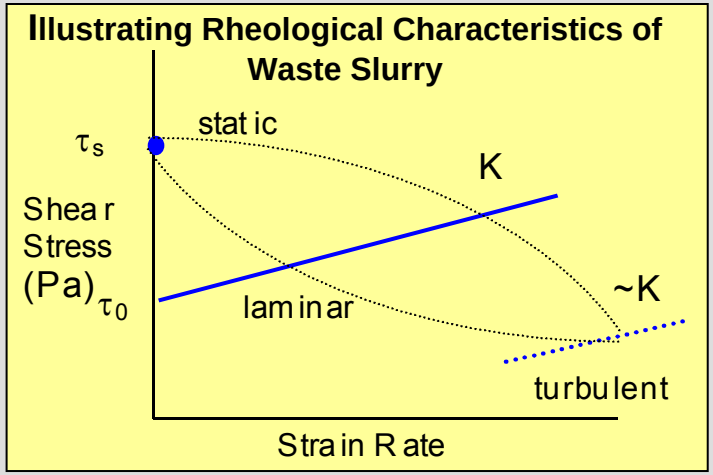
- Technical basis
  - Develop scaled testing approach
  - Validate approach- limited testing at scales
- Rate existing designs
  - (3 unique designs in WTP)
- Improved PJM designs
- PJM/sparge hybrid designs

Theory of PJM Operation with Non-Newtonian Materials

- Model problem: Cavern formation
  - Initially gelled material
  - Representative of restart after mixing shutdown
  - Good mixing system will eliminate cavern
- Rheological model
  - Static gel formation with shear strength τ_s
  - Bingham plastic laminar flow rheology with yield stress τ_0 and consistency K
  - Turbulent flow characteristics determined by high shear consistency ~K

Typical Pulse Jet Mixer System





Cavern Formation from a Steady Jet

- Turbulent wall jet

$$u(z) = c_J u_d d/z \qquad \tau_f = C_f \rho u^2/2$$

- Force balance at static interface

$$\text{at } z_C \approx H_C + T/2 \qquad \tau_f = \tau_s$$

$$H_C/T = a(d/T)Re_\tau^{1/2} - 1/2$$

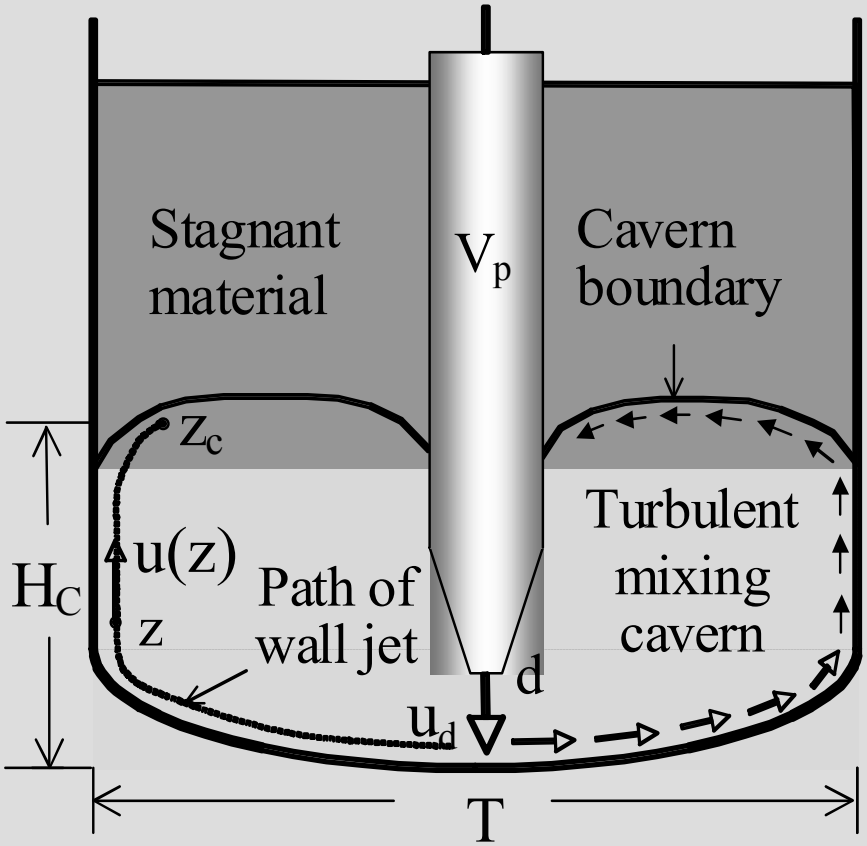
- Yield Reynolds Number

$$Re_\tau = \rho u_d^2/\tau_s$$

- Reynolds number dependence

$$Re_d = \rho u_d d/k \qquad C_f\ ,\ c_J = f(Re_d)$$

$$a \sim Re_d^{-\beta}$$



Theory of PJM Operation in Non-Newtonian Materials

- Cavern Formation from a Steady Jet
  - Turbulent jet theory with force balance at interface predicts cavern height
- Yield Reynolds number
  - Ratio fluid force to material strength

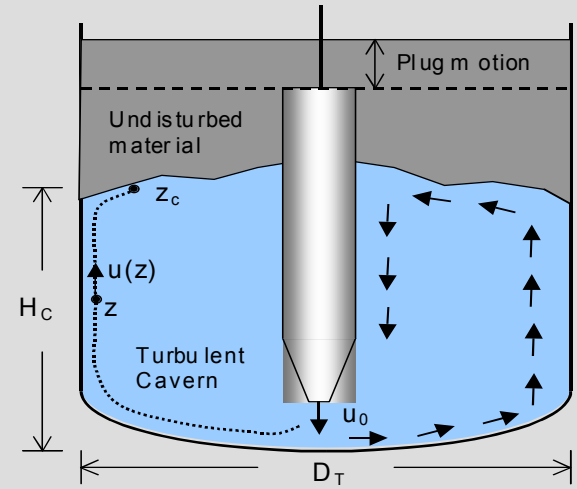
$$Re_\tau = \rho u_0^2 / \tau_s$$

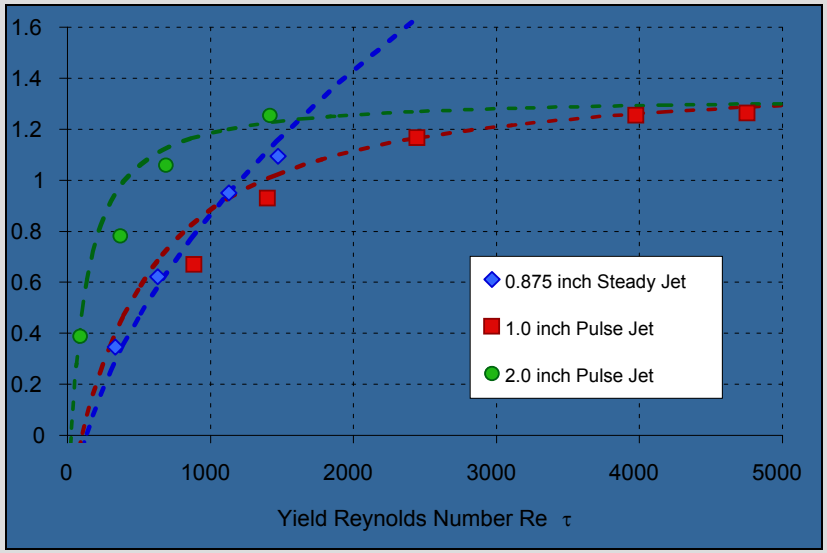
- Effects of pulsation
  - Ratio PJM drive time to flow establishment time

$$t_D / t_{ss} \sim V_p / d_0^3 Re_\tau$$

- Predicted cavern height

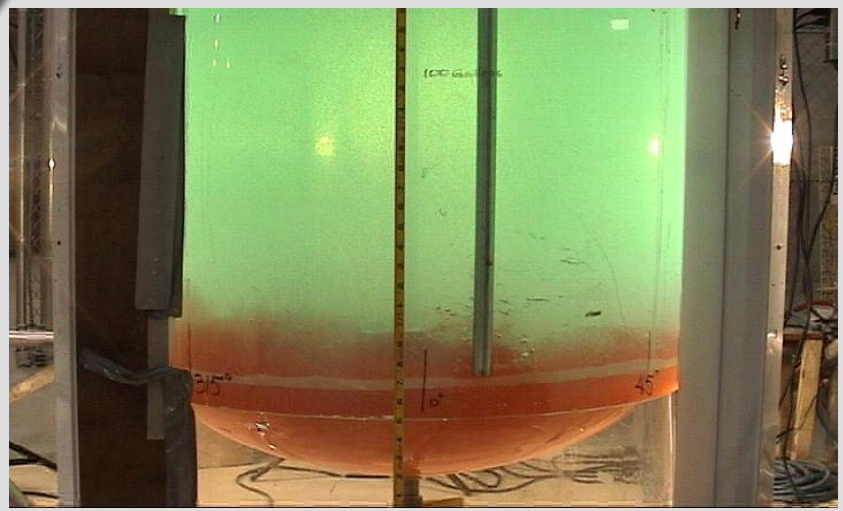
$$\frac{H_c}{D_T} = a \frac{d_0}{D_T} Re_\tau^{1/2} \left(1 - \exp(-c \frac{V_p}{d_0^3 Re_\tau})\right)^{1/2} - \frac{1}{2}$$

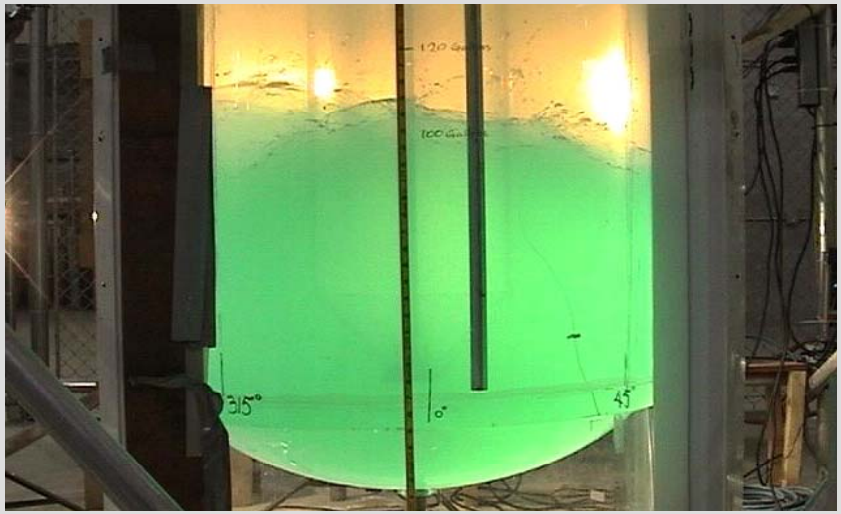


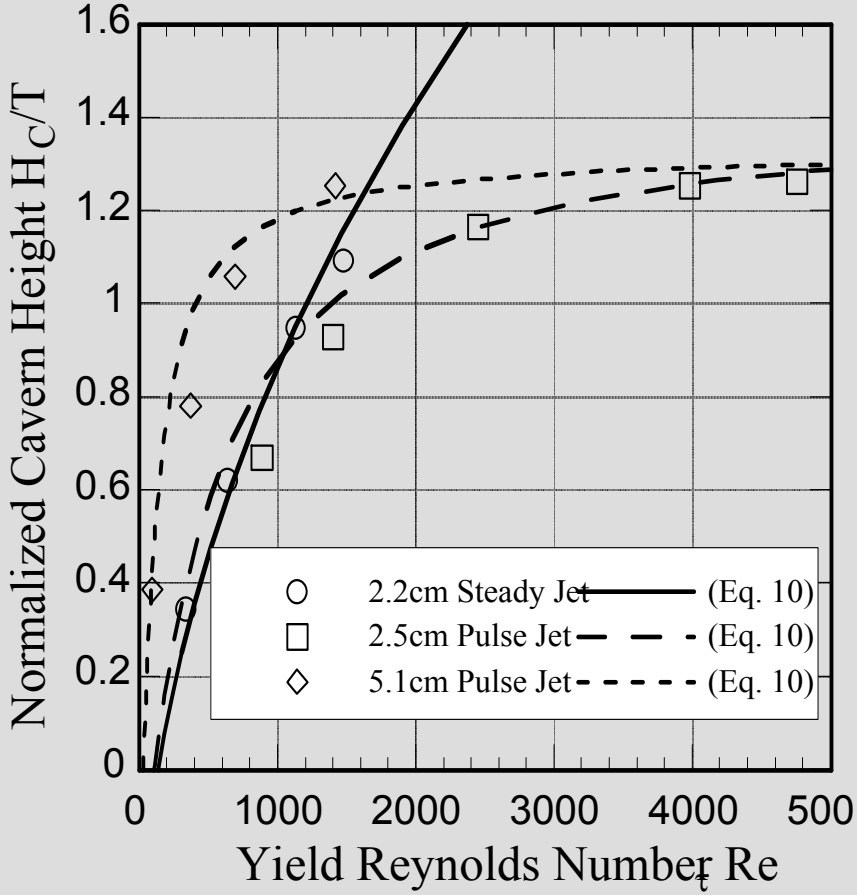


Non-dimensional cavern height as a function of yield Reynolds number for a single PJM in Laponite

Single-PJM Cavern Tests (laponite)

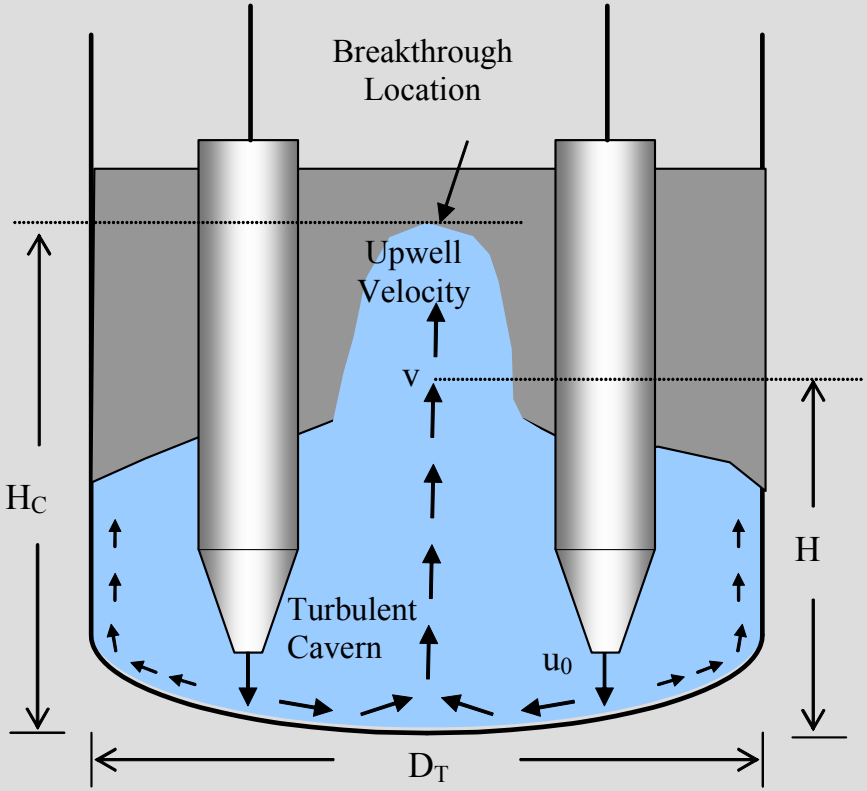






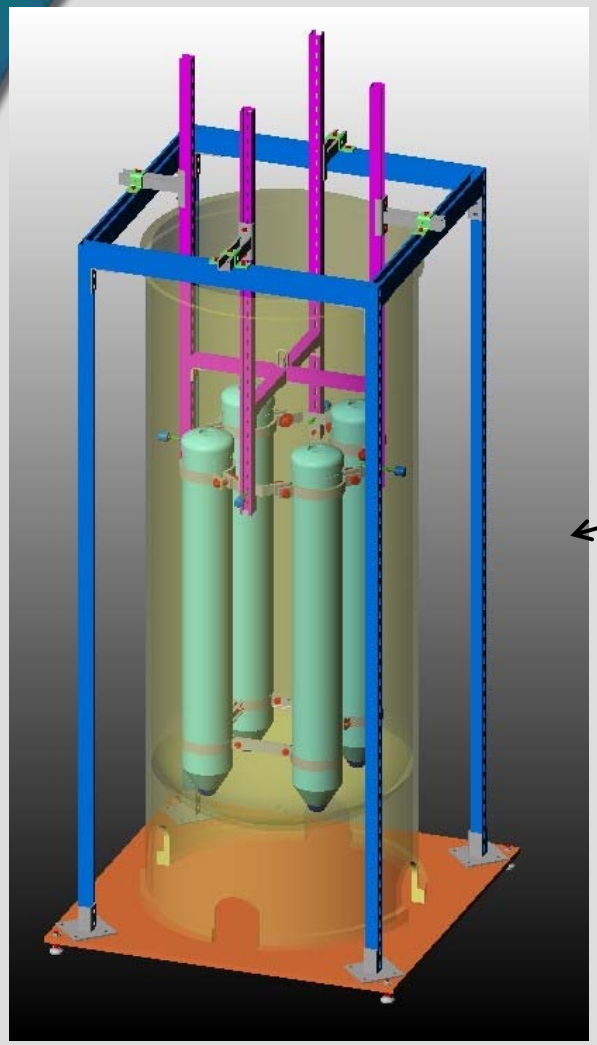
Test to Verify Scaled Testing Approach

- 1PJM Tests
  - Simulant selection
  - Verify cavern formation theory
- 4PJM Tests
  - Downward firing PJMs
  - Performed at 3 scales
- Simulants
  - Laponite
    - Transparent
    - Adjustable shear strength
  - Kaolin/Bentonite Clay
    - Opaque
    - Adjustable yield stress/consistency
- Test conditions
    - Rheology (20 -120 Pa)
    - Velocity (3-30 m/s)



- Types of measurements
  - Cavern height (Laponite)
  - Breakthrough velocity (clay & Laponite)
  - Upwell velocity (clay)

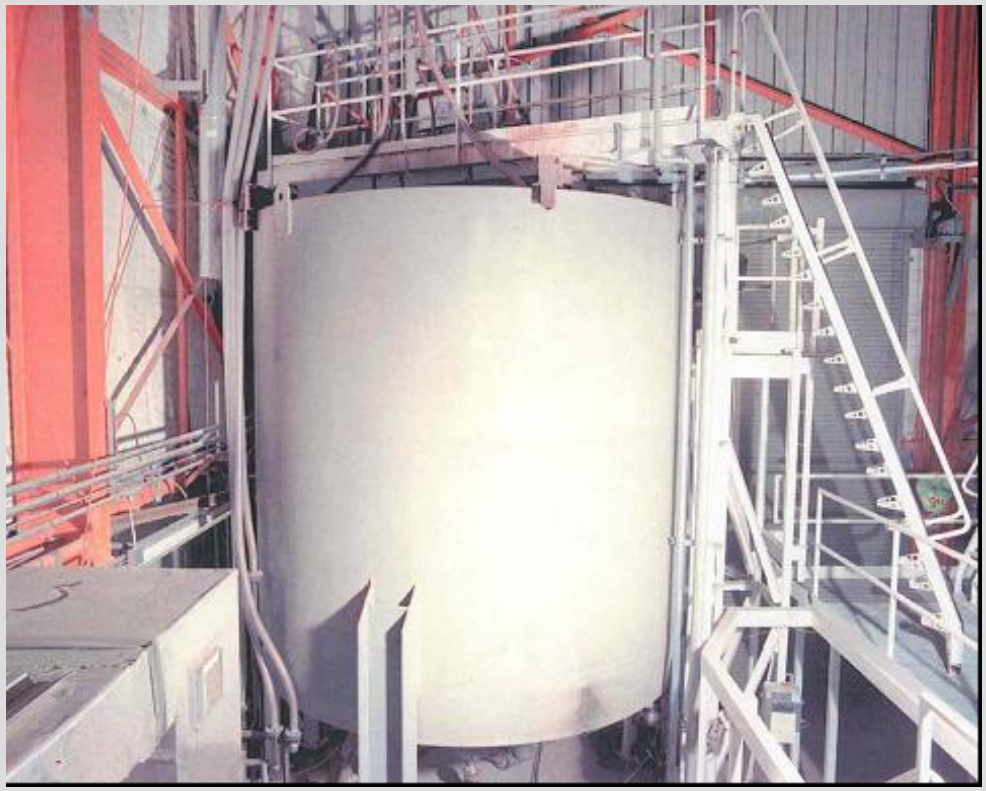
Small Scale Test Stands



- Battelle 1/4-scale 4 PJM Test Vessel
  - 34 in. diameter
  - 250 gallons
  - Acrylic vessel
  - Compressed air/vacuum PJM drive system
- SRNL 1/9-scale 4 PJM Test Vessel
  - 17 in. diameter
  - ~30 gallons
  - Acrylic vessel
  - Compressed air/vacuum PJM drive system

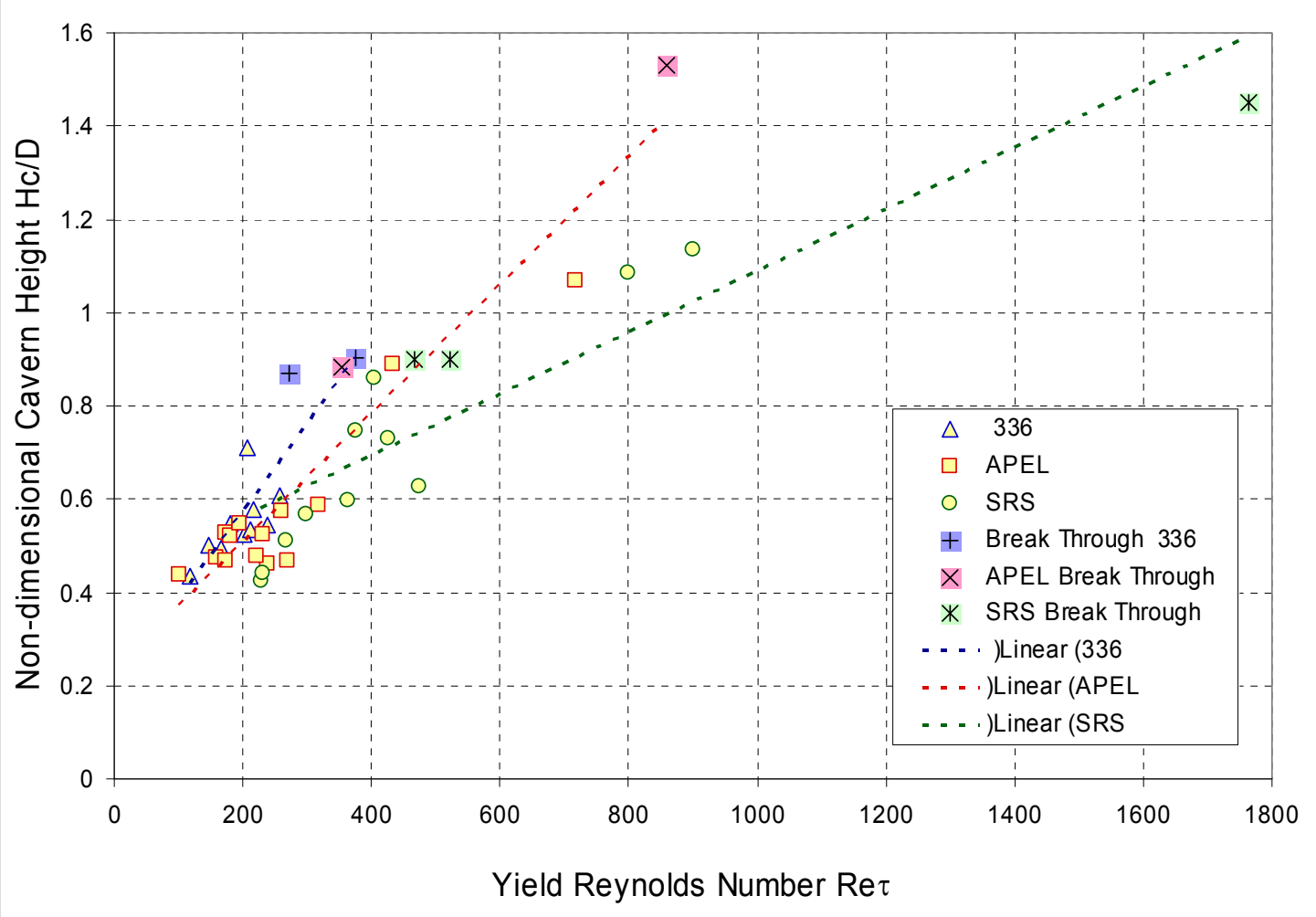
Large-Scale Test Stand at Battelle

- Battelle 336 4 PJM Test Vessel
  - ~13 ft. diameter, ~12,000 gallons
  - Steel construction
  - Prototypic AEA Compressed air PJM drive system



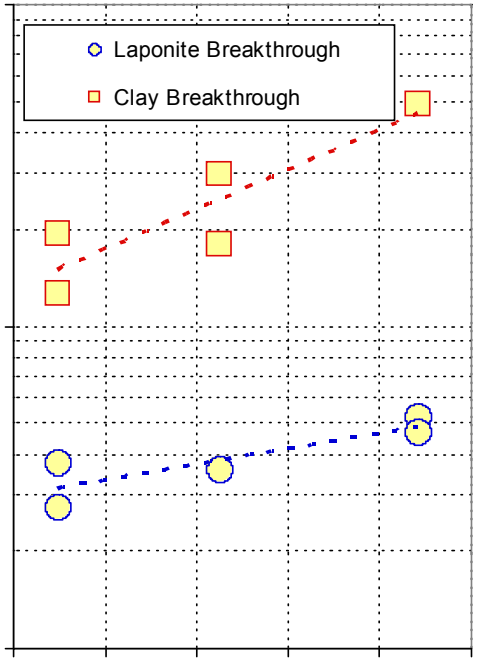
- Pulse tube prior to installation
  - 24 in. diameter
  - 2 in. conical nozzle

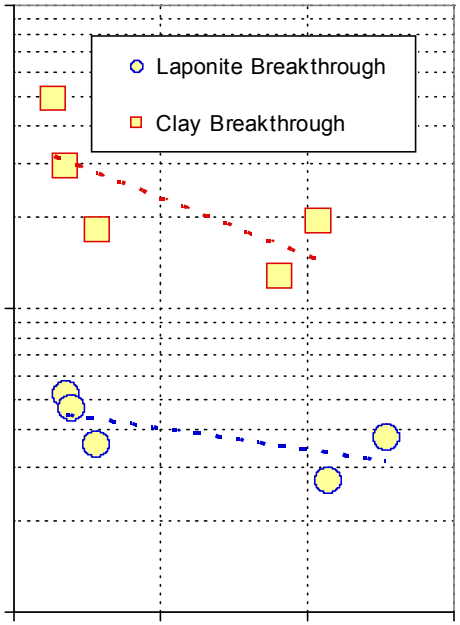
Scaling Data Comparisons



Comparison of cavern position for tanks of 3 different scales with Laponite simulant.

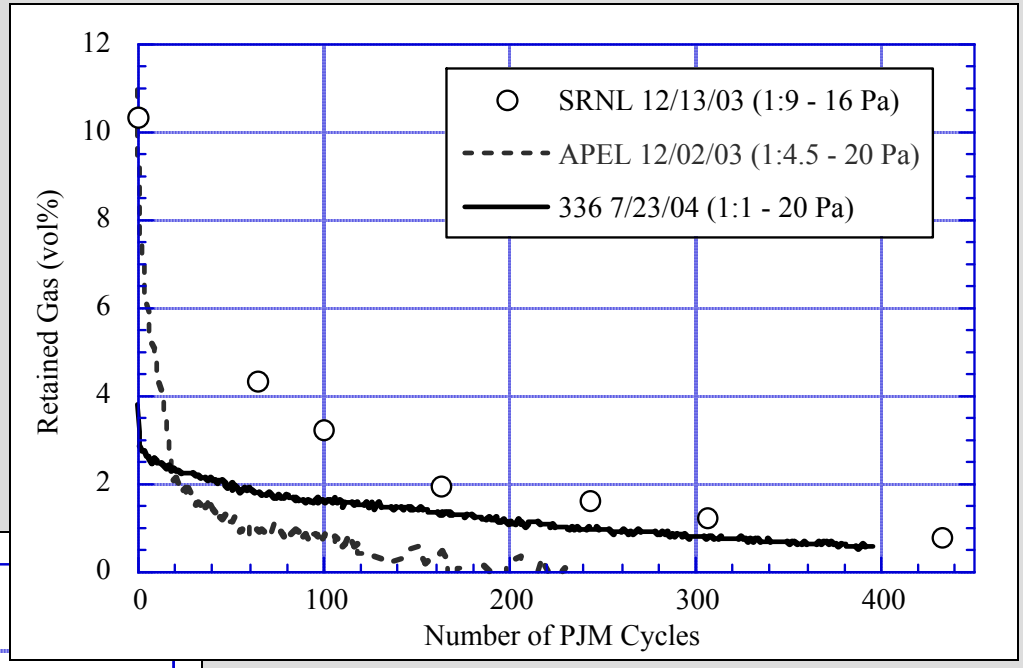
Scaling Data Comparisons

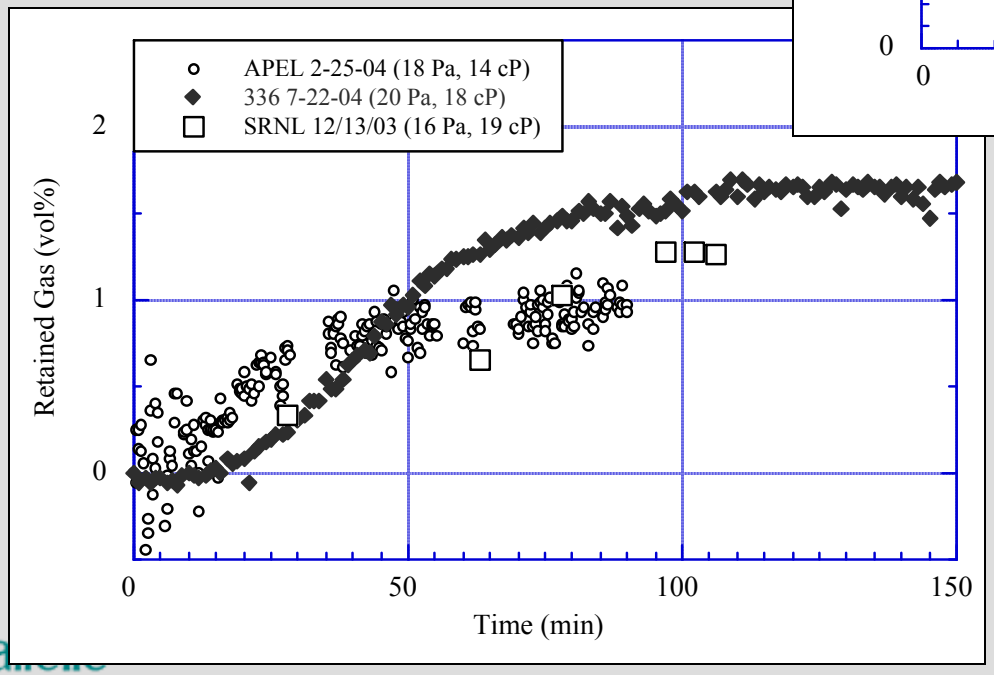




Comparison of surface breakthrough velocity for tanks of 3 different scales with Laponite & clay simulants.

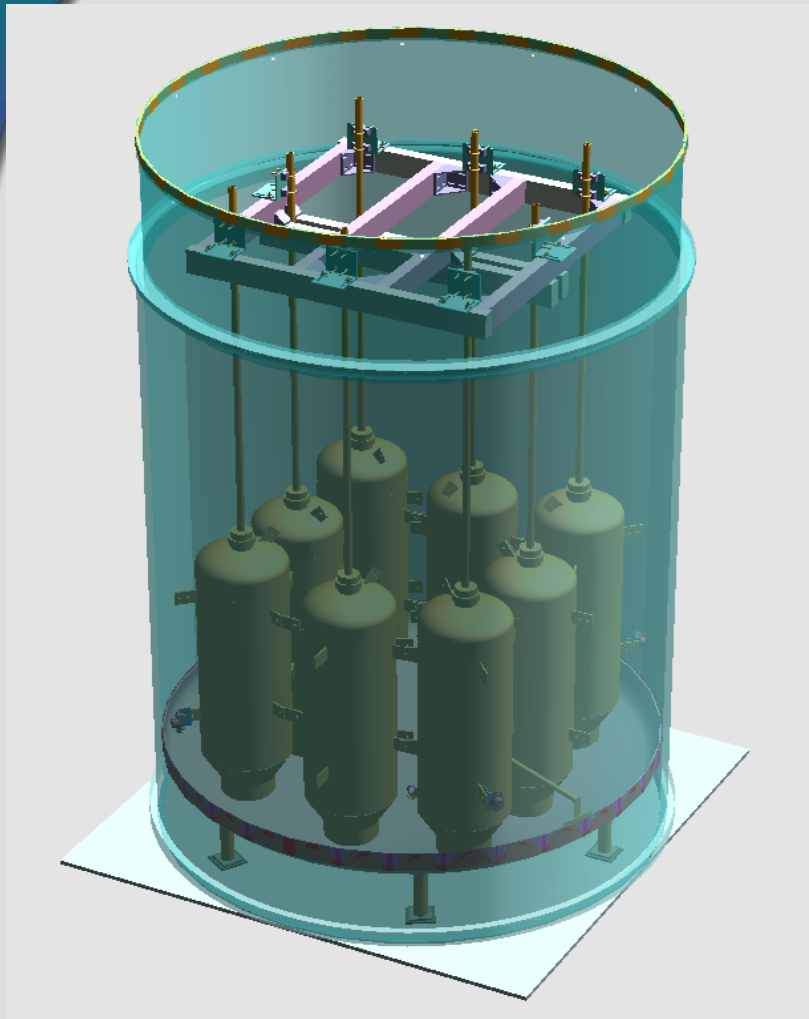
Gas hold-up & release- tests at 3 scales

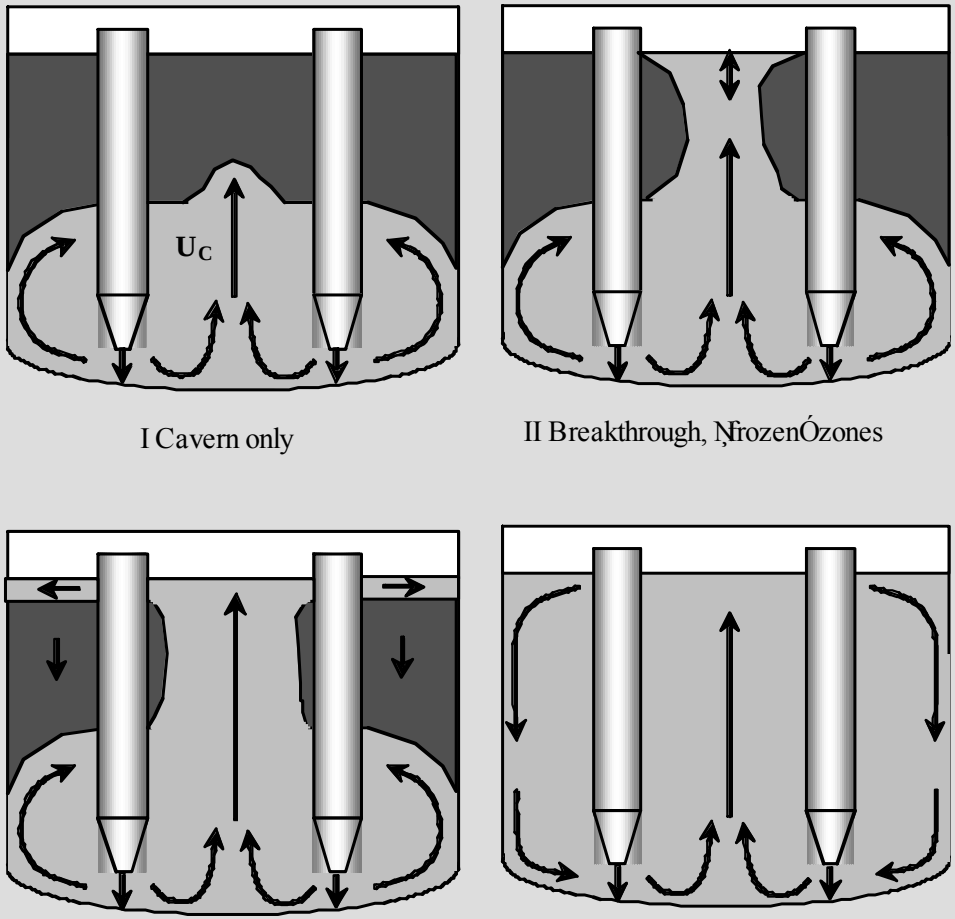




A.5.41

Baseline Designs





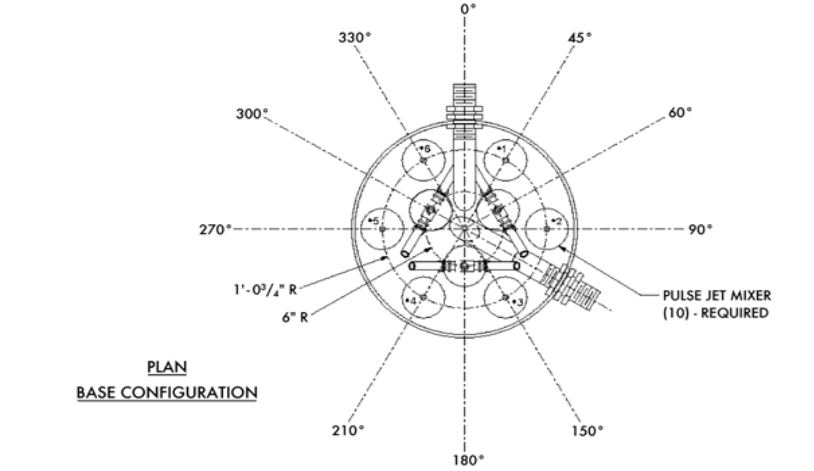
I Cavern only

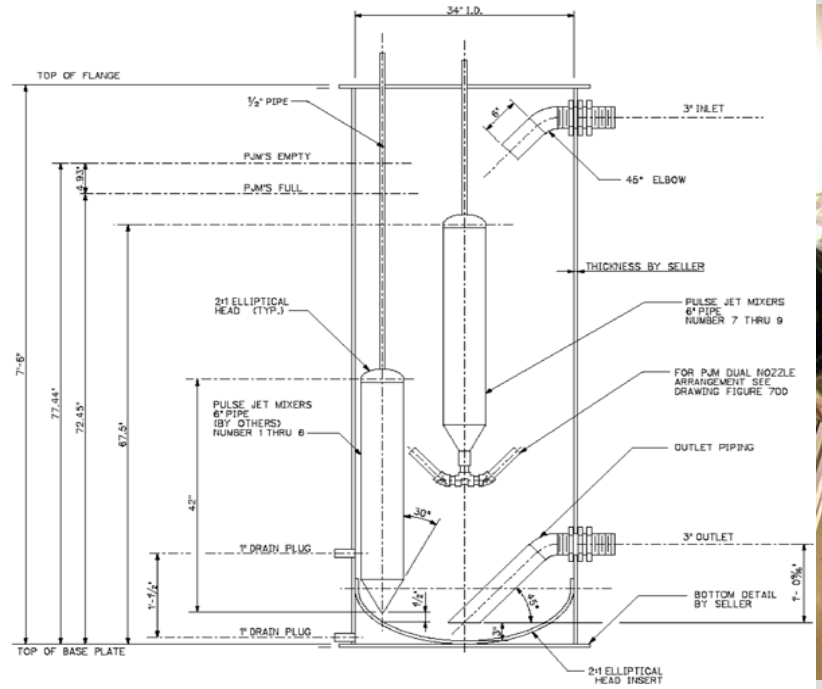
II Breakthrough, Ñfrozen Ózones

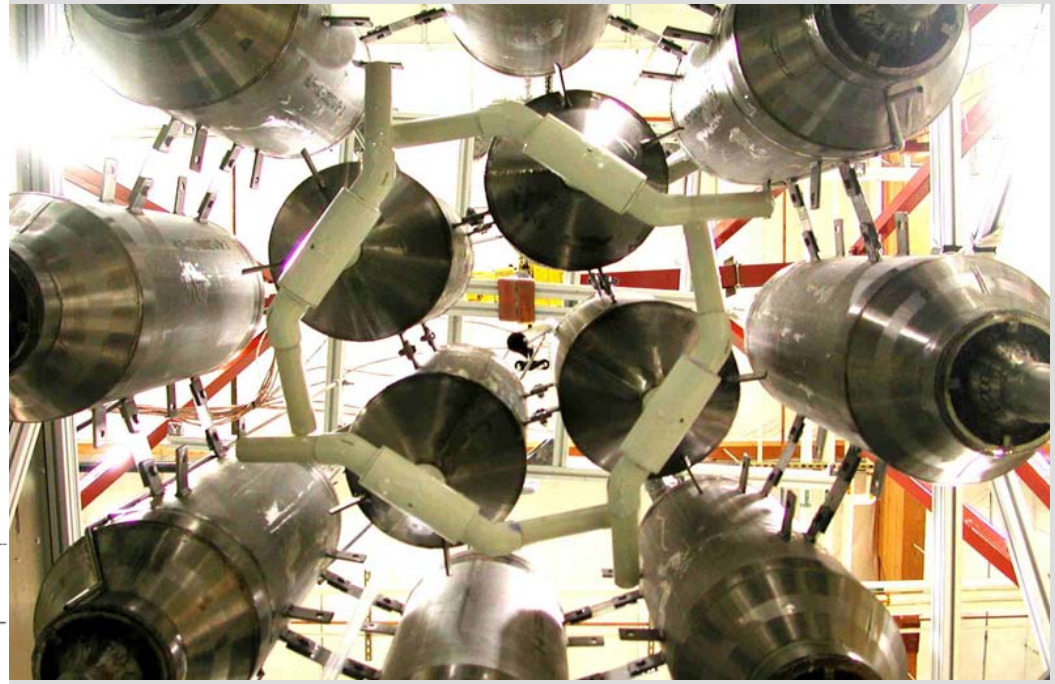
III Breakthrough with slow peripheral movement

IV Full turbulent mixing

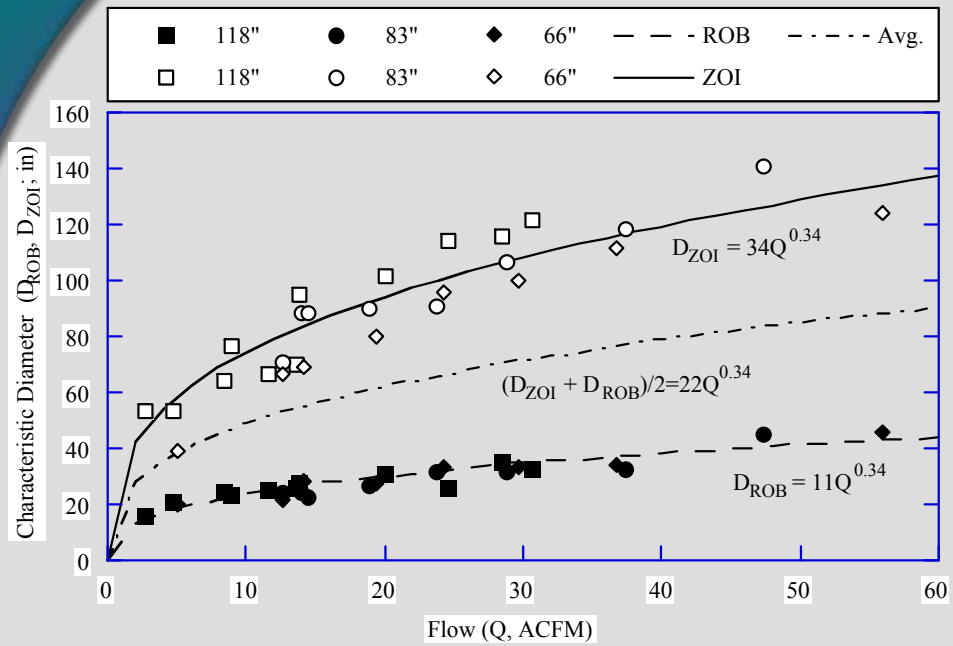
Improved PJM designs

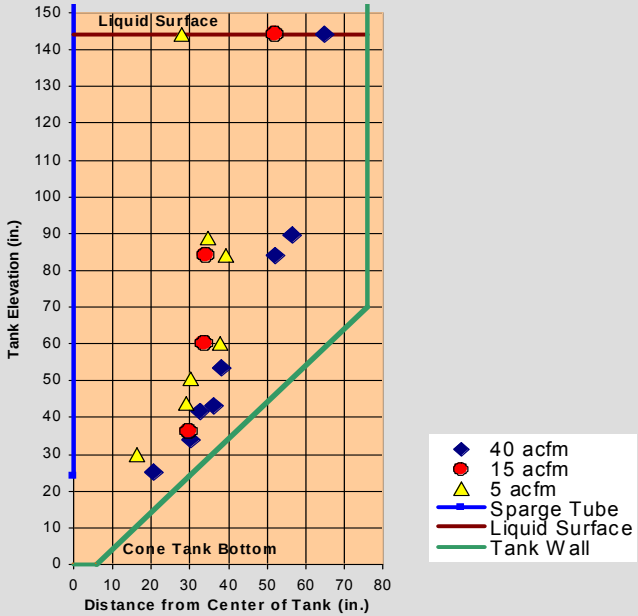


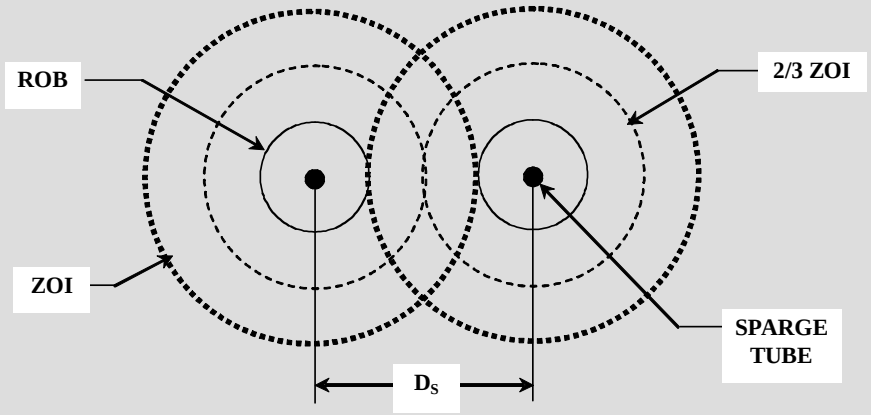


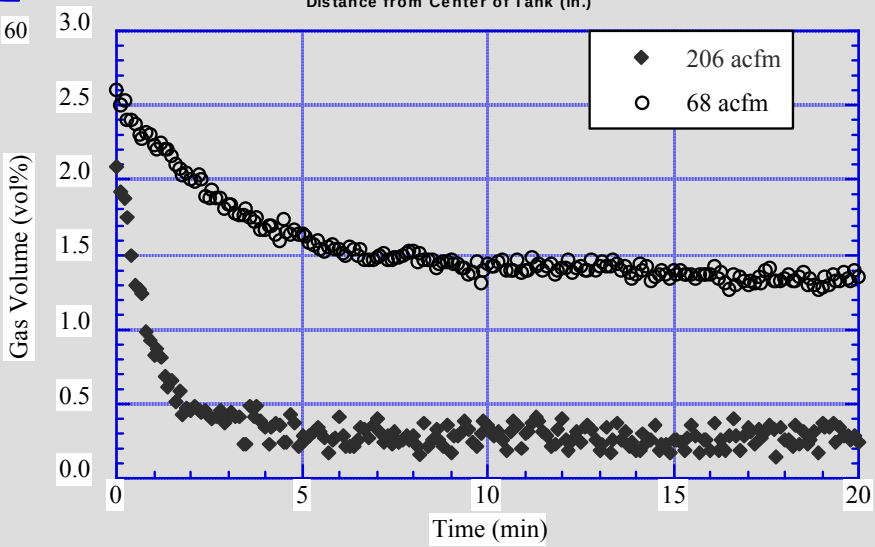


Air sparging in Bingham Plastic Slurry

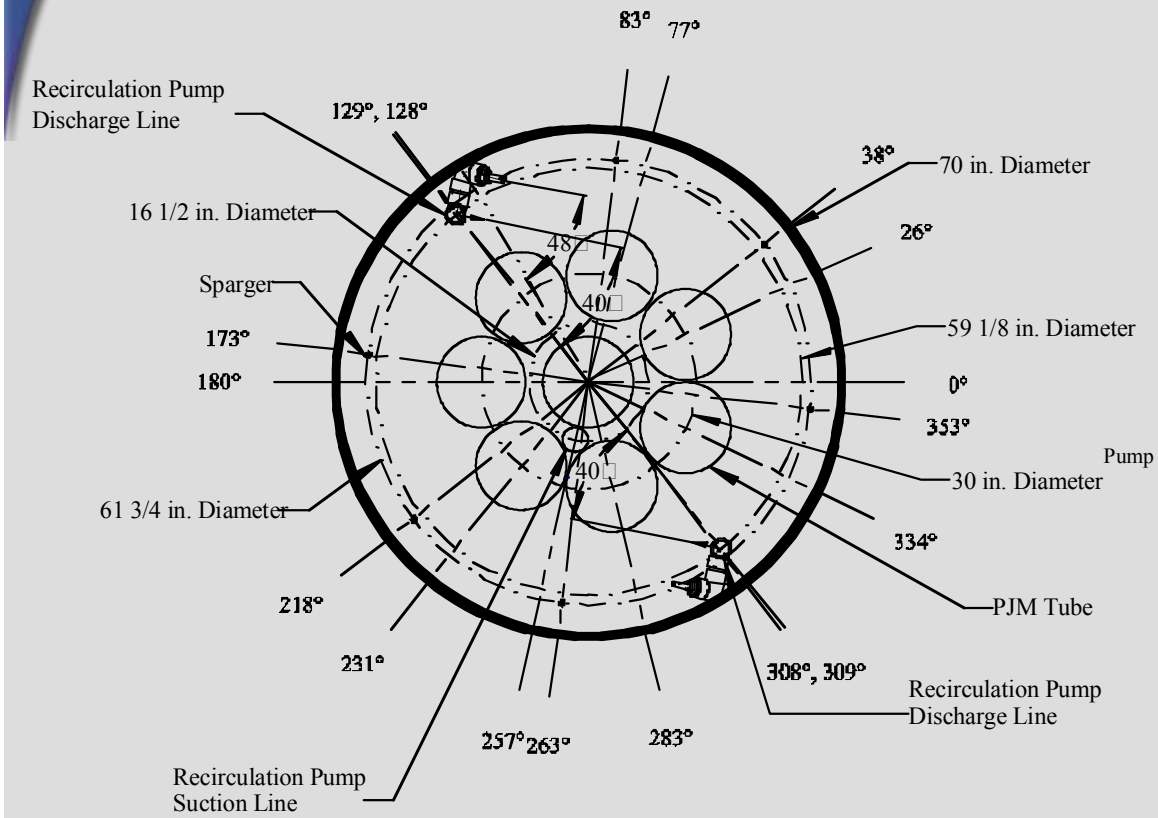


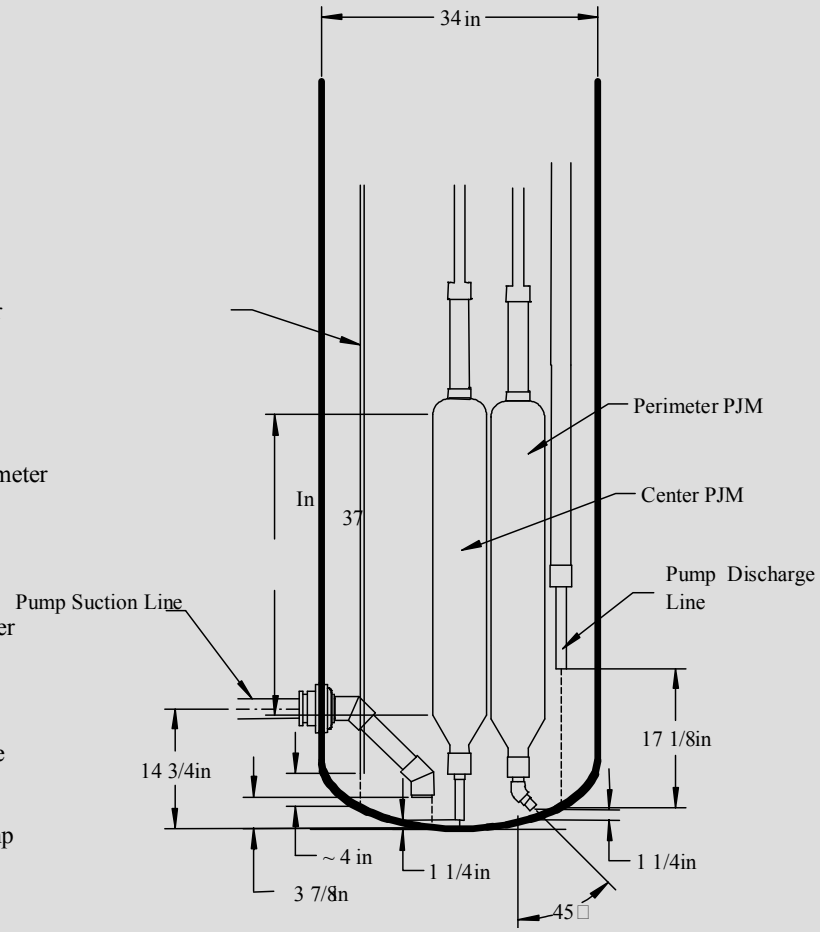






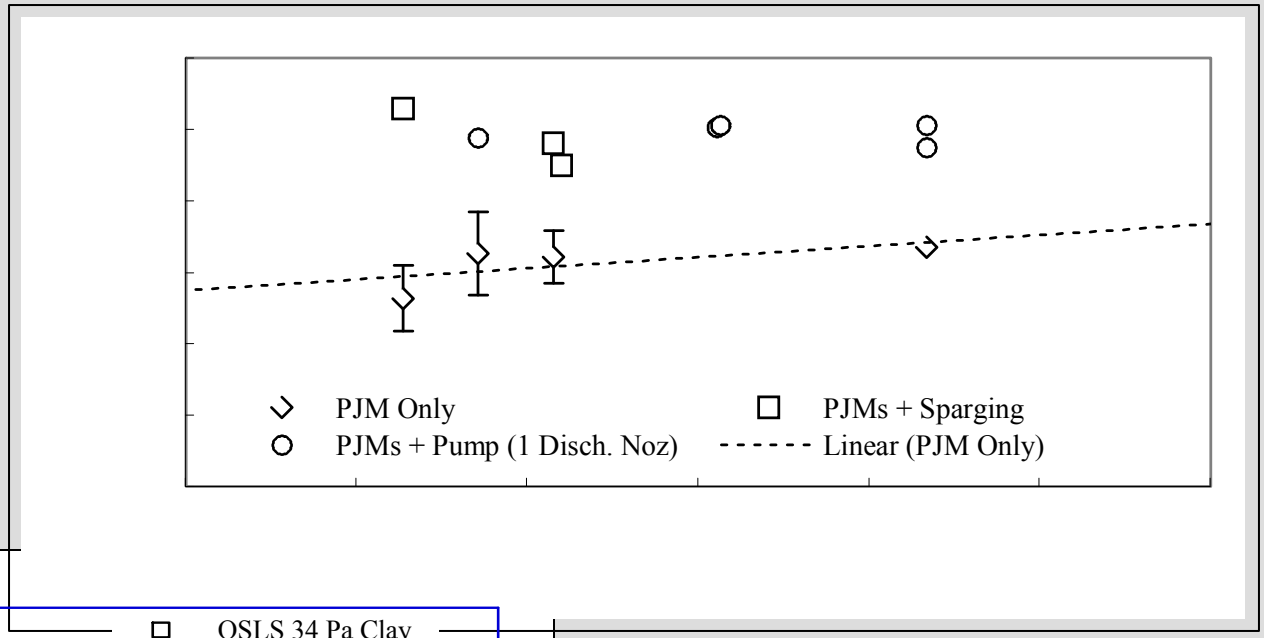
PJM/air-sparge hybrid designs

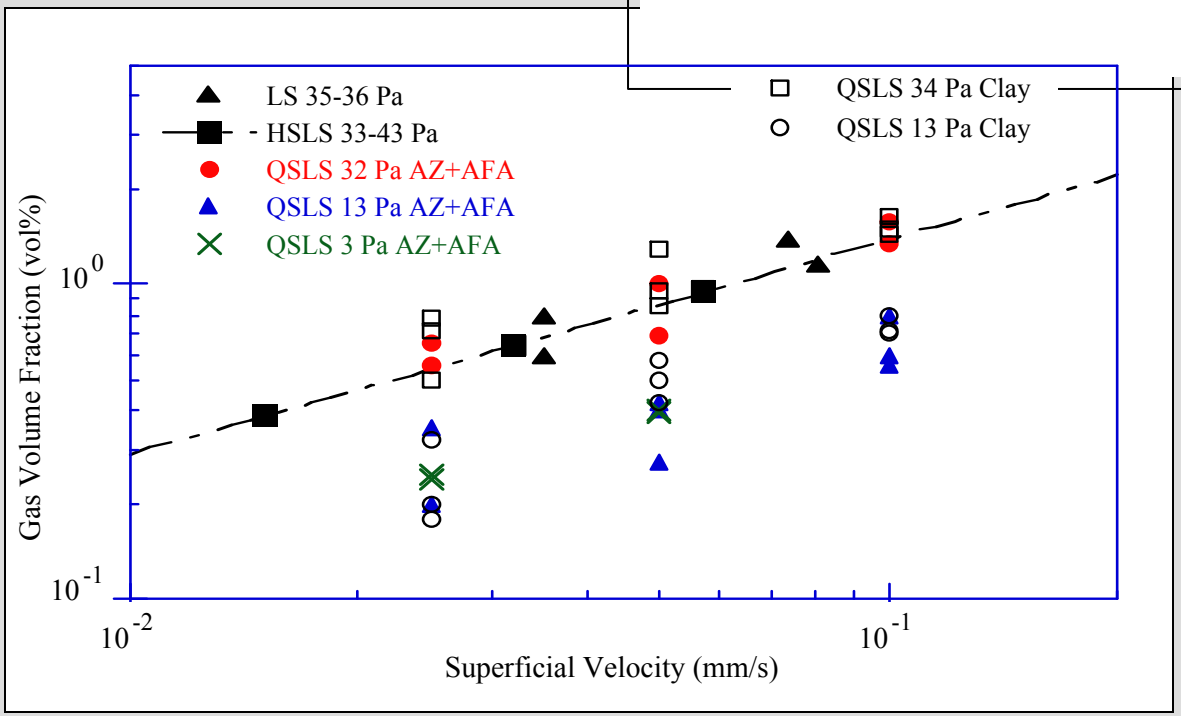




A.5.45

Final Design Mixing Performance





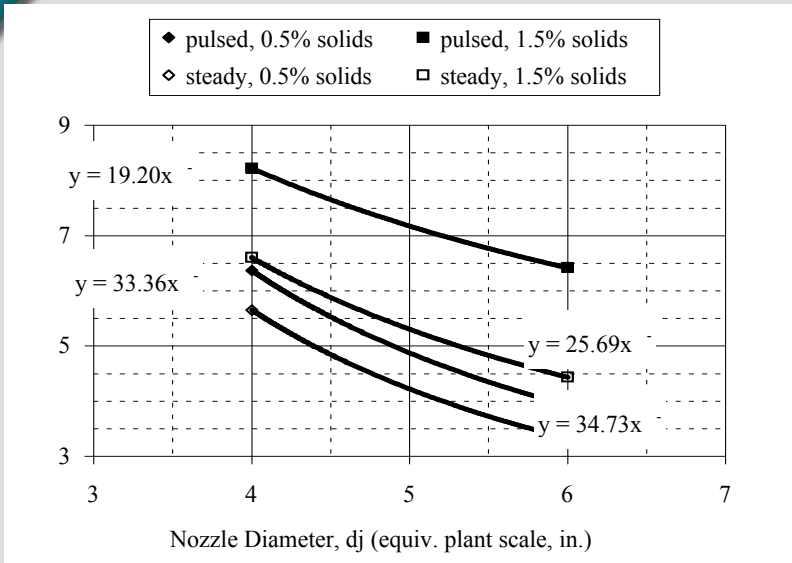
M3- Rating WTP Mixing Systems

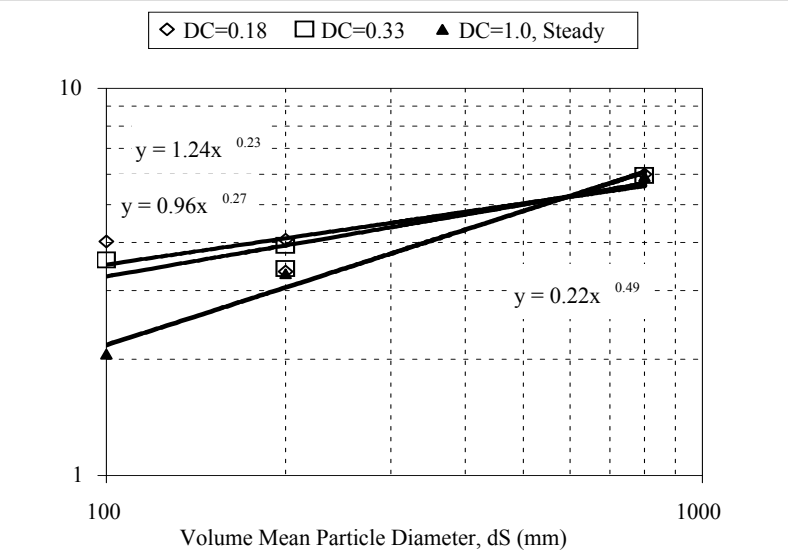
- Rate mixing system designs for balance of WTP vessels
  - Normal operations & mixing restart
- Broad range of potential waste conditions
  - Non-cohesive (settling) solids - cohesive solids
  - Wide range of solids size, density, slurry rheology
- 18 different vessel/mixing system geometries
- Primary metrics
  - Off-bottom suspension
  - Vertical solids distribution
  - Blend times
- Work in 3 phases: non-cohesive, cohesive, gas handling

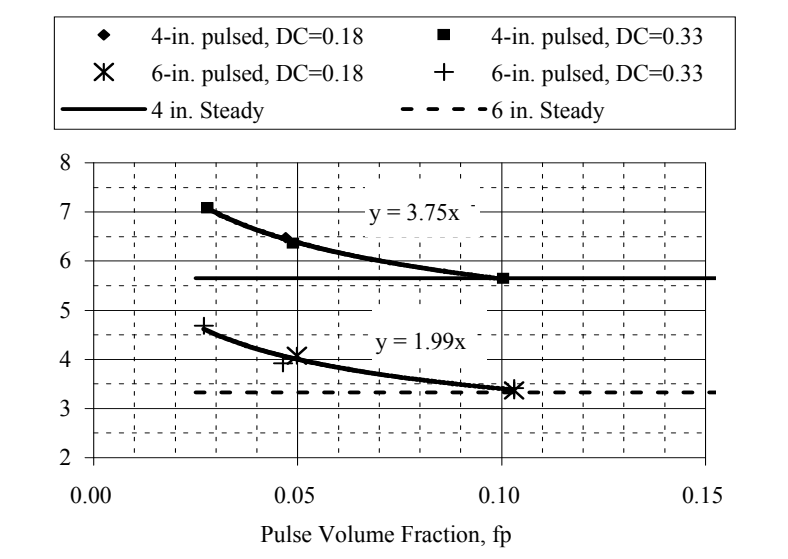
Preliminary tests with non-cohesive solids

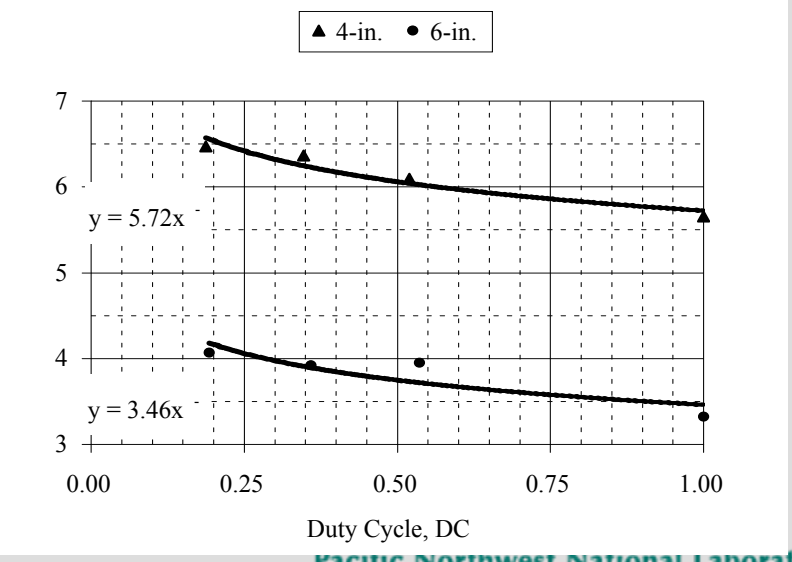
- Simulants
  - Glass spheres (low grade), S ~ 2.47
  - 3 sizes: d_s = 63-100, 150-210, 600-800μm
  - 2 solids loadings: ϕ_s = 0.005 & 0.015
- Vessel geometries
  - 34-in., 1/13.4-scale of HLP-22
  - 12 tubes, 0.3 & 0.45-in nozzles (4 & 6-in. full scale)
- Operational
  - Pulse volume fraction ϕ_p = 0.025 - 0.10
  - Duty cycle: DC = 0.18, 0.36, 0.5, 1 (steady)
- Measurements
  - Ujs & peak cloud height

Some off-bottom suspension results









A.5.49

Preliminary data for information only

Correlating just-suspended velocity

- Assume Zwietering values for un-tested parameters

$$Ucs = k(H/D')^{0.14} g^{0.5} (s-1)^{0.43} (D')^{1.3}$$

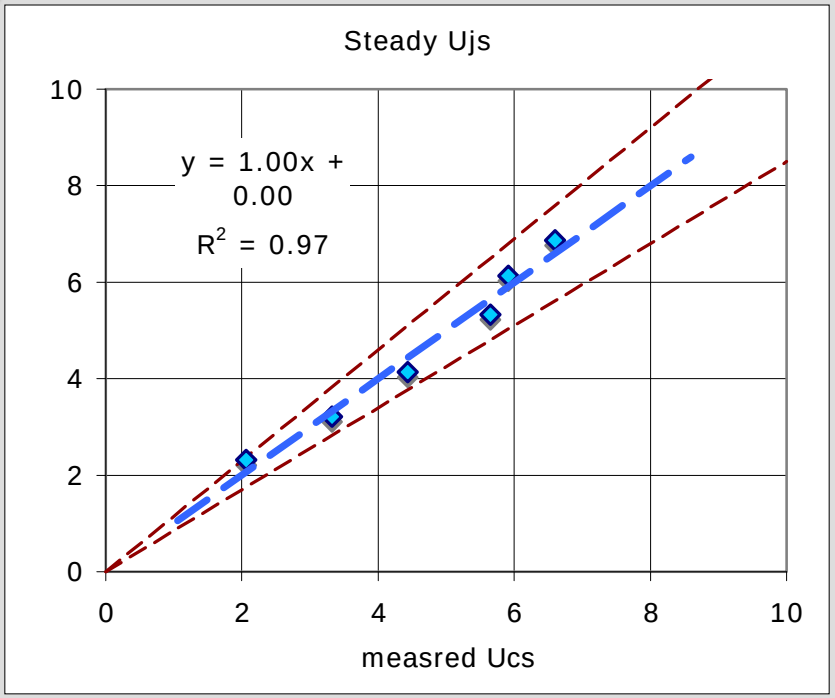
$$\times \quad (d_s)^{a_5} (d_j)^{a_6} (100 s\phi_s)^{a_7} (DC)^{a_8} (\phi_p/(1+\phi_p))^{a_9}$$

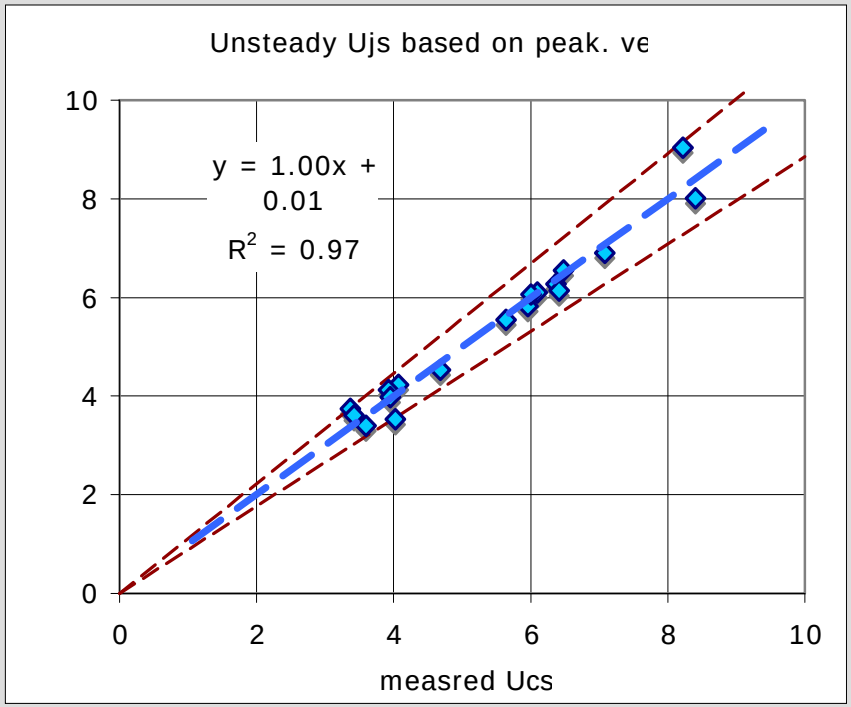
$$D' = D/\sqrt{N}$$

	Steady	Pulsed
k	0.78	0.23
d_s	0.47	0.26
d_j	-1.3	-1.06
$S\phi_s$	0.23	0.34
DC	-	-0.06
ϕ_p	-	-0.18

Preliminary data for information only

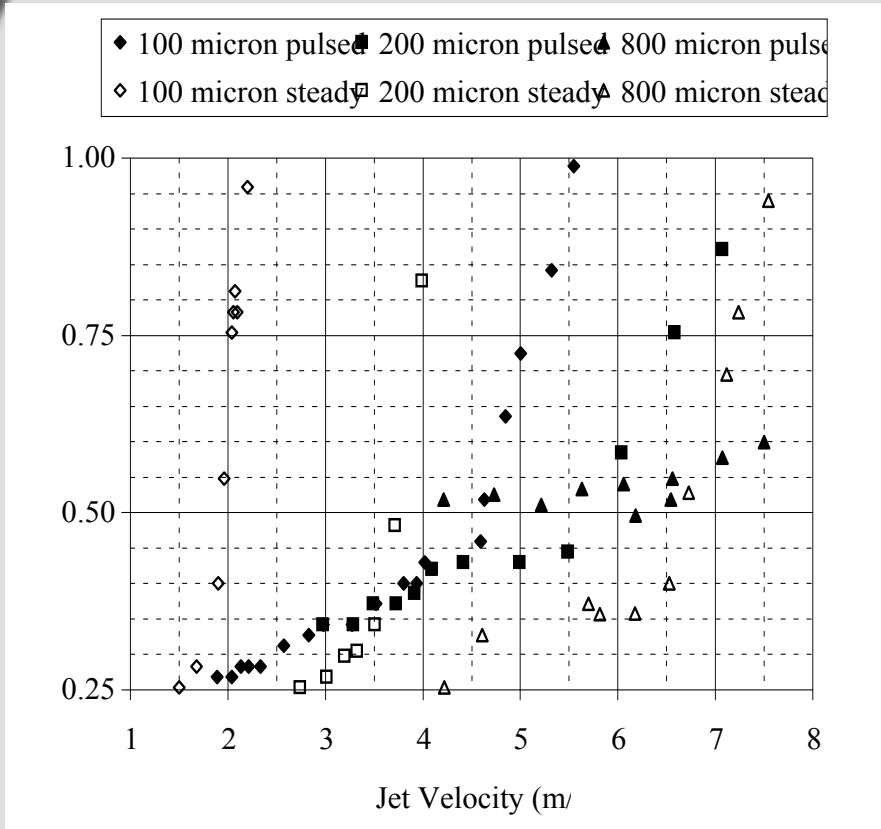
Data correlation: off-bottom suspension

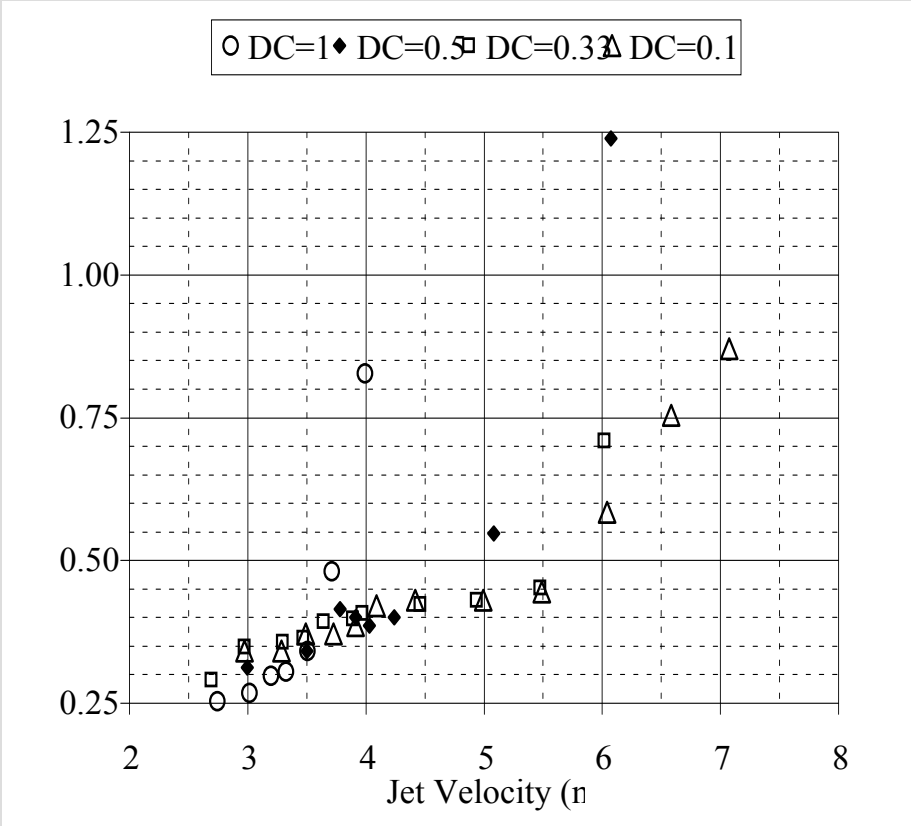




- Suggests pulsation effects small at low concentration
- Scale-up to plant conditions: design likely inadequate
  - More testing at additional scales & higher solids required

Cloud height data





A.5.52

Preliminary data for information only

Correlating cloud-height

- Simple energy argument
  - Energy per pulse ~ change in potential energy of solids

$$\phi_p F_H \sim \frac{\phi_s}{\phi_d} \qquad F_H = \frac{u^2}{2(s-1)gH_c}$$

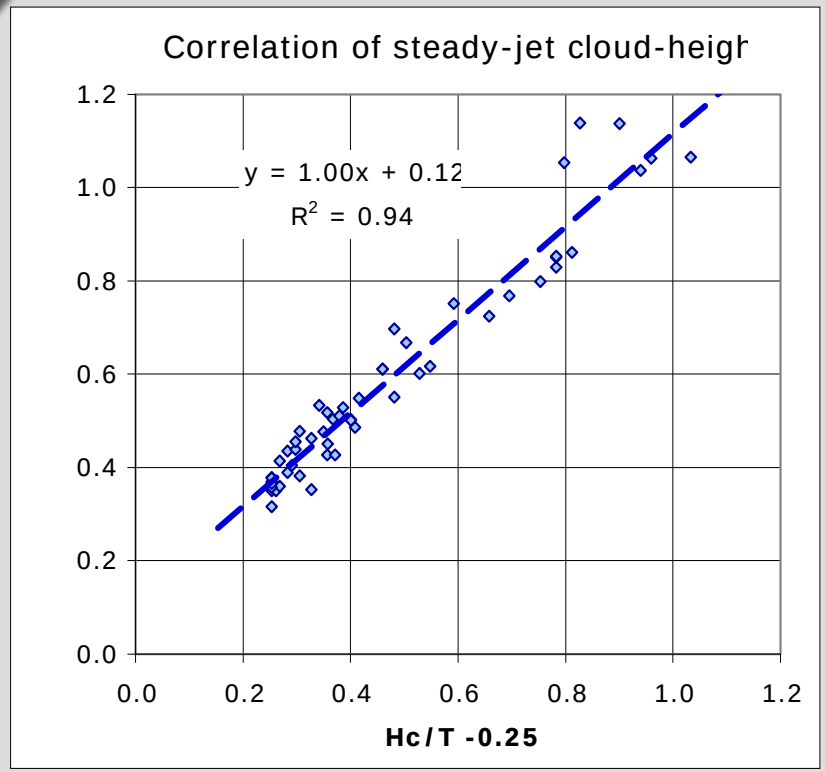
$$\frac{H_C}{D} \sim F_D \frac{\phi_p \phi_d}{\phi_s} \qquad F_D = \frac{u^2}{2(s-1)gD}$$

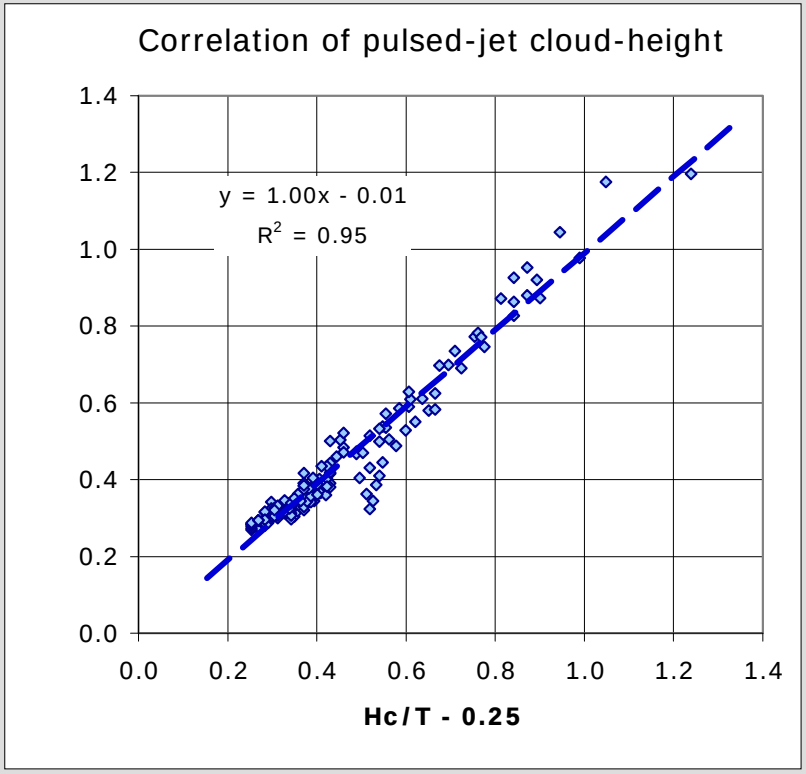
- Attempt correlation of the form

$$\frac{H_C}{D} \sim F_D^{a1} \phi_p^{a2} \phi_d^{a3} \phi_s^{a4} \qquad \text{include} \quad d_s/D \quad \text{or} \quad u_s/u$$

A.5.53

Correlation of cloud-height data





	k	U	ϕ_s	ϕ_d	d_s/T	ϕ_p	DC
Steady	2.8	2	-0.56	1.7	-1.1	-	-
Pulsed	7.1	2	-1.1	1.0	-0.5	0.3	0.25

Preliminary data for information only

Summary of findings

- Just suspended velocity
  - Unsteady effects minor
    - DC effects negligible
    - There is evidence this breaks down at higher concentration where time to suspend > drive time
  - Similar solids size effect
  - Concentration exponent 2x
  - Effect of nozzle size as expected
  - To be sure, need more data

Summary, cont.

- Vertical distribution
  - Strong bulk density stratification effect
  - Unsteady effects appear to dominate
    - Exponents on DC & PVF
    - Fundamental behavior
  - Weak solids size dependence:
    - Define U_{JH} ("just to H…"). Then $U_{CH} \sim d_s^{0.25}$
  - Strong concentration effect: $U_{CH} \sim \phi_s^{0.5}$
  - Strong pulsation effect: $U_{CH} \sim \phi_p^{-0.5}$

A.5.56

Predicting Slurry Pump Performance in Nuclear Waste Storage Tanks

SRNL™
SAVANNAH RIVER NATIONAL LABORATORY

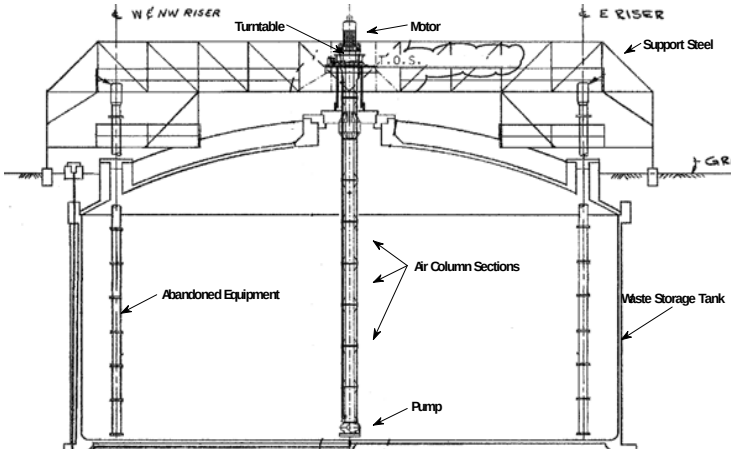
We Put Science To Work

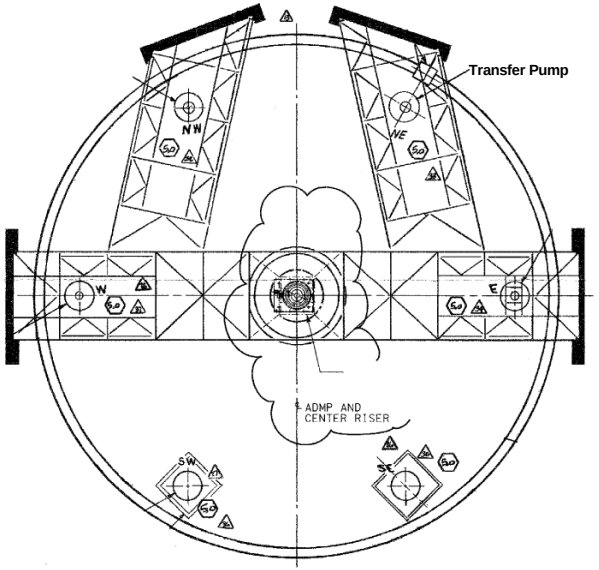
Robert A. Leishear
David B. Stefanko
Si Y. Lee
Richard Dimenna
M. Augeri
M. Hubbard
Process Engineering
1/18/08

ABSTRACT

- **Residual radioactive waste was removed from a waste tank in the F-Area Tank Farm (FTF) at Savannah River Site (SRS), using the advanced design mixer pump (ADMP).**
- **Known as a slurry pump, the ADMP is a 55 foot long pump with an upper motor mounted to a steel super structure**, which spans the top of the waste tank. The motor is connected by a long vertical drive shaft to a centrifugal pump, which is submerged in waste near the tank bottom. The pump mixes, or slurries, the waste within the tank so that it may be transferred out of the tank.
- **The tank is a 1.3 million gallon, 85 foot diameter underground waste storage tank, which has no internal components such as cooling coils or structural supports. The tank contained a residual 47,000 gallons of nuclear waste, consisting of a gelatinous radioactive waste known as sludge and particulate zeolite.** The prediction of the ADMP success was based on nearly thirty years of research and the application of that research to slurry pump technology. Many personnel at SRS and Pacific Northwest National Laboratories (PNNL) have significantly contributed to these efforts.
- This presentation summarizes that research, which is pertinent to the ADMP performance. In particular, **a computational fluid dynamics (CFD) model was applied to predict the performance of the ADMP in a waste tank.**
- **Essentially, this presentation consists of a brief summary of several publications for the American Society of Mechanical Engineers, 2004, Fluids / Heat Transfer Conference.** Each of the papers, Parts I – IV, discuss modeling, testing, and the historical performance of slurry pumps, which were needed to predict the FTF results.
- **The CFD model results and the experimental validation of those results will be published in the January, 2008, ASME Journal of Fluids Engineering.**

ADMP Tank Installation





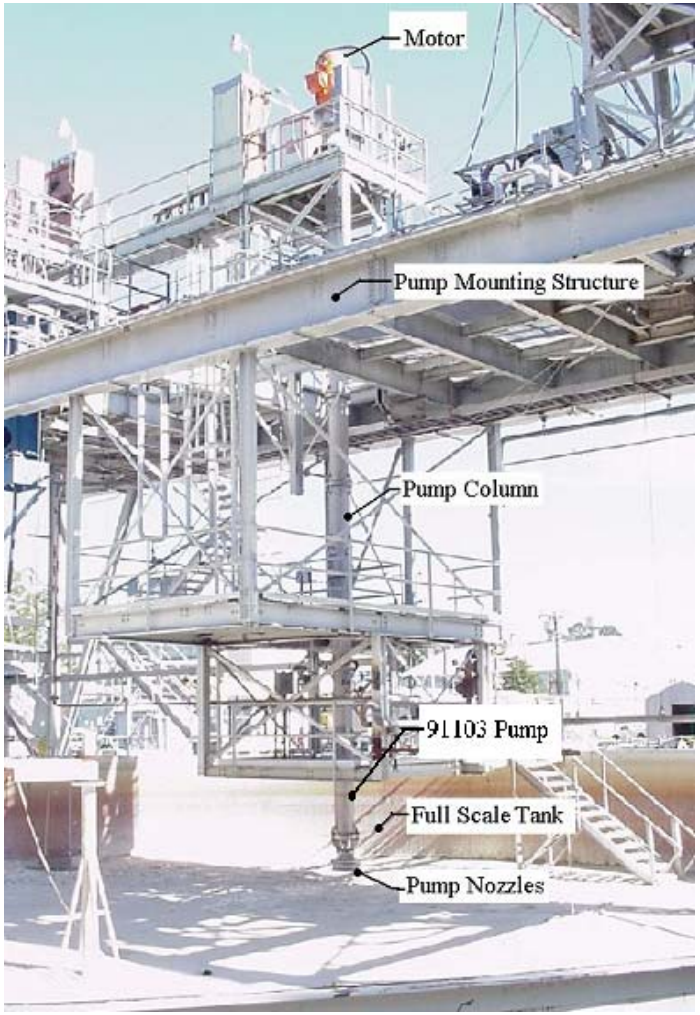
- Single pump installed in the center of an 85 feet diameter, 33 feet tall, underground waste storage tank.
- No internal tank obstructions

A.5.59

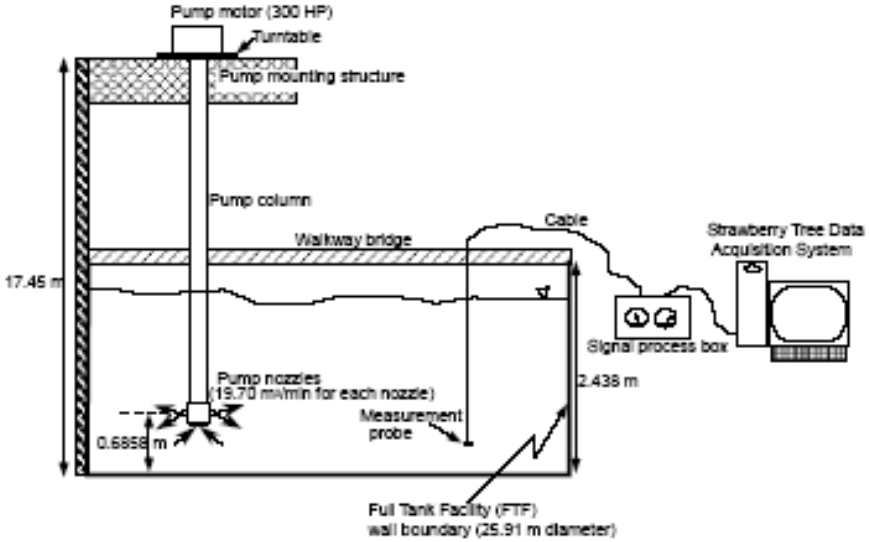
ADMP, Advanced Design Mixing Pump

SRNL

Test Facility



- Pump installed off center in a full scale, 85 feet diameter, 8 feet deep tank.
- Rotating walkway permitted velocity measurements anywhere in the tank



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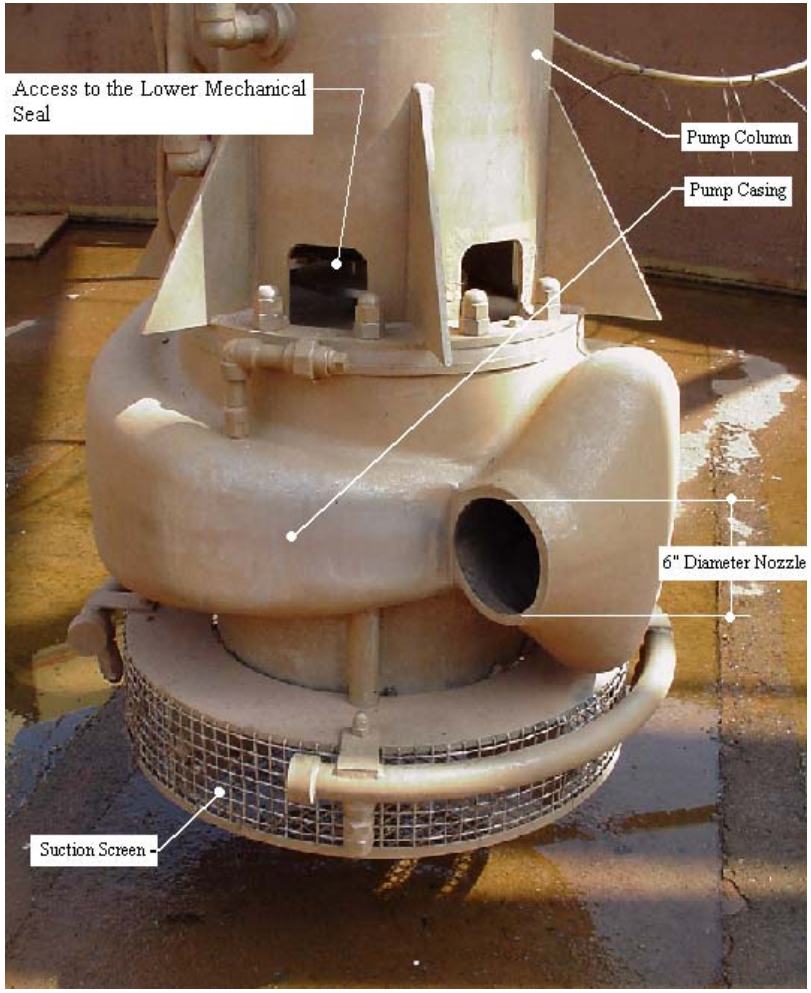
ADMP Construction



- ADMP = 10400 gpm pump
- Rotates at 1/5 – ½ rpm
- Other slurry / mixing pumps at SRS
  - Quads = 5200 gpm
  - Standards = 1200 gpm

A.5.62

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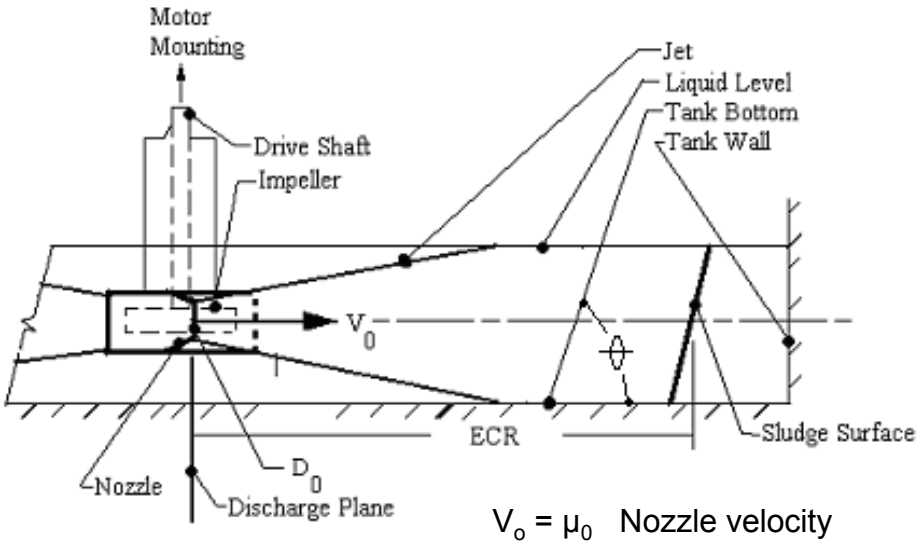
Sludge mixing, Churnetski



$V_o = \mu_0$ Nozzle velocity
D_0 Nozzle diameter
ρ Density
τ Yield stress

- **Predicted by the effective cleaning radius (ECR), which is derived from shear equations.**

$$ECR = 0.40 \cdot D_0 \cdot V_0 \cdot \sqrt{\frac{\rho}{\tau}} \cdot \left(100 \frac{cm}{m}\right)$$

- **Additional erosion effects not modeled**
- **SRS "Rule of thumb"**
  - **3 ft/sec minimum velocity needed to suspend sludge**
  - **1 ft/sec average tank velocity needed to keep sludge suspended**

A.5.63

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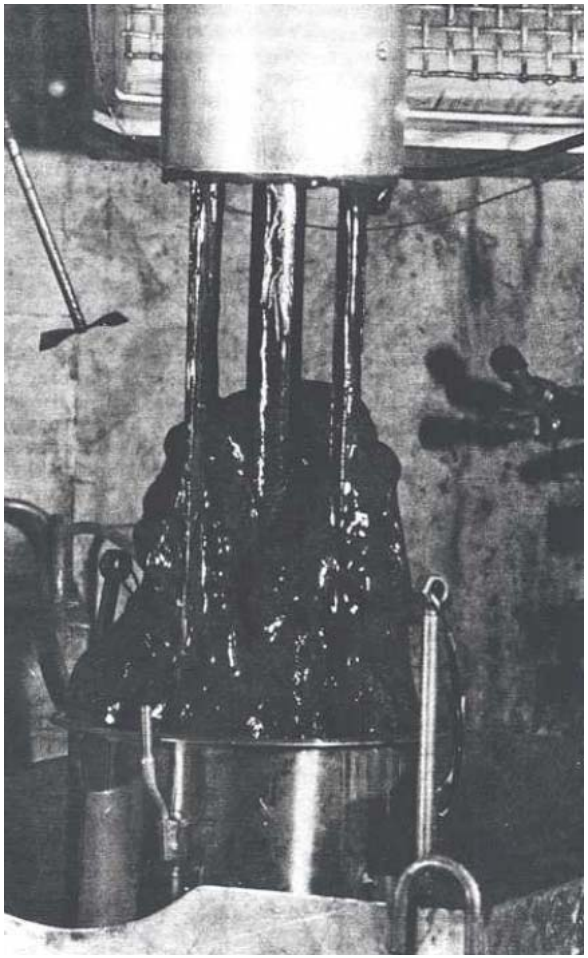
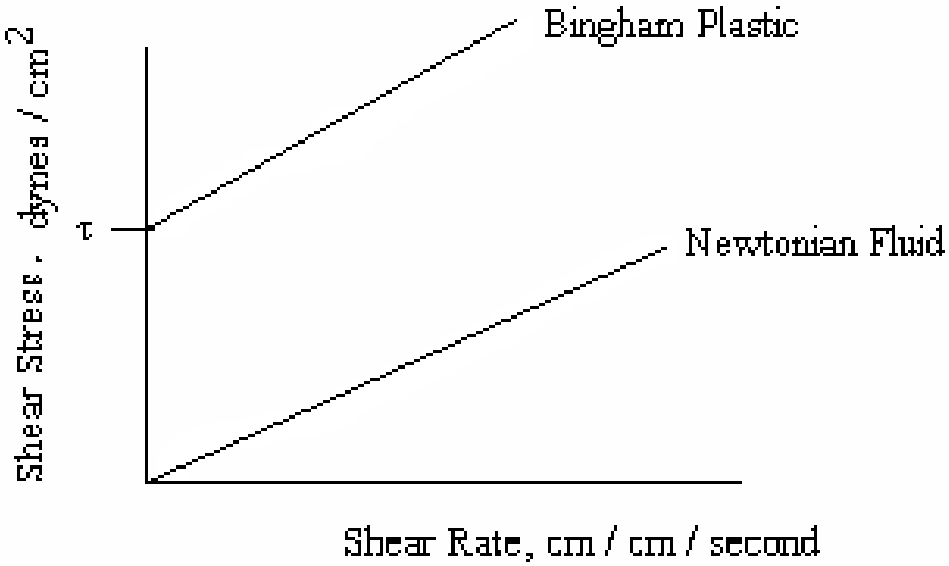
Tank Waste

- **Sludge:**
  - **$NaNO_3$, $NaNO_2$, $NaAlO_2$, Na_2CO_3, and Na_2SO_4**
  - **Radioactive and stable fission products (< 1% each)**
  - **$Fe(OH)_3$, $Al(OH)_3$, MnO_2, $CaCO_3$, Zeolite, and SiO_2.**
- **Zeolite**
  - **Porous, granular alumino-silicate solid, which may have its interstitial voids filled with large unattached molecules or water**

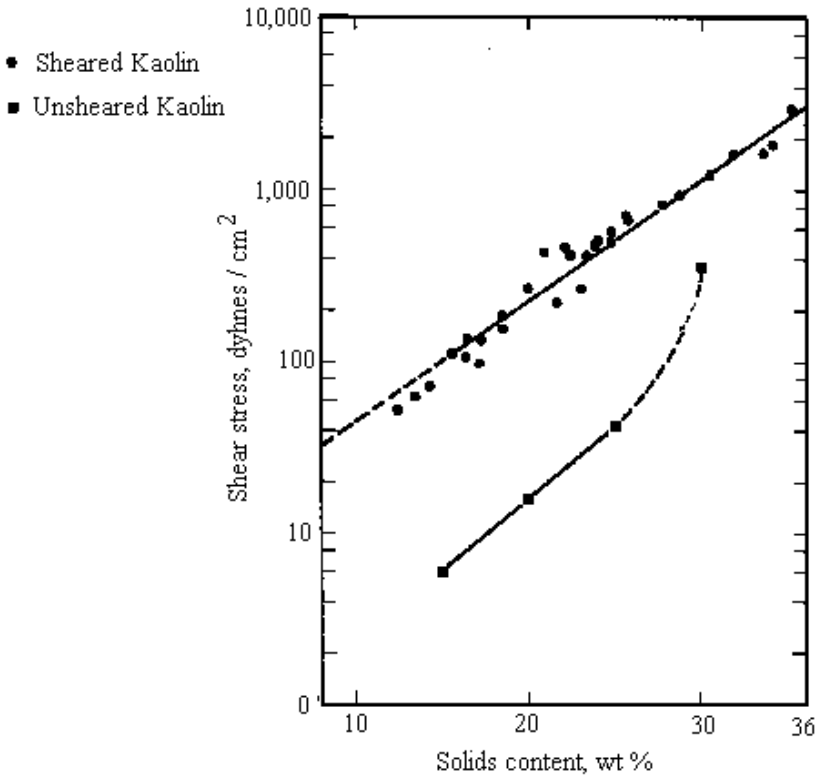
Sludge / Bingham Plastic



Stone



Kaolin Modeling



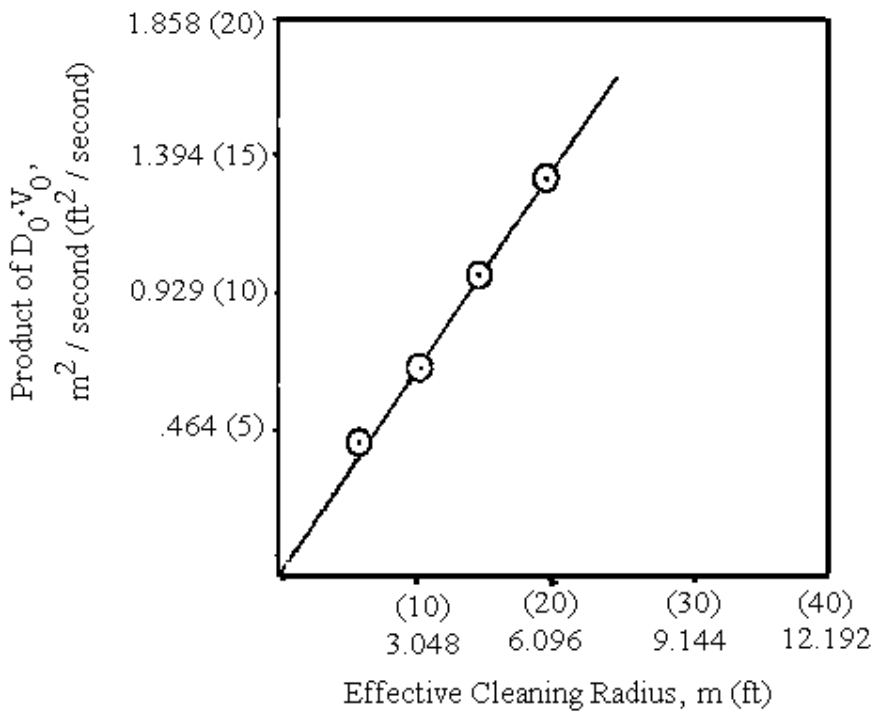
Selby

- Material properties similar to sludge
- Mixing breaks up the Kaolin platelets and increases the yield stress up to 30 hours of mixing
- Indexed pump increased the ECR by ≈ 3 % at the test facility for a free jet away from the wall

Hansen

A.5.66

Kaolin Test Results



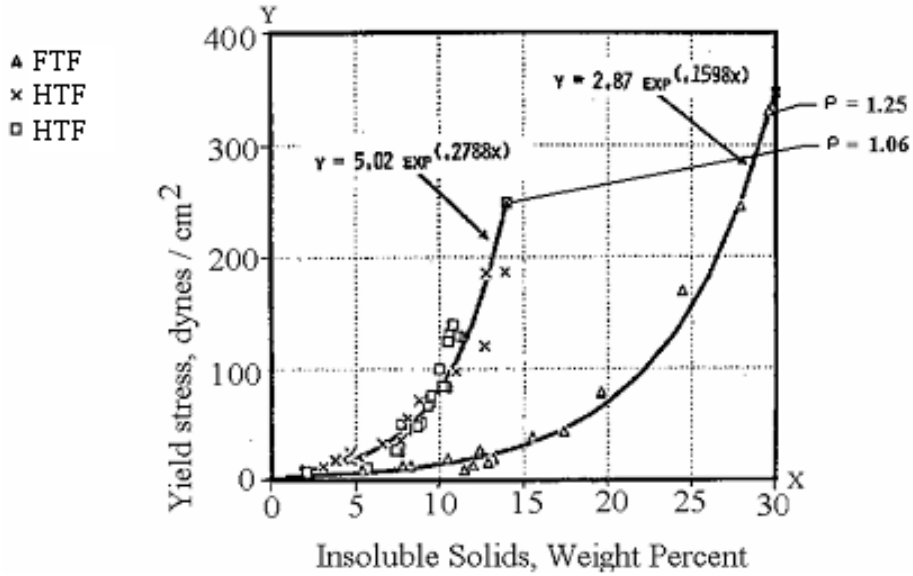
Churnetski

- **The effective cleaning radius is proportional to the pump discharge diameter and velocity**
- **Demonstrated with Kaolin tests**

A.5.67

SRNL

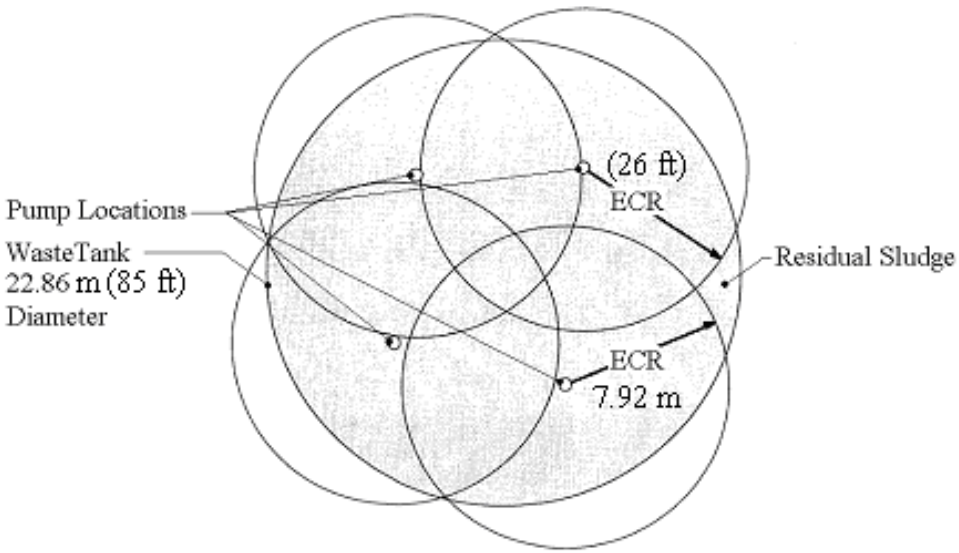
Sludge Properties, Hamm



- Worst case sludge properties assumed
- Bounding sample was allowed to settle for a year

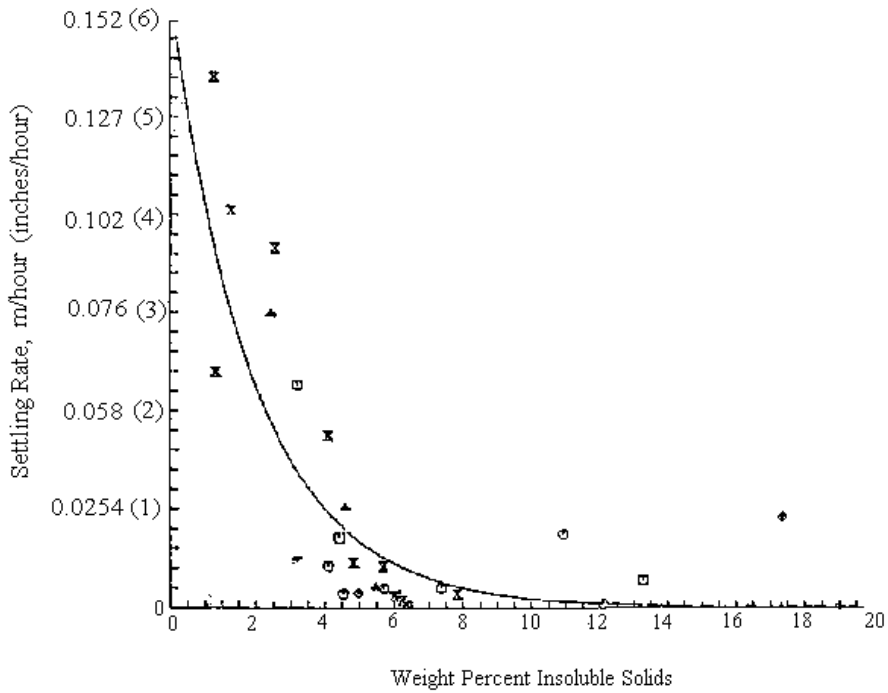
A.5.68

Historical Results



- Previous FTF facility results
  - ECR accurately predicted residual waste in a tank, which contained dried, solid, sludge and installed cooling coils
  - Indexing the pump removed additional waste
  - Historical rheological data available for the figure shown
  - Calculated a 2.27 feet / second minimum velocity to suspend waste
- Rheological data unavailable for the case considered here
  - Historical transfer data was similar for both cases
- Material properties assumed to be similar for use in the ECR equation

Suspension



Motyka

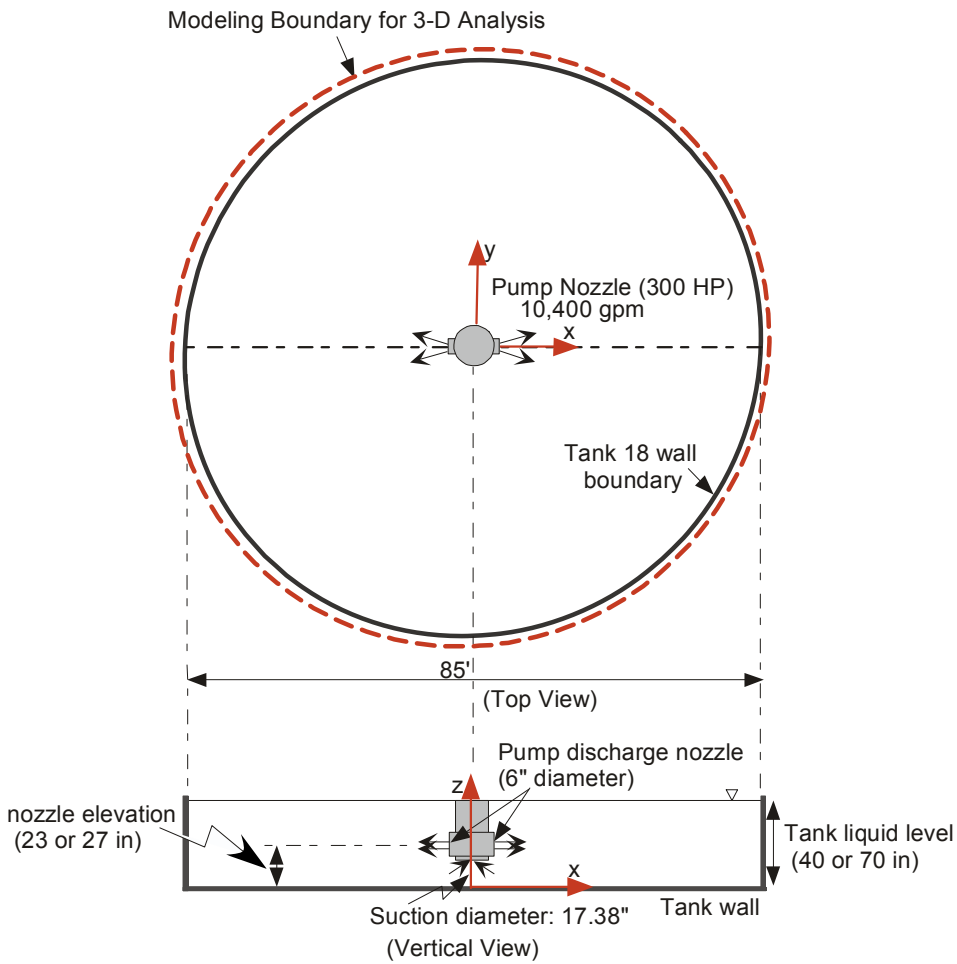
- **Limited data on settling rates of sludge indicated effective mixing**

$$V_s \propto D_p \cdot g_c^2 \cdot \left(\frac{\rho_p - \rho_L}{18 \cdot \mu} \right)$$

- **Operations experience indicated that sludge settling was not expected to be a problem**

A.5.70

CFD models



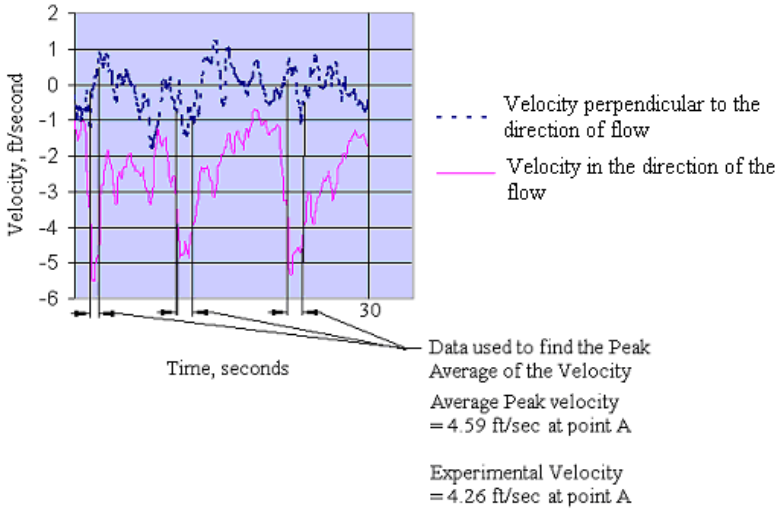
- **Fluent® models**
  - Validated off-center pump model. Single phase fluid,

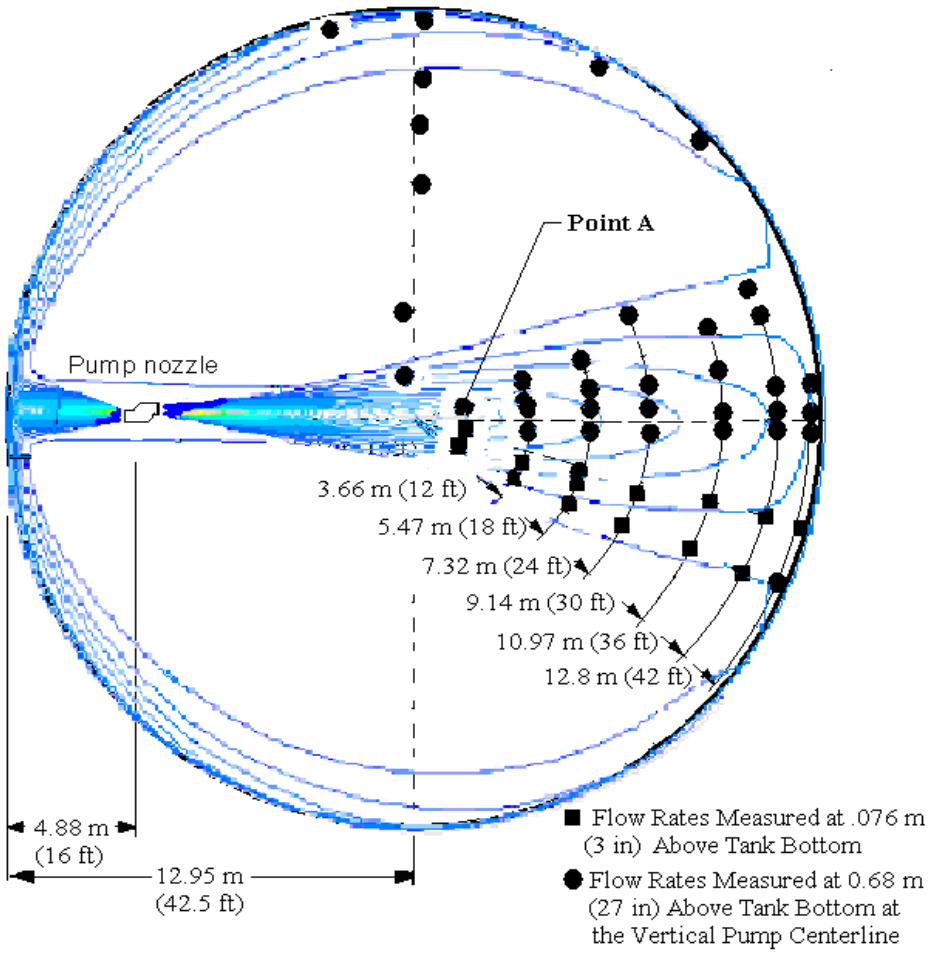
    SpG = 1.0
  - Final central pump model shown. SpG = 1.2
- **260,000 elements were used in the CFD models**
- **Uniform flow at the nozzle exits**
  - Previous research showed that tangential nozzles provide higher flow rates than radial nozzles for identical impellers
  - This work showed that smaller nozzles with equivalent μ_0 D provided higher velocities along the nozzle discharge plane
- **Selection of the κ-Є turbulence model provided better agreement with experiment**

A.5.71

SRNL

Full Scale Testing

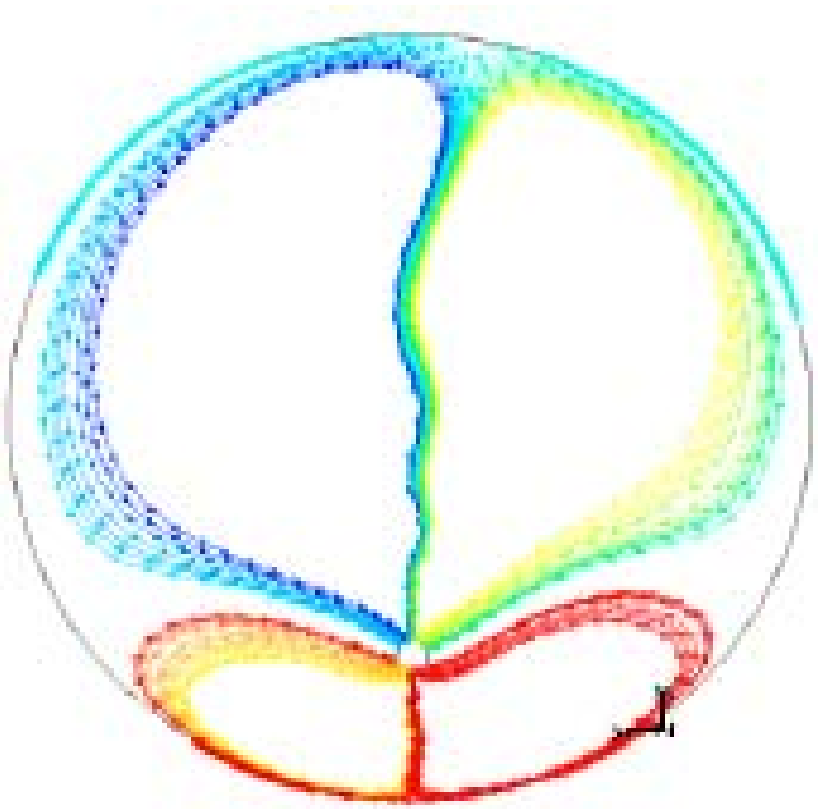




A.5.72

SRNL

Comparison of CFD to experimental results

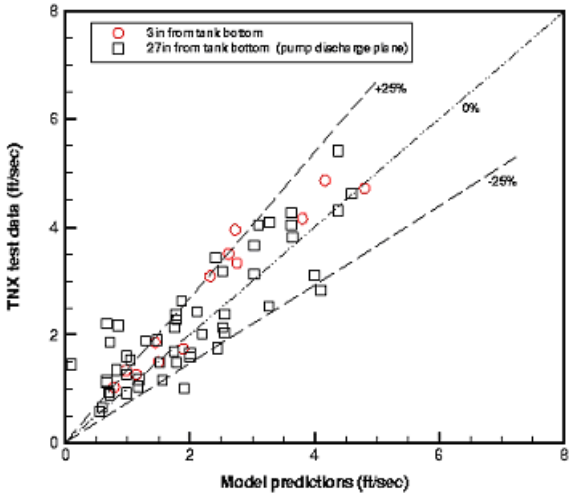


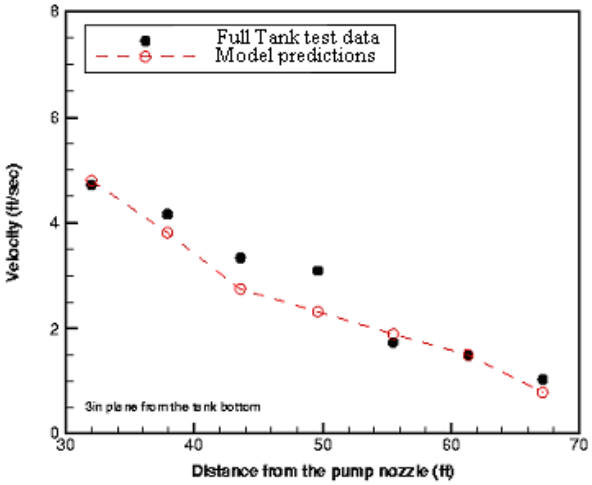
- Plot shown is ten seconds after pump startup
- 2-D model was unacceptable
- 3-D model agreed with observations
- Flow field oscillated with respect to the pump

A.5.73

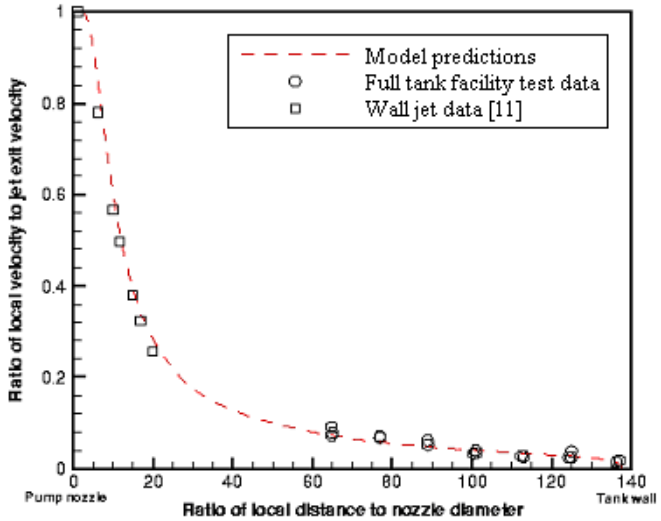
SRNL

Test Data for a Fixed Jet



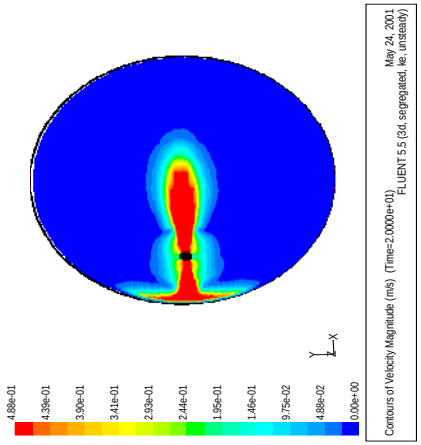


- Experimental results are within 25 % of predictions for a fixed jet impinging on a wall.
- Previous CFD models in the literature were only within 100 – 200 % accuracy



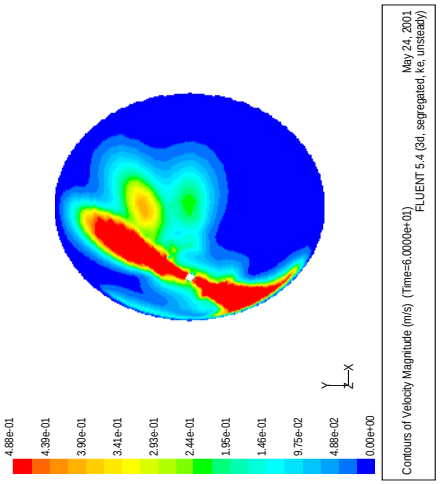
A.5.74

Rotating jet

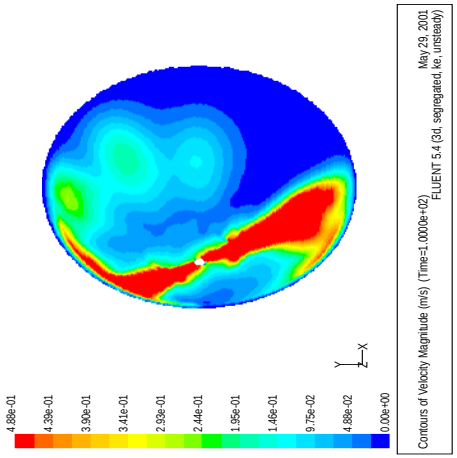


20 seconds

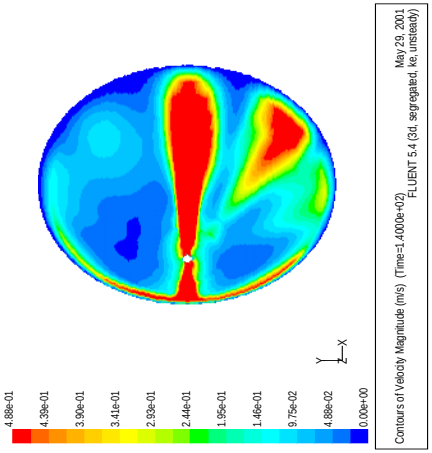
- 30 ° steps in rotation modeled ¼ rpm rotation
- Rotating jet data not measured
- Models look reasonable, qualitatively
- ECR slightly less for a rotating jet than for a fixed jet for a wall jet configuration



60 seconds



100 seconds



140 seconds

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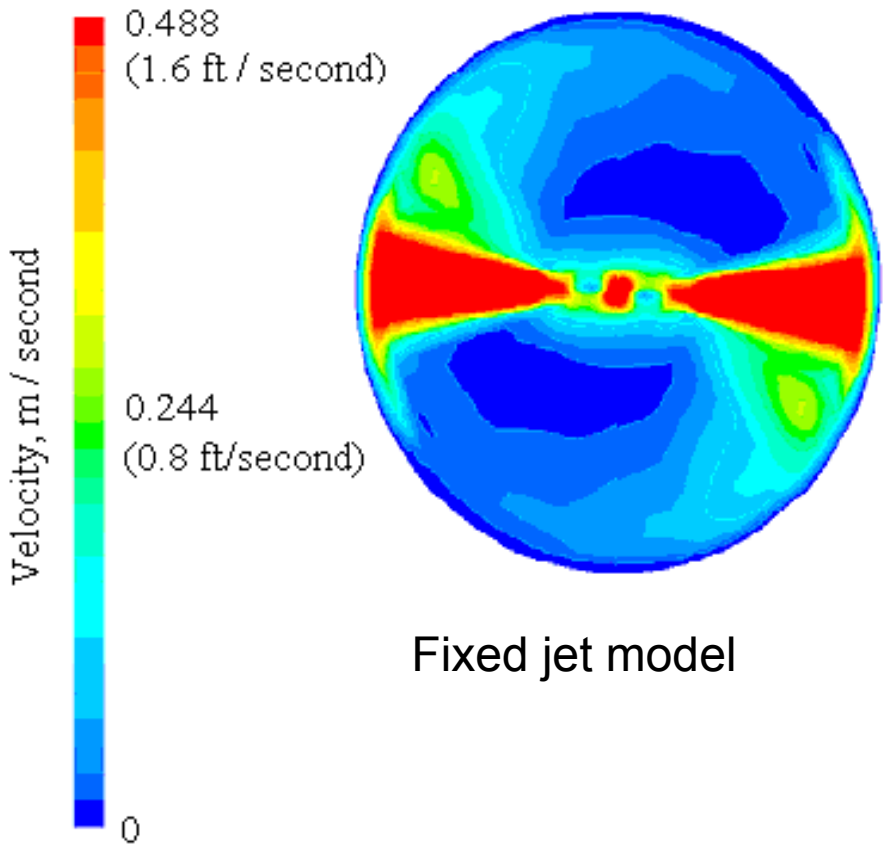
ECR Prediction for Mixing, Leishear, Lee, Stefanko, Dimenna



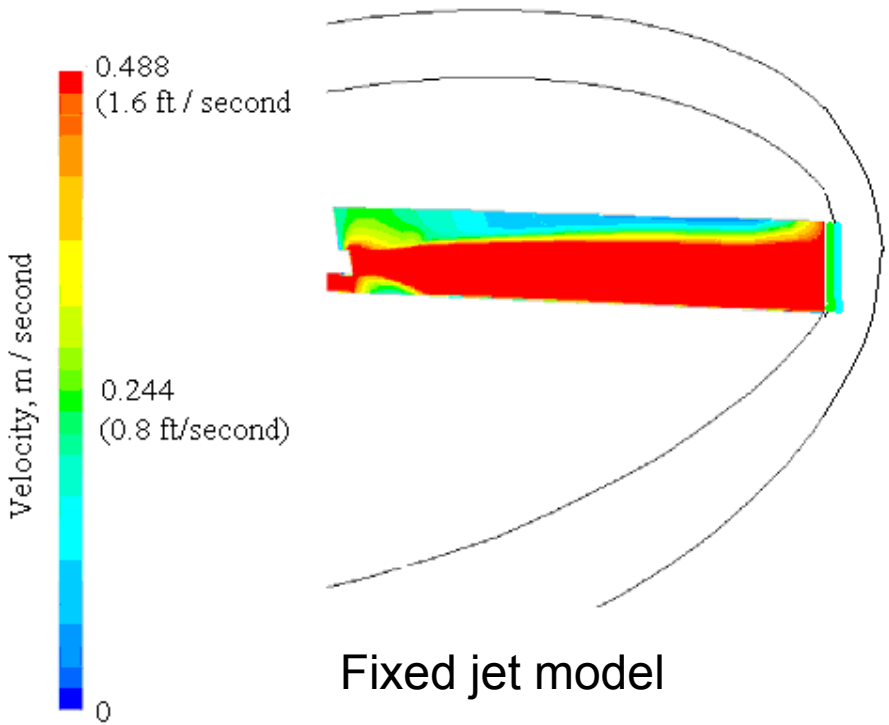
Fixed jet model

- Validated single phase model used to approximate two phase flow.
- To predict where mixing would occur, the experimental minimum velocity predicted by the ECR equation was used (2.27 ft / second)
- The modeled location of this velocity near the tank wall was then assumed to be the final sludge interface

A.5.76

SRNL

Jet Cross Section



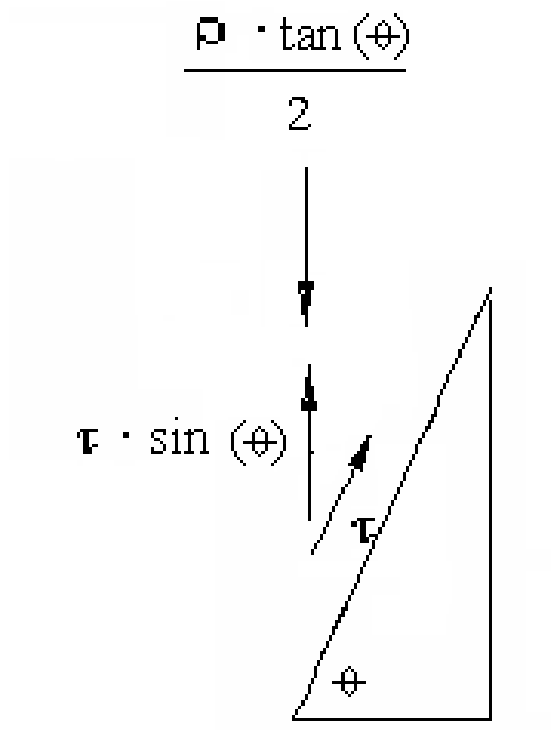
Fixed jet model

- CFD model predicts that some sludge will exist near the tank wall
- However, the actual tank geometry showed that some of the sludge should slide down into the higher velocity flow path
- 0-1400 gallons of residual waste was predicted depending on wall effects

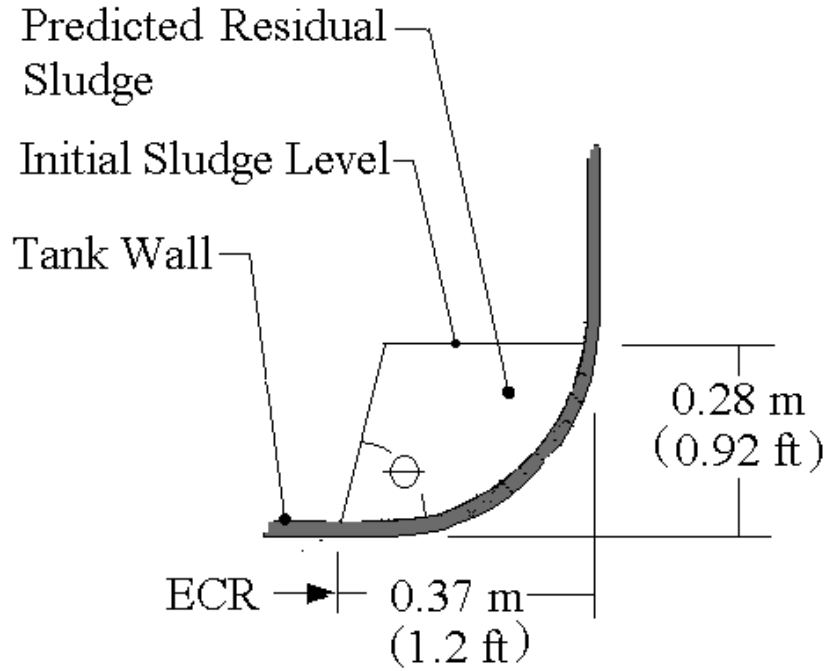
A.5.77

SRNL

Tank geometry effects on mixing at the wall

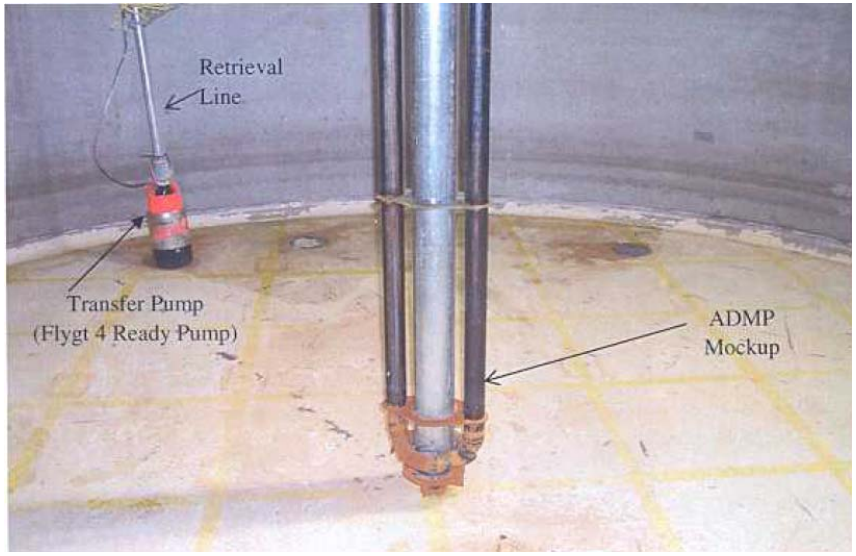


Angle of repose



A.5.78

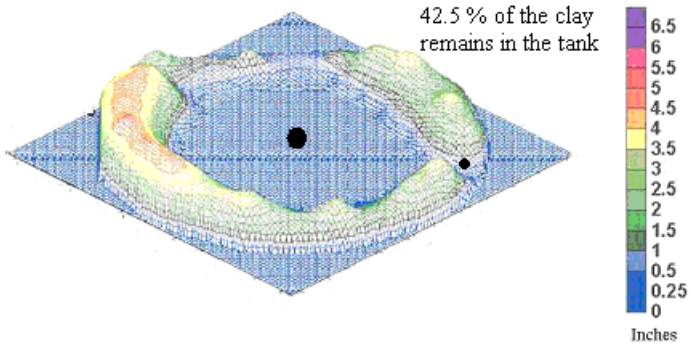
Quarter Scale Model, PNNL, Enderlin



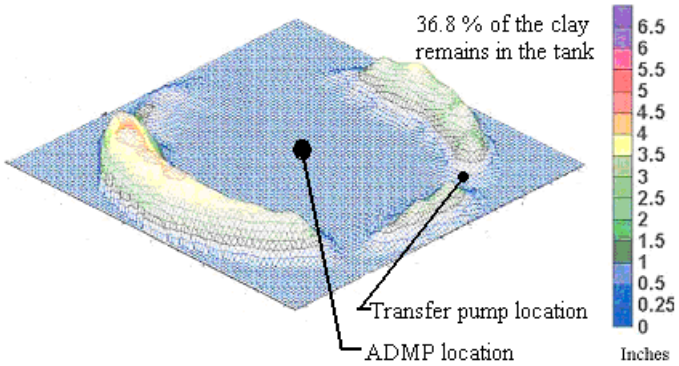
A.5.79

SRNL

Quarter Scale Kaolin Results

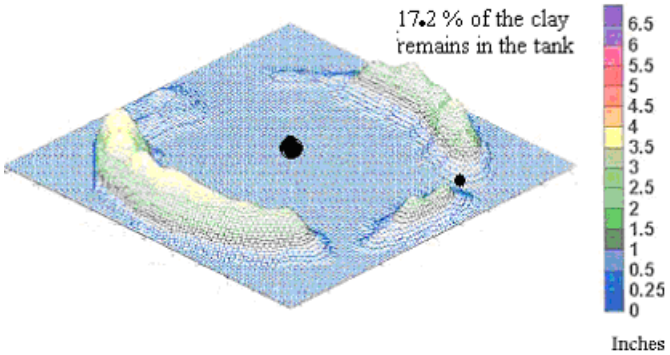


2.23 hours

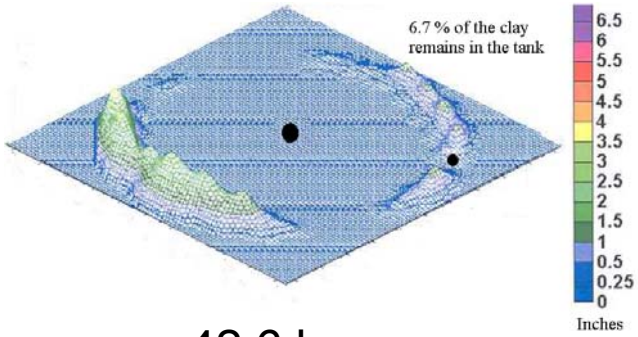


9.81 hours

- 10° rotations at 5 minute intervals
- Effective mixing of sludge predicted, even though quarter scale mixing was non-uniform
- Four mixing cycles were predicted



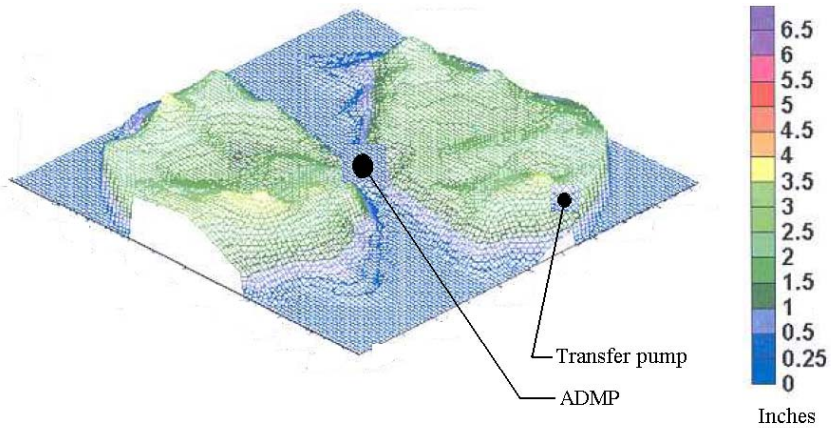
15.8 hours



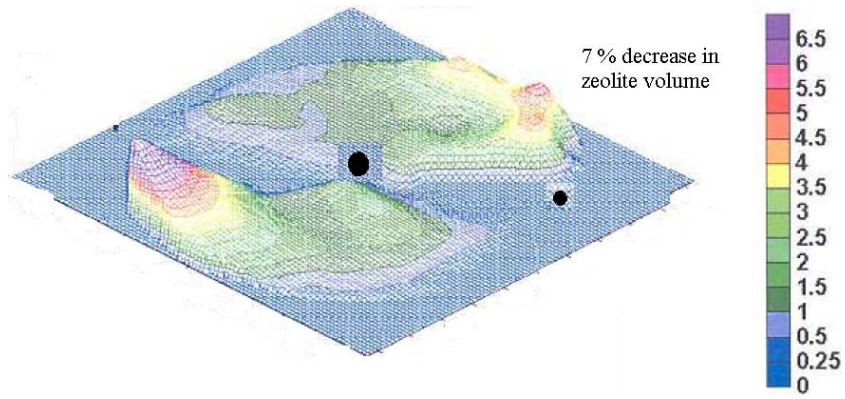
42.6 hours

SRNL

Quarter Scale Zeolite Mixing



11 minutes



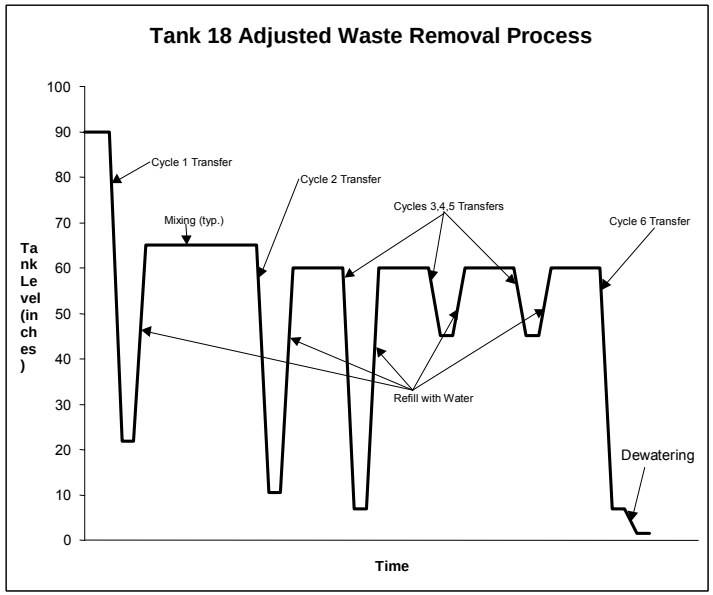
179 minutes

- Three hours of pump operation
- Fast settling solid, like sand
- Zeolite moved around in the tank, similar to sediment transport
- Only a small amount of Zeolite was transferred
- Ineffective transfer operation

A.5.81

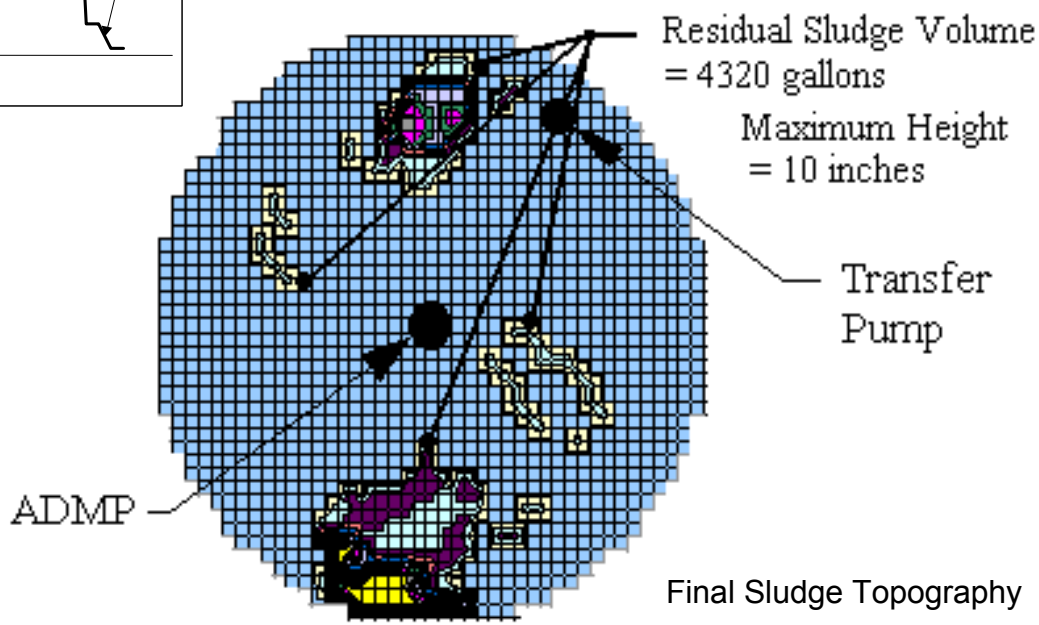
SRNL

Waste Tank Results, Augeri, Hubbard, Thomas



Waste removal Process

- Six waste processing cycles consisted of mixing, transferring, and refilling with water
- Sludge was mixed all the way to the wall
- Heel removal in process for Zeolite
- Foaming / low motor current issues?



Final Sludge Topography

A.5.82

Conclusions

- The ECR equation provides a conservative estimate for sludge removal, provided the sludge properties are known.
- Some additional waste removal can be obtained through pump indexing.
- CFD models were validated for future use in waste tank mixing and analysis of fluid jets
- The minimum fluid velocity required to produce a force sufficient to mix characterized sludge is approximately 2.27 feet / second.
- Particulate Zeolite is not effectively removed using a slurry pump.

References

- *Analysis of Turbulent Mixing Jets in a Large Scale Tank,* 2008, Lee, Dimenna, Leishear, Stefanko, ASME Journal of Fluids Engineering.
- *Mixing in Large Scale Tanks, IV,* Augeri, Hubbard, Thomas , 2004, ASME Joint Heat Transfer and Fluids Engineering Conference.
- *Mixing in Large Scale Tanks, III, Predicting Slurry Pump Performance*, Leishear, Lee, Dimenna, Stefanko, 2004, ASME Joint Heat Transfer and Fluids Engineering Conference.
- *Mixing in Large Scale Tanks, II, Full Scale Pump Testing*, Leishear, Stefanko, Lee, Dimenna, 2004, ASME Joint Heat Transfer and Fluids Engineering Conference.
- *Mixing in Large Scale Tanks, I, Flow Modeling of Turbulent Mixing Jets*, Lee, Dimenna, Stefanko, Leishear, 2004, ASME Joint Heat Transfer and Fluids Engineering Conference.
- *ADMP Mixing of Tank 18F Sludge; History, Modeling, Testing, and Results,* R. Leishear, M. Augeri, R. Dimenna, D. Stefanko, S. Lee, J. Thomas, M. Hubbard, 2004, WSRC-TR-2004-00036
- *Performance Analysis for Mixing Pumps in Tank 18*, 2001, Lee, Dimenna, WSRC-TR-2001-00391
- Additional references included in each of the reports and publications.

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Integrated Engineering, Construction, and Management Solutions

Impeller Diameter Reduction of a Radioactive Sludge Transfer Pump

T. B. Caldwell

Savannah River Site

Liquid Waste Division

January 18, 2008

Corollary

Every project is at least ten times more difficult to implement in the field than <u>ever imagined</u> when first thought of at the desk

Famous Last Words: "…and how hard can that be? "

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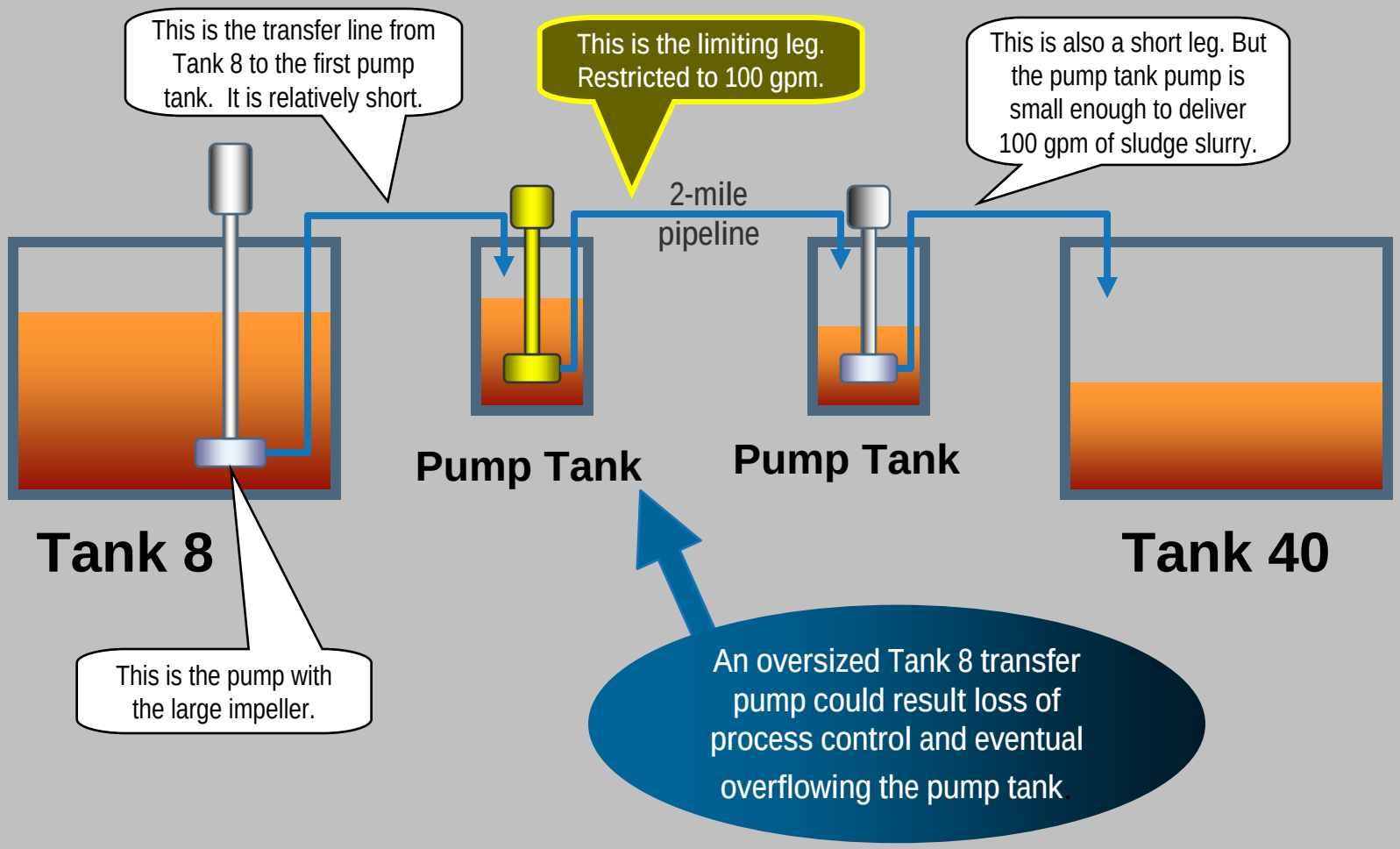
Summary

The Savannah River Site trimmed the impeller of a radioactive sludge transfer pump from 11 inches in diameter to 8 inches (a reduction of over 25%). This resulted in a more favorable flow and head performance for waste removal operations.

Tank 8 to Tank 40 Transfer Path



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Design Restrictions

- tank 8 pump variable speed motor can operate no slower than 1100 rpm
- because of column resonance, motor should not be operated between 1500 and 1800 rpm
- tank slurry conditions highly variable
- the minimum flowrate is 70 gpm

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Step by Step

Step 1 – identify slurry conditions

Percent Solids	Yield Stress (dynes/cm^2)	Consistency (cP)	Specific Gravity	Static Head (ft)
5	6	3	1.1	3.8
10	14	5	1.2	6.2
15	32	8	1.4	20.4
18	51	10	1.5	20.4

Operating Band

Step 2 - develop system curve

- Texas A&M flow model corrected for sludge simulant empirical data

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Governing Equations – Friction Factors

<u>laminar flow</u> friction factor from the Buckingham equation

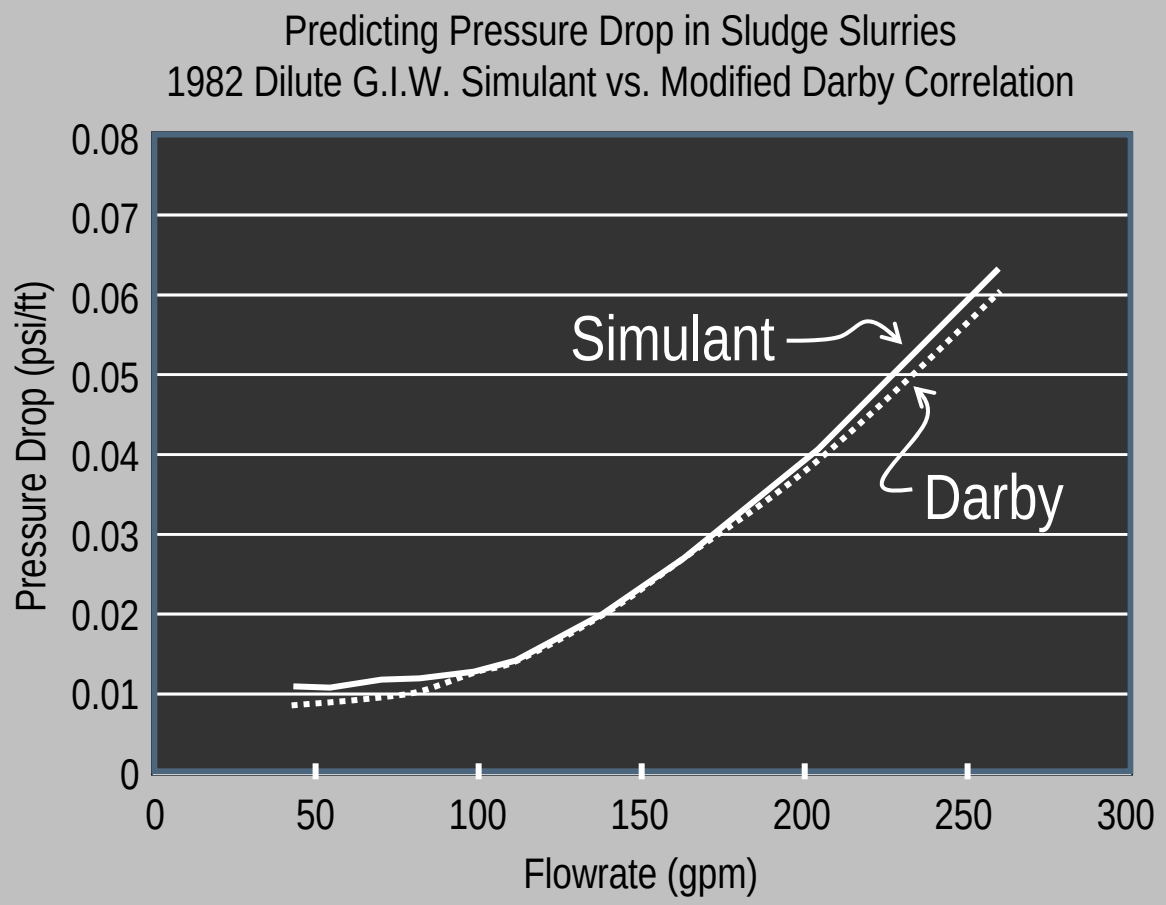
$$f_{\mathrm{L}} = \frac{16}{N_{\mathrm{Re}}}\left[1 + \frac{N_{\mathrm{He}}}{6\,N_{\mathrm{Re}}} - \frac{N_{\mathrm{He}}^{4}}{3\,f_{\mathrm{L}}^{3}\,N_{\mathrm{Re}}^{7}}\right]$$

<u>turbulent flow</u> friction factor from test data

$$f_{\mathrm{T}} = 10^{a}\,N_{\mathrm{Re}}^{-0.193} \quad \text{where}$$

$$a = -1.53\left[1 + 0.146\,e^{-2.9\times10^{-5}\,N_{\mathrm{Re}}}\right]$$

Curve Fitting the Friction Factor



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Governing Equations – Pipe Flow

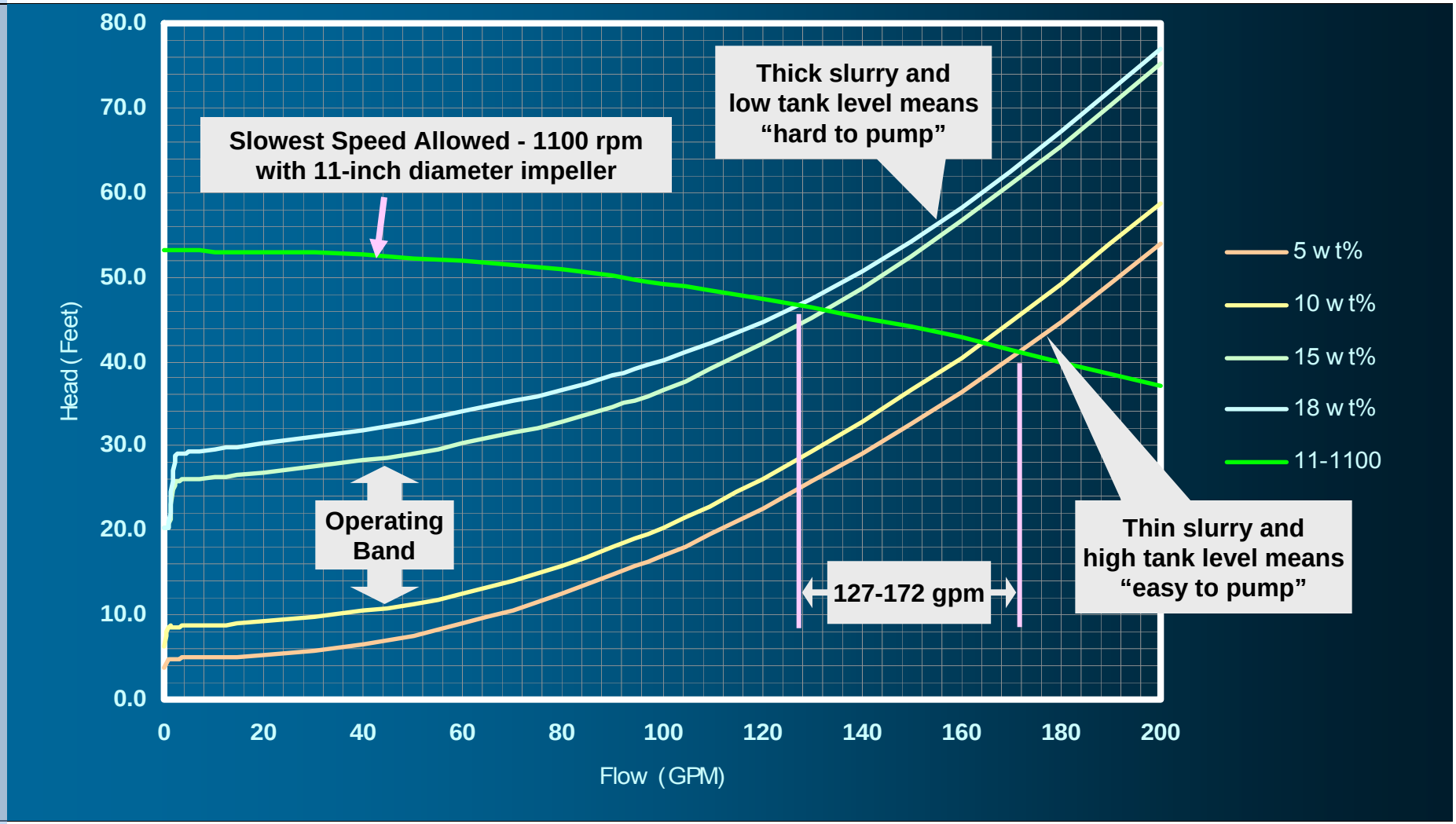
Reynolds number for Bingham plastic fluid in pipes

$$N_{\mathrm{Re}\,BP} = \frac{4Q\rho}{\pi D\eta\left[1+\dfrac{\pi D^3\tau_0}{24Q\eta}\right]}$$

Two-K Method by Hooper

$$K = \frac{K_1}{N_{\mathrm{Re}\,BP}} + K_\infty\left[1+\frac{1}{\mathrm{ID}_{\mathrm{in}}}\right]$$

Step 3 - determine head requirements



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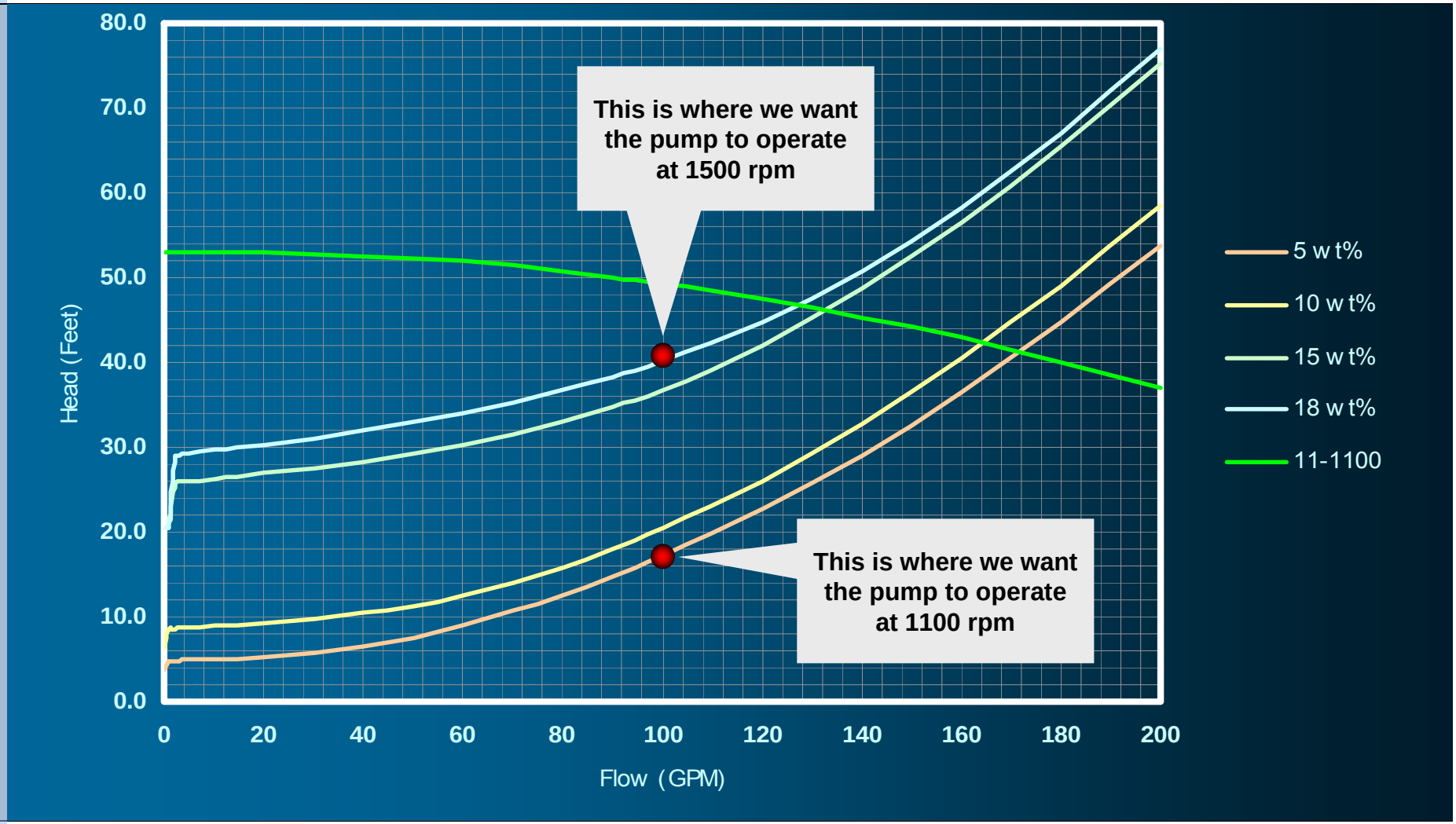
Step 3 - determine head requirements (cont'd)



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Step 4 – impeller sizing

Affinity Laws

$$\frac{S_1}{S_2} = \frac{D_1}{D_2} = \frac{Q_1}{Q_2} = \frac{\sqrt{H_1}}{\sqrt{H_2}}$$

D = impeller diameter
H = head
Q = flowrate
S = pump speed

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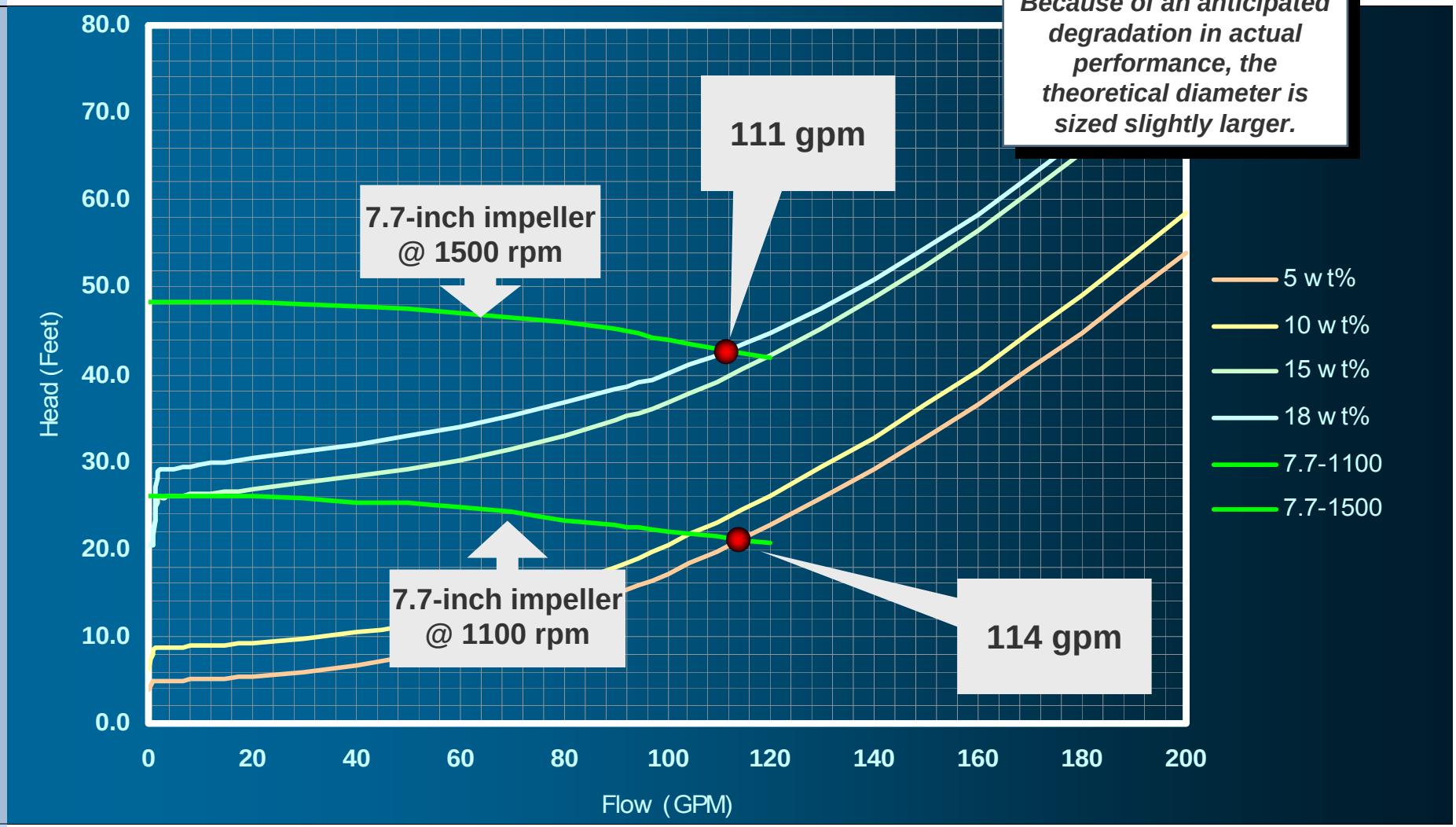
Step 4 – impeller sizing (cont'd)



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Step 5 – impeller size correction

- any diameter reduction greater than 10% will deviate from the affinity laws.
  - The head and flow will be less than predicted.
  - The degree of deviation is an educated guess.
  - Reduce diameter in stages
- desired to trim and test the impeller once.
- uncorrected diameter (7.7 inches) chosen to allow slightly higher flow and head.

Step 5 – impeller size correction (cont'd)

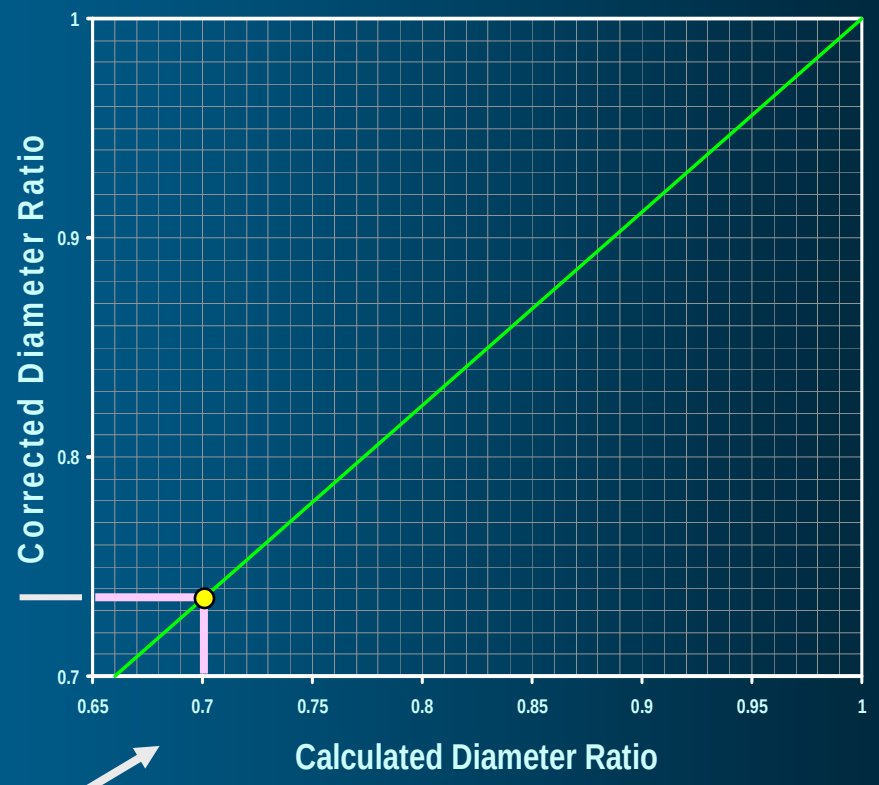
- two industry proven methods to correct calculated impeller diameters:
  - Stepanoff Method
  - Rütshi Method
- both were used to provide a correction factor based on engineering judgment.

Step 5 - Impeller Size Correction

Stepanoff Method

$$D_{Corrected} = 0.735 \times 11.0''$$

$$D_{Corrected} = 8.09 \text{ inches}$$



0.735

$$\frac{D_{Calculated}}{D_{Original}} = \frac{7.7}{11.0} = 0.7$$

"Centrifugal and Axial Flow Pumps", A. J. Stepanoff, 2nd edition, 1957. Figure 5.11. Page 87. Used by Permission. John Wiley & Sons, Inc. New York, New York

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Step 5 - Impeller Size Correction

Rütshi Method

$k = 0.88$

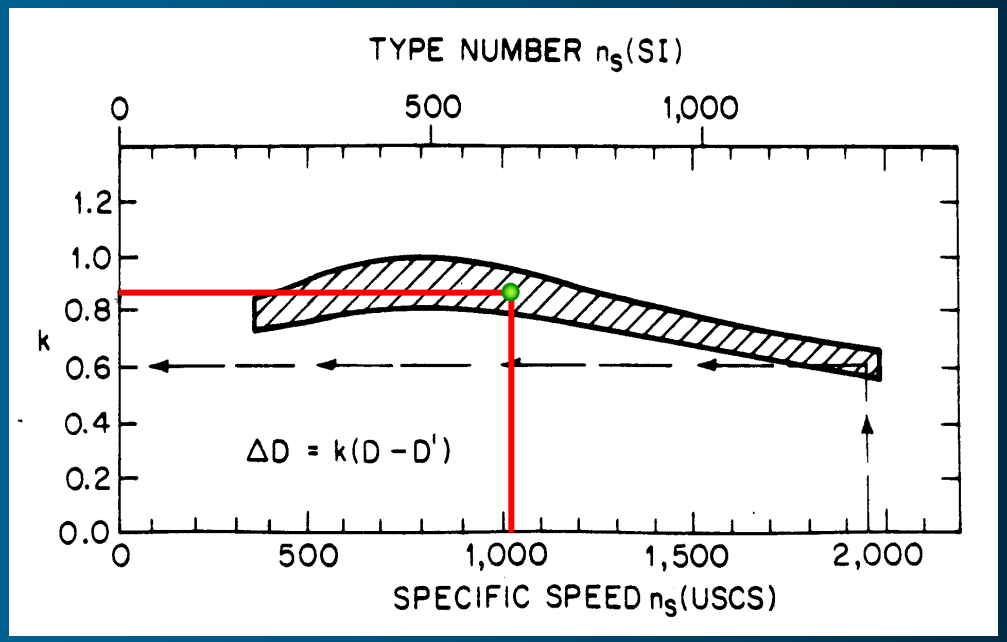
$\Delta D = k(D - D')$

$\Delta D = 0.88(11 - 7.7)$

$\Delta D = 2.90 \text{ inches}$

$D_{Corrected} = 11 - 2.90$

$D_{Corrected} = \underline{8.10 \text{ inches}}$



First determine Specific Speed

n_s Input

H = 155 ft

Q = 395 gpm

S = 2250 rpm

$$n_s = \frac{S\sqrt{Q}}{H^{0.75}} = \frac{2250\sqrt{395}}{155^{0.75}} \approx \underline{\underline{1020}}$$

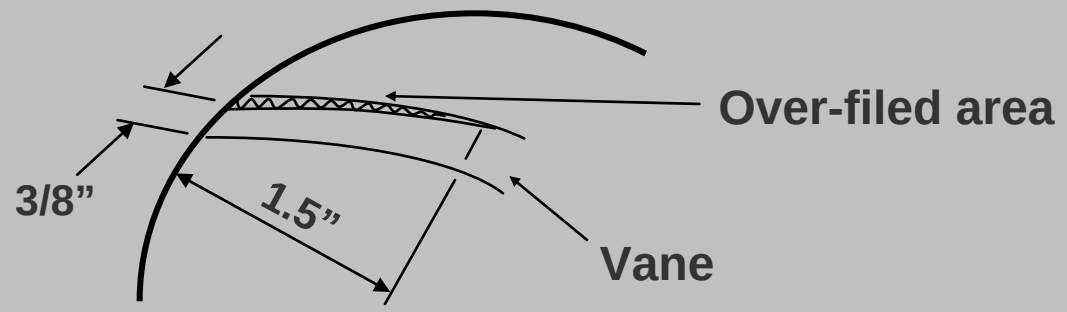
"Untersuchungen an Spiralgenhäusepumpen verschiedener Schnelläufigkeit," Schweiz. Arch. Angew. Wiss. Tech., Volume 17. No. 2. Figure 13, Page 38. February 1951. Used by permission.

Impeller Size Correction Summary

- both methods resulted in an impeller diameter of roughly 8.1 inches
- because of the concern that the pump might be oversized, the recommended size was 8.0 inches
- this would provide sufficient low-end performance without sacrificing high end characteristics

Step 6 – trim impeller

- cut vanes and shrouds to 8.0 inches
- overfile impeller vanes and blend the file 1.5 inches into vane as shown:



- speed balance impeller and install

Transfer pump being inserted into Tank 8

long shafted telescoping centrifugal pump suspended from crane.

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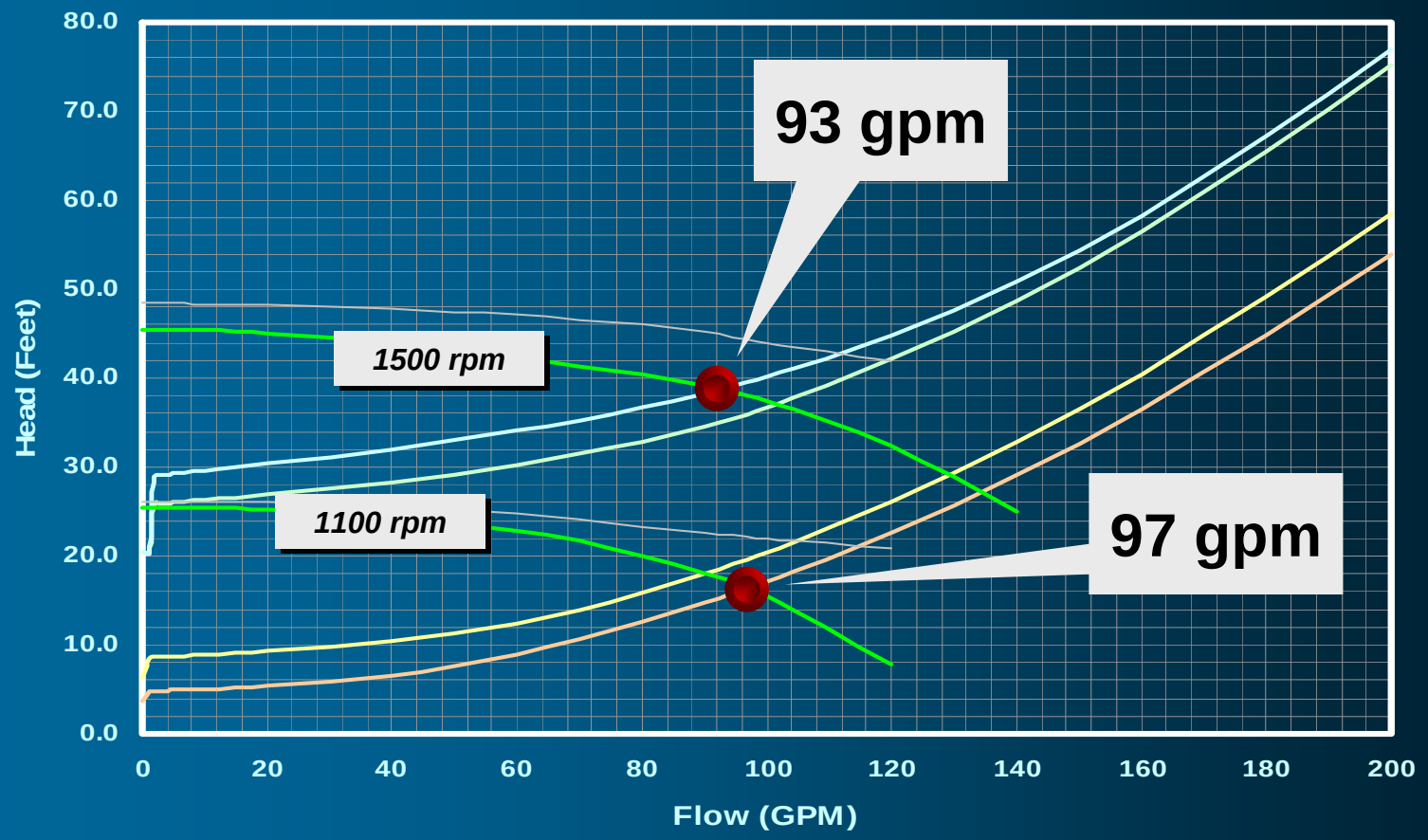
Step 7 – field results



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Examples of slurry Retrieval, Pipeline Transport & Plugging and Mixing issues at Sellafield in the UK

Tim Tinsley & Dominic Rhodes
Nexia Solutions

nexiasolutions

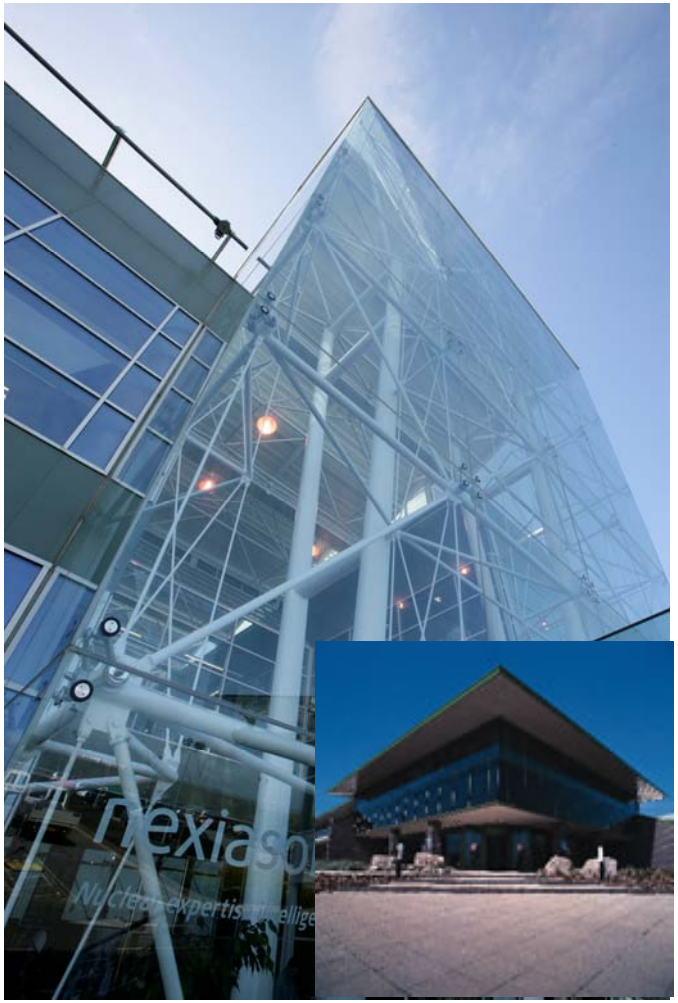
Nuclear expertise intelligently applied

Agenda

- Overview of Plant and Processes
- Examples of solid formation, and issues
- Examples of characterisation and management of solids

Nexia Solutions

- Pre 1996: Multi Centred Nuclear Technology Tradition
- 1996: Integrated R&T Function
- 2003: Nuclear Sciences and Technology Services (NSTS)
- 2003: Acquired AEA (T) Nuclear Science Business
- 2005: Nexia Solutions operate with full subsidiary Status
- **2008: National Nuclear Laboratory**



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Remit for the UK's National Nuclear Laboratory

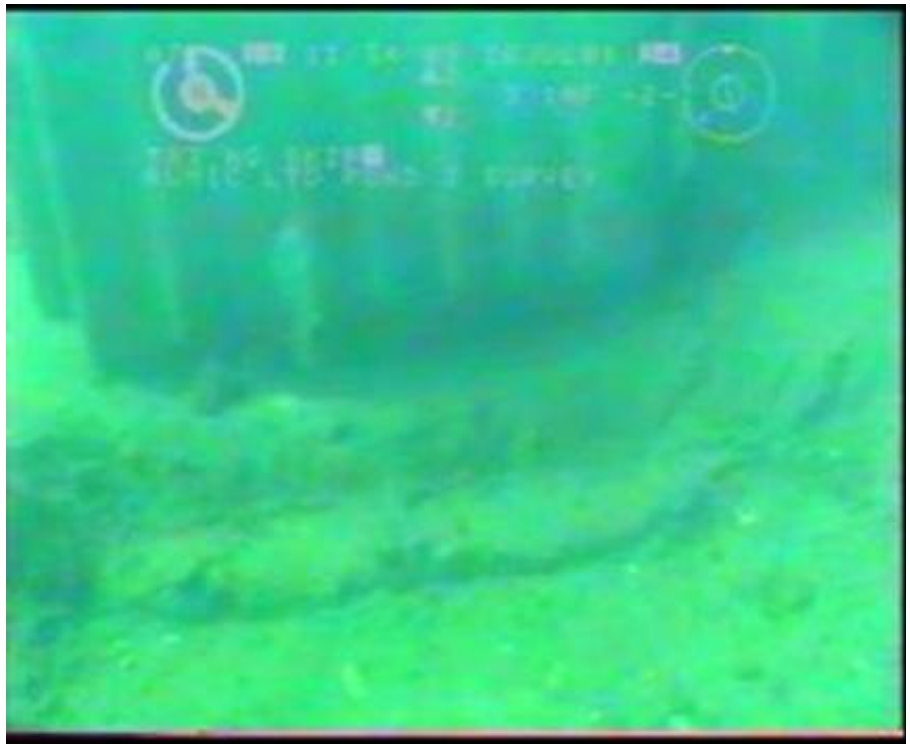
- Customer funded Laboratory with a key role to support the UK's strategic R&D requirements
- Development of the UK's R&D supply base
- Increased links to Universities
- Involvement in international programmes

The Sellafield Site

Plants & Process at Sellafield

- Variety of operational/legacy plants, ranging in age and hence engineering design
- Fuel storage Ponds, reprocessing plants, Supporting treatment plants, Interim storage plants, Product finishing plants
- Acid based solvent extraction
- Evaporation, storage and Vitrification
- Magnox corrosion product sludges
- Powder formation
- External conditions impose other constraints

Example of older Pond & Sludge



Example of complexity of pipework

Complex stainless steel pipework in the HAL Cell.

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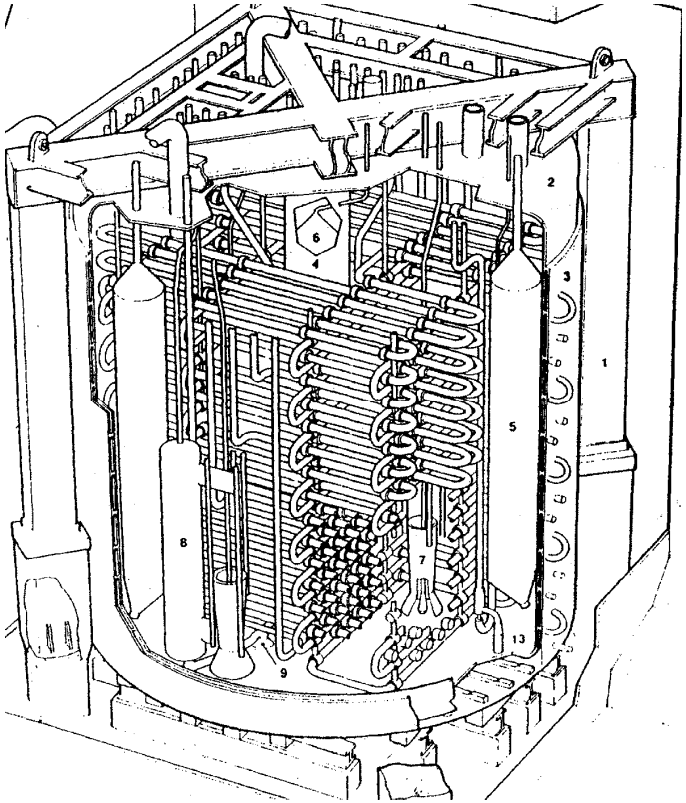
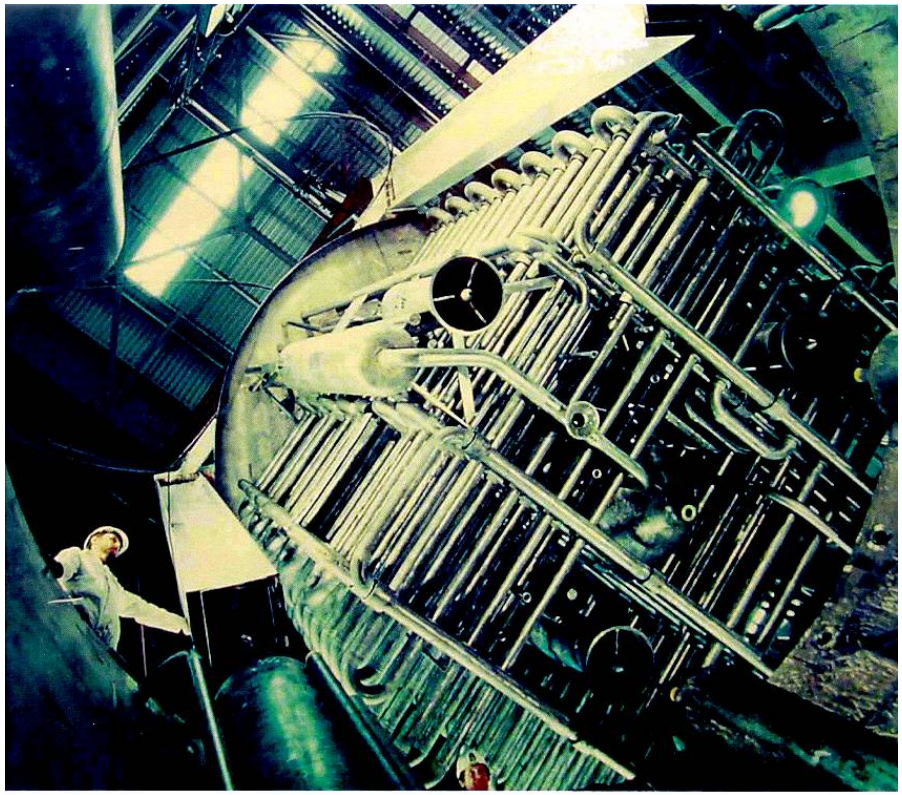
Example of complexity of pipework





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Solid formation

- Solids can form due to a number of mechanisms
  - Precipitation - planned as product formation
  - Precipitation – consequence of concentration
  - Crystallisation – consequence of concentration, or result of change of condition
- Solids can be deposited due to a number of mechanisms
  - Cold surface crystallisation
  - Poor mixing or suspension
  - Settling during transport in pipes

‘Designed in’ problems

- Gravity fed part filled low slope pipes
- Changed in external conditions (temperature)
- Dilution (ejectors) & concentration (evaporation)
- Obstructions
- No access
- Non continuous mixing

Highly Active Liquor chemistry

- HA raffinates contain;
  - Fission products (e.g. Sr, Mo, Zr, Cs, Ba, lanthanides).
  - Actinides (Np, Am, Cm), including low levels of U & Pu.
  - Plant corrosion products (Fe, Cr, Ni).
  - Other fuel & process additives (e.g. Gd, Mg, Al).
  - Acidic (Nitric Acid)
  - It is also heat producing (~4 watts / litre) and has an activity of ~17 TBq / litre

- During the concentration process these give several precipitates;
  - Zr phosphates.
  - Barium/strontium nitrate.
  - Cs phosphomolybdate.
  - Lanthanide nitrates or Mg lanthanide nitrates.

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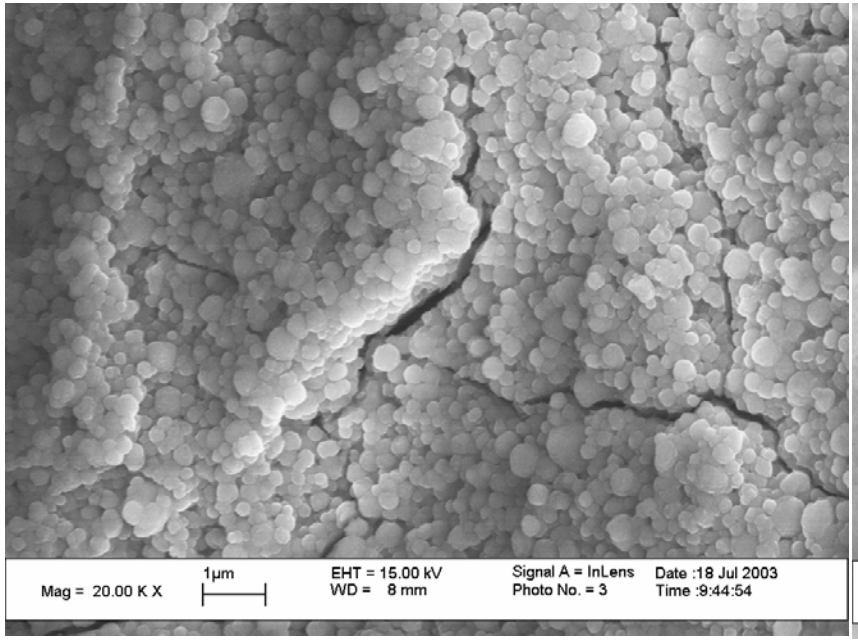
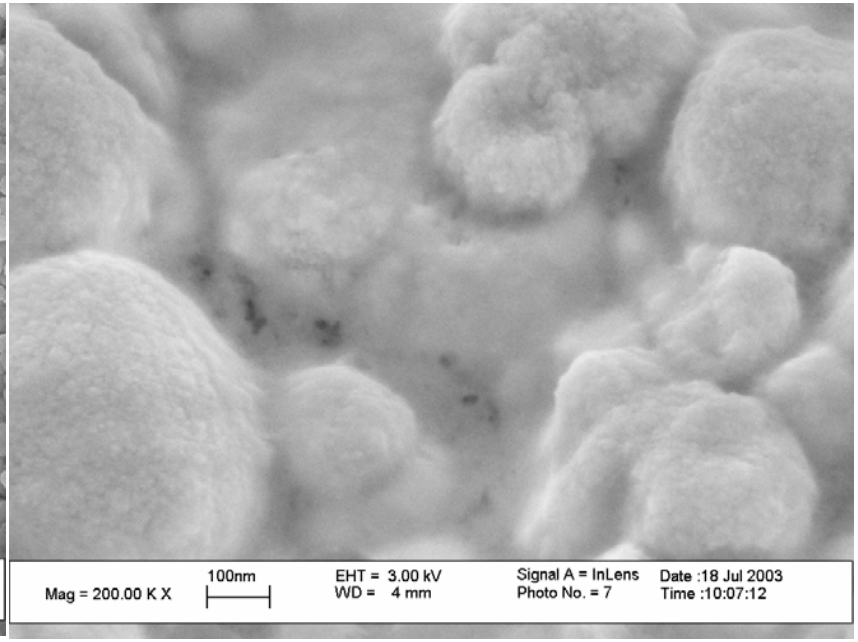
Cs phosphomolybdate (CPM) issues

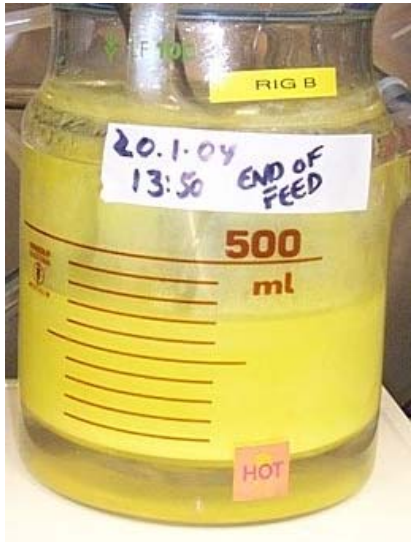
- Fast settling (*ca.* 4 g/cm^3).
- Contains radioactive isotope ^{137}Cs, *ca*. 50% of Cs.
- Formed during evaporation, but converts to zirconium molybdate which is slow settling but does not contain ^{137}Cs





Formation of CPM -HA simulant evaporation

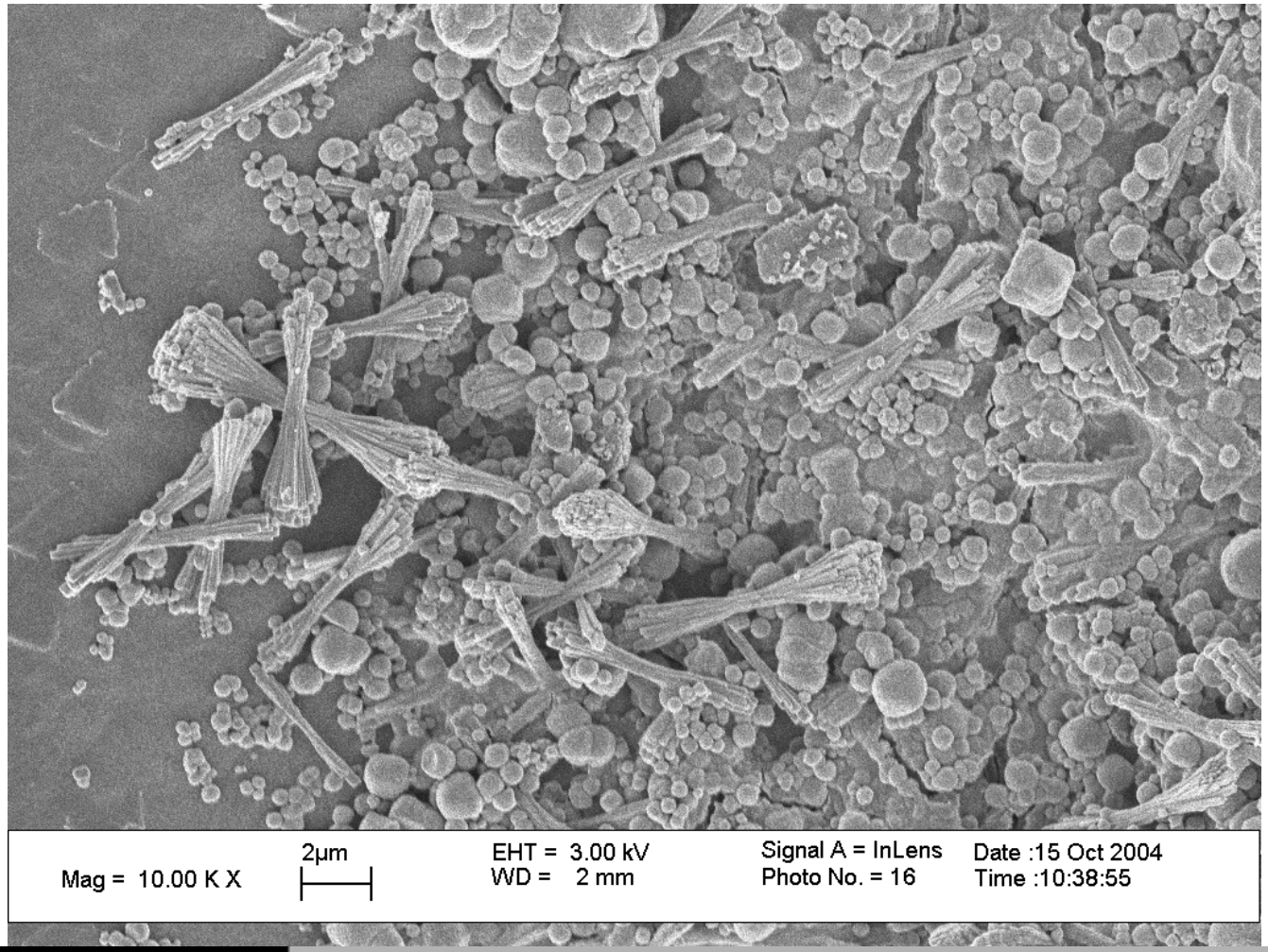
Day 1



Day 7 end feed

Day 20

An example of ageing Highly Active Liquor



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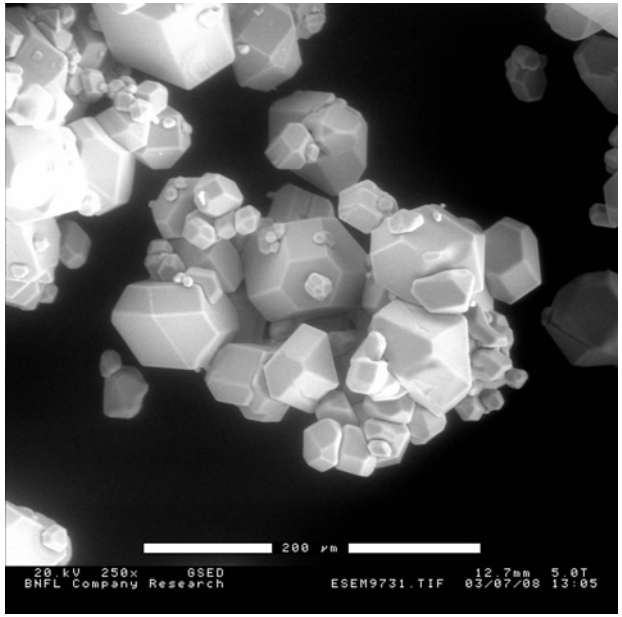
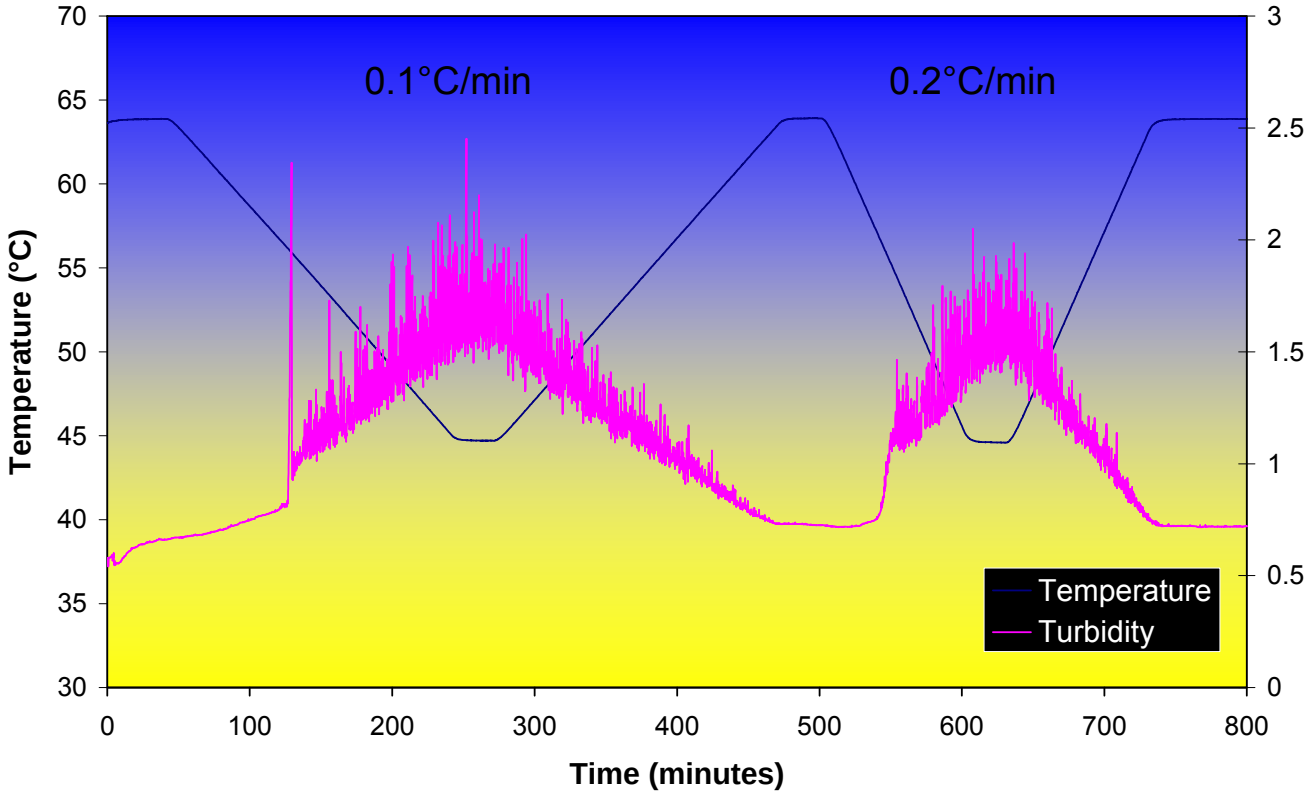
Barium/strontium nitrate issues

- Crystalline material formed during evaporation
- Acidity and temperature sensitive in the range used for storage
- Hence changes in these conditions can lead to formation of solids e.g. cold surfaces, addition of other liquors
- Can block pipes when transferred
- Noticeable meta stable zone width



Barium/strontium nitrate - Meta-stable zone widths

Whilst the precipitation occurred at the same temperature, irrelevant of cooling rate, and within 1°C of the solubility limit , there was a noticeable hindrance to dissolution.



Meta-stable zone widths for 1:1 barium:strontium nitrate (solution) at two heating/cooling rates.

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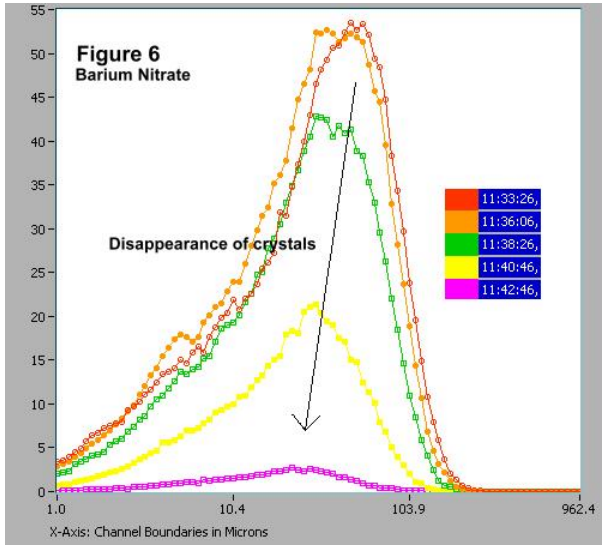
Characterisation of solids

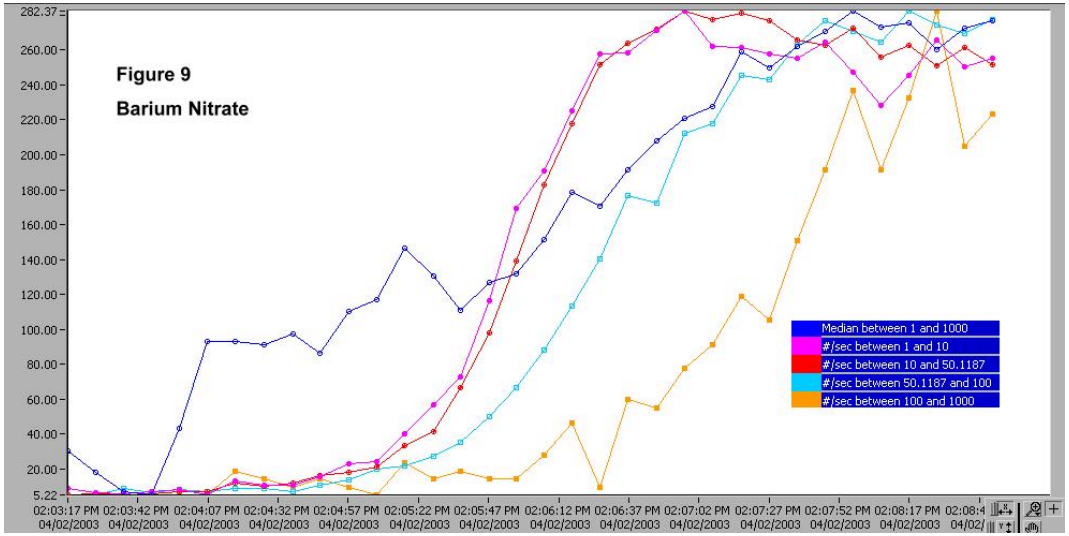
- In situ typically very difficult, but not impossible
- HAL samples limited to 3-5ml
- Use simulants, where inactive isotopes are used following a simulated process to real plant
- Chemical modelling to determine sensitivities

Particle sizes – In situ with simulants

- Particle sizing in situ using a Lasentec probe



Particle sizes during the dissolution (warming) of a barium nitrate / nitric acid solution. The mean particle size is 71µm



Particle sizes towards the end of a precipitation - there is a significant increase in particle count, especially in the fine region. There is also an increase in coarse particle count towards the concluding period.

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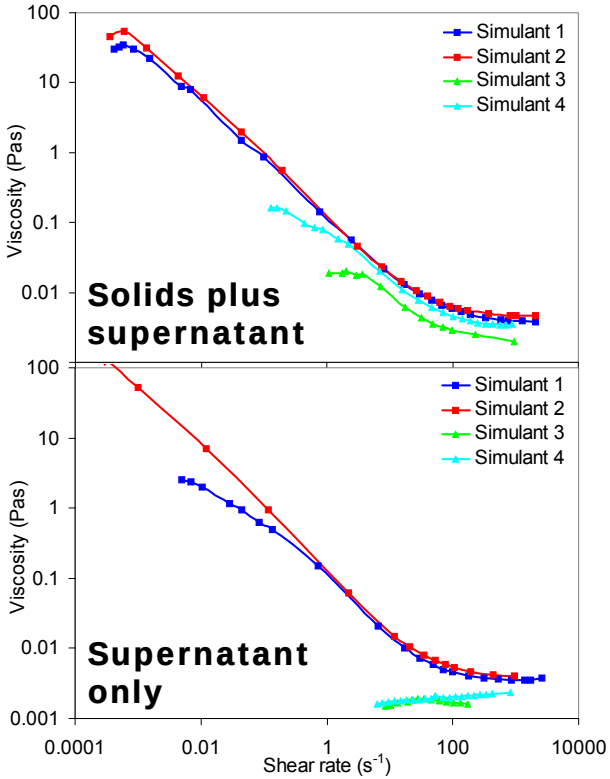
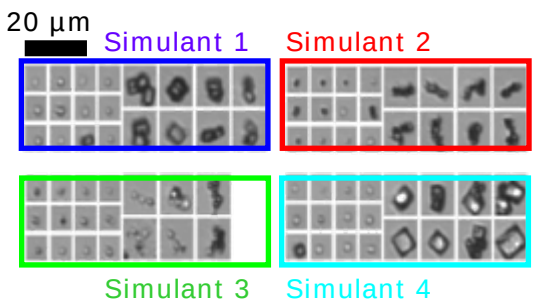
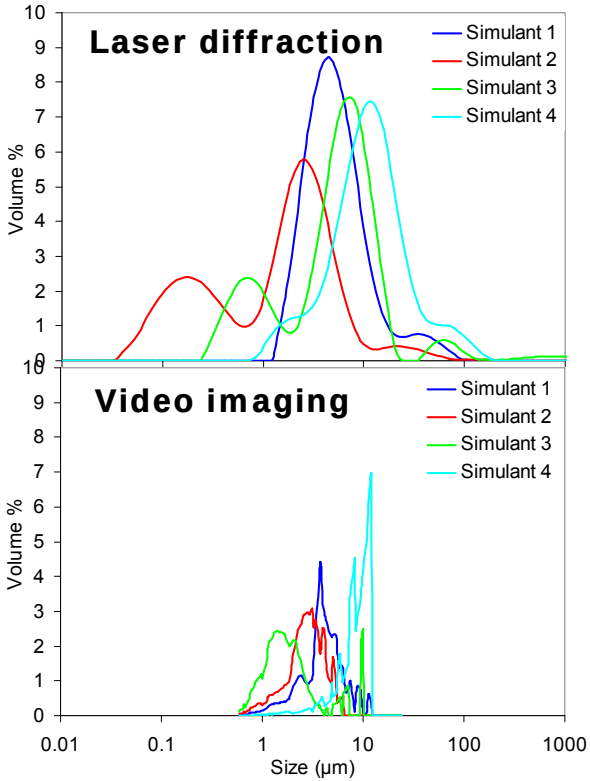
Typical characterisation of simulant suspended solids



- Rheology Flow Curves
- Elemental Analysis, XRD
- Particle and Solution Density
- Solids Concentration
- Particle Size and Shape





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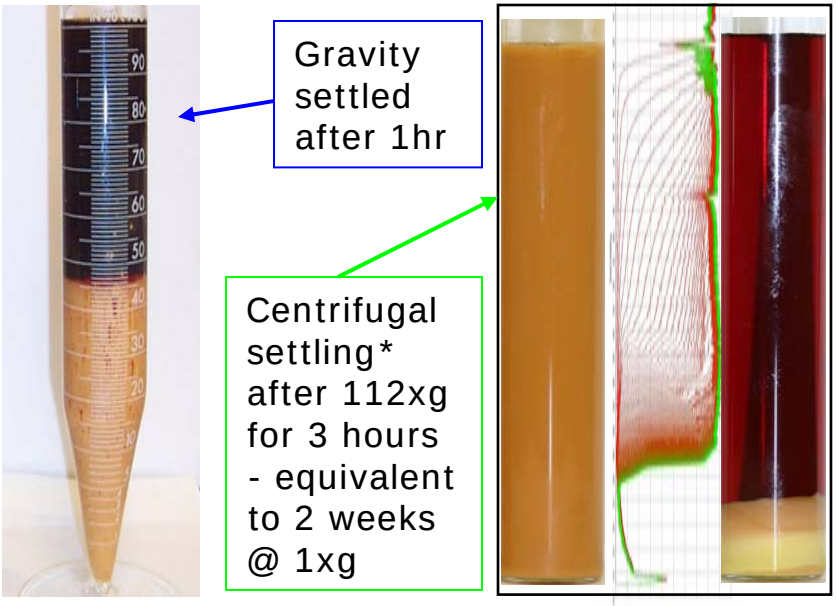
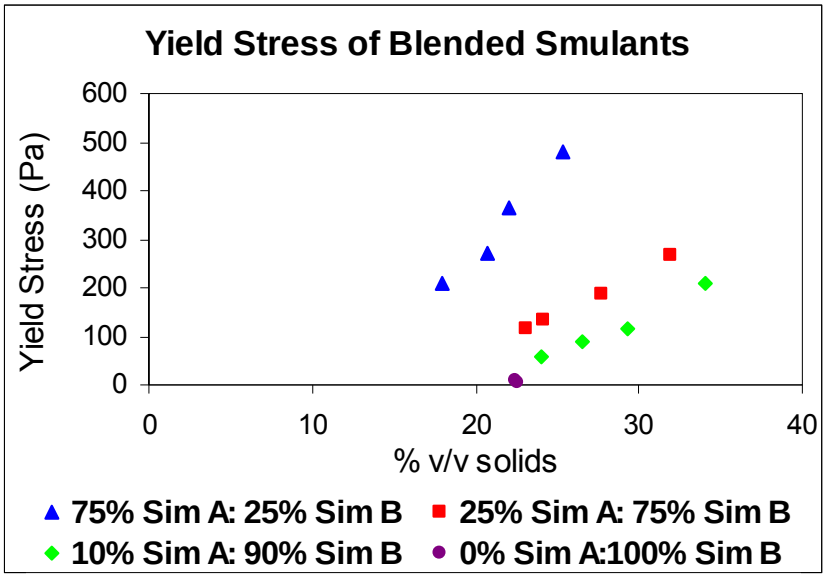
Inactive Simulant Characterisation

- Settling and sedimentation behaviour



- Shear yield stress at potential settled bed conditions



*using LUMiFuge 116 Stability Analyser

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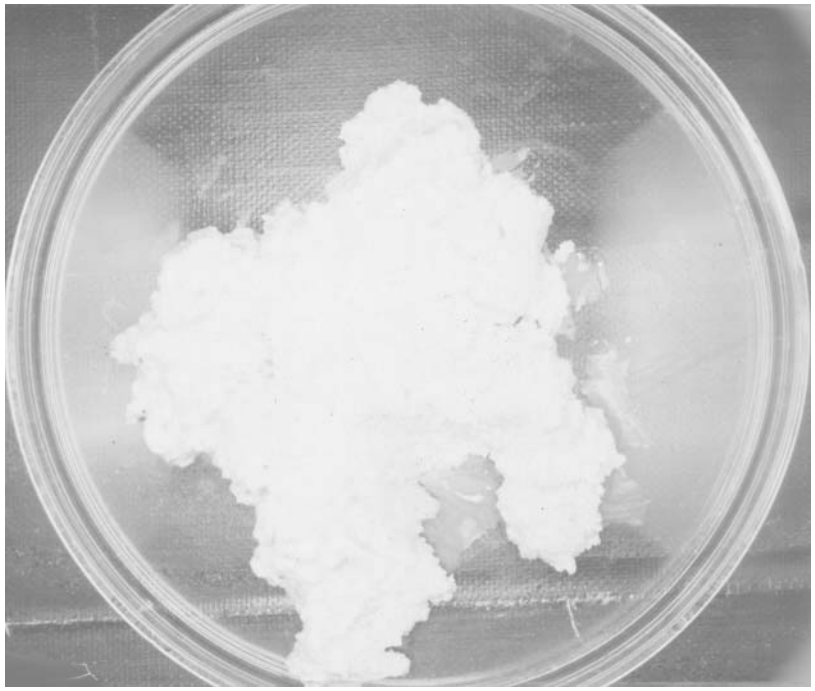
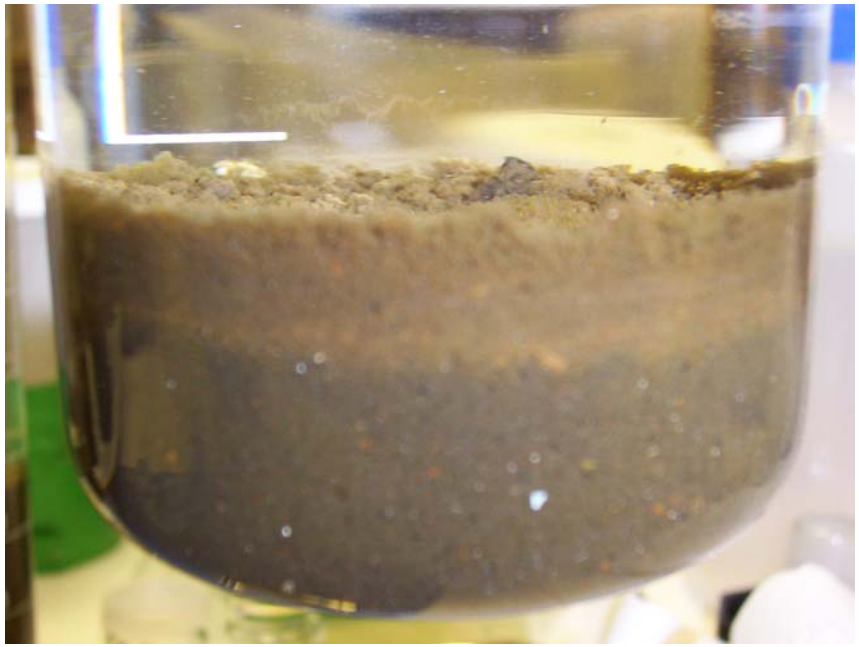
Retrieval & Treatment of Legacy Wastes

- Retrieval of legacy wastes and conversion to forms for safe storage
  - Largely cladding wastes (Mg / Al) and residual fuels
  - In ponds and silos
- Challenges
  - Characterising the waste
  - Hydrogen
  - Potential for reactive materials
  - Liquid and aerial discharges

Examples of sludges (1)



Examples of Sludges (2)



Components of sludges

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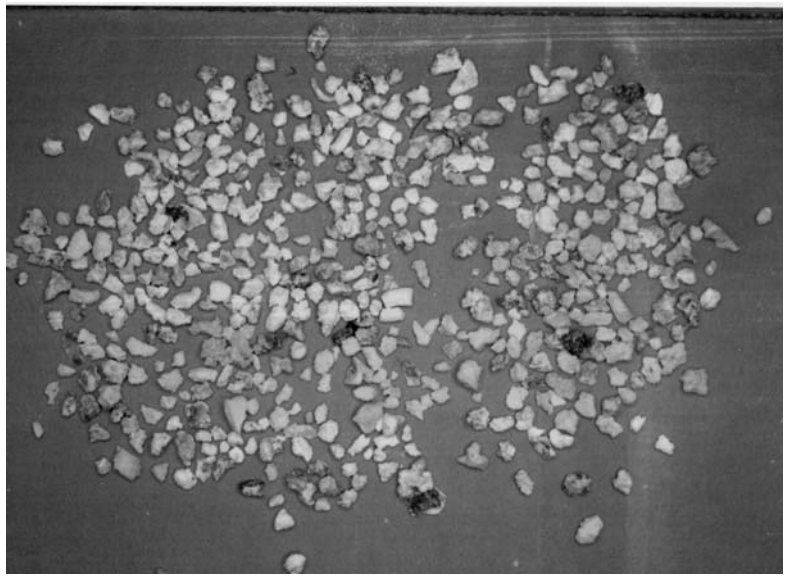
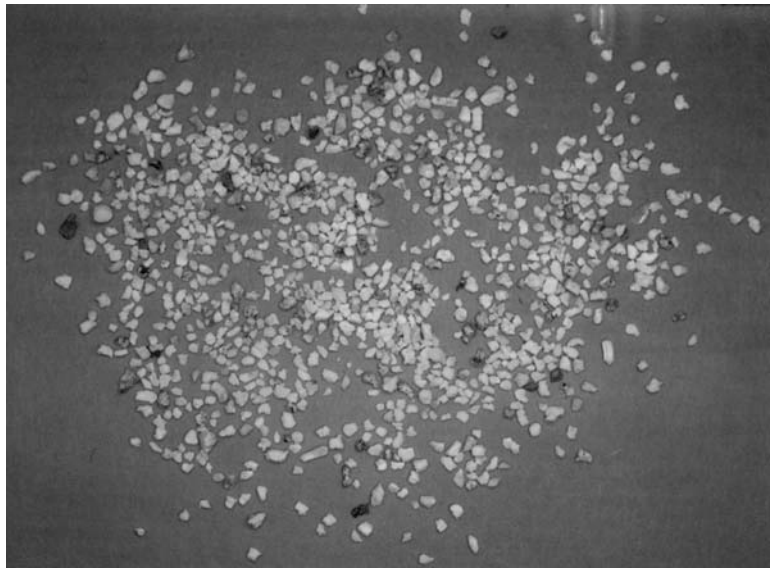
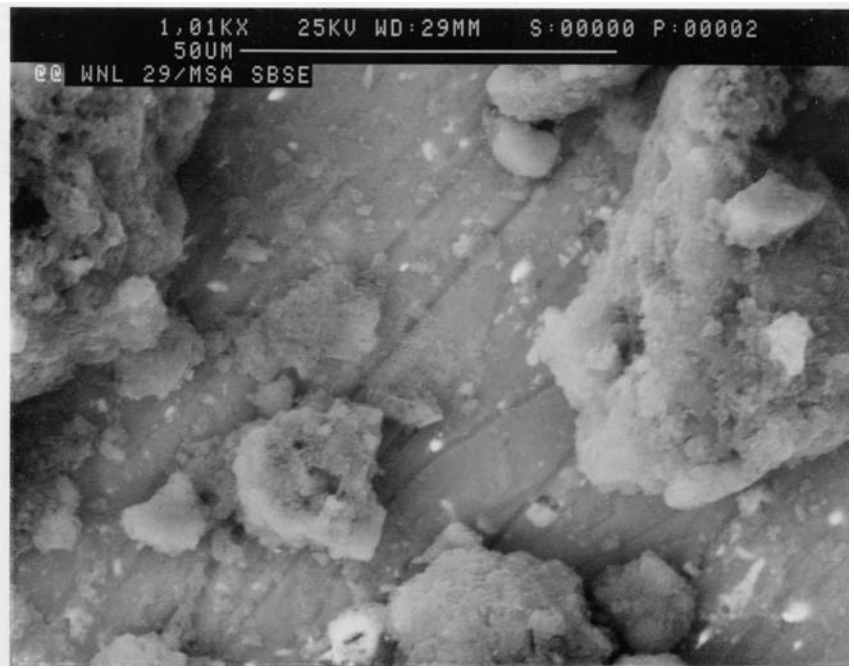
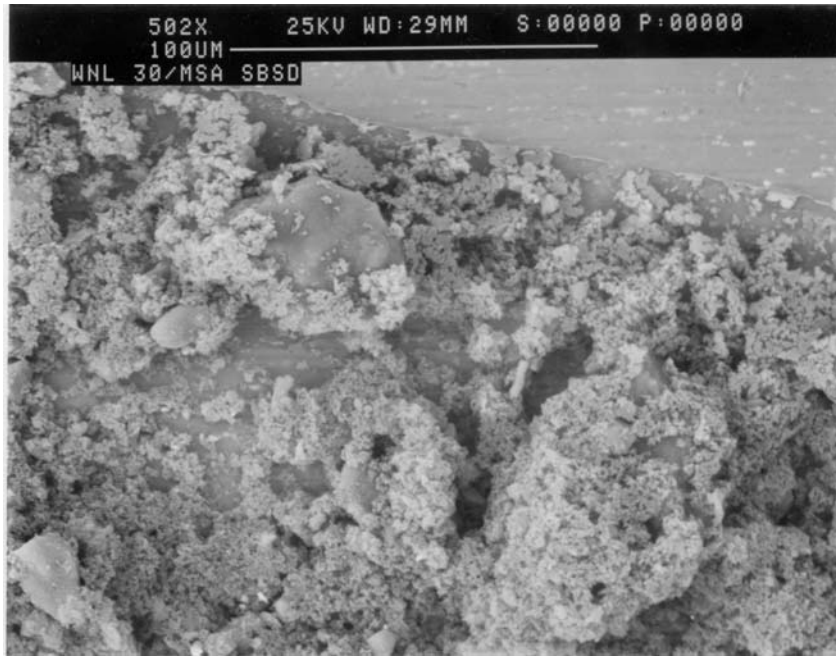
Sludge components getting smaller





Sludge components (very small)





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Sludge properties

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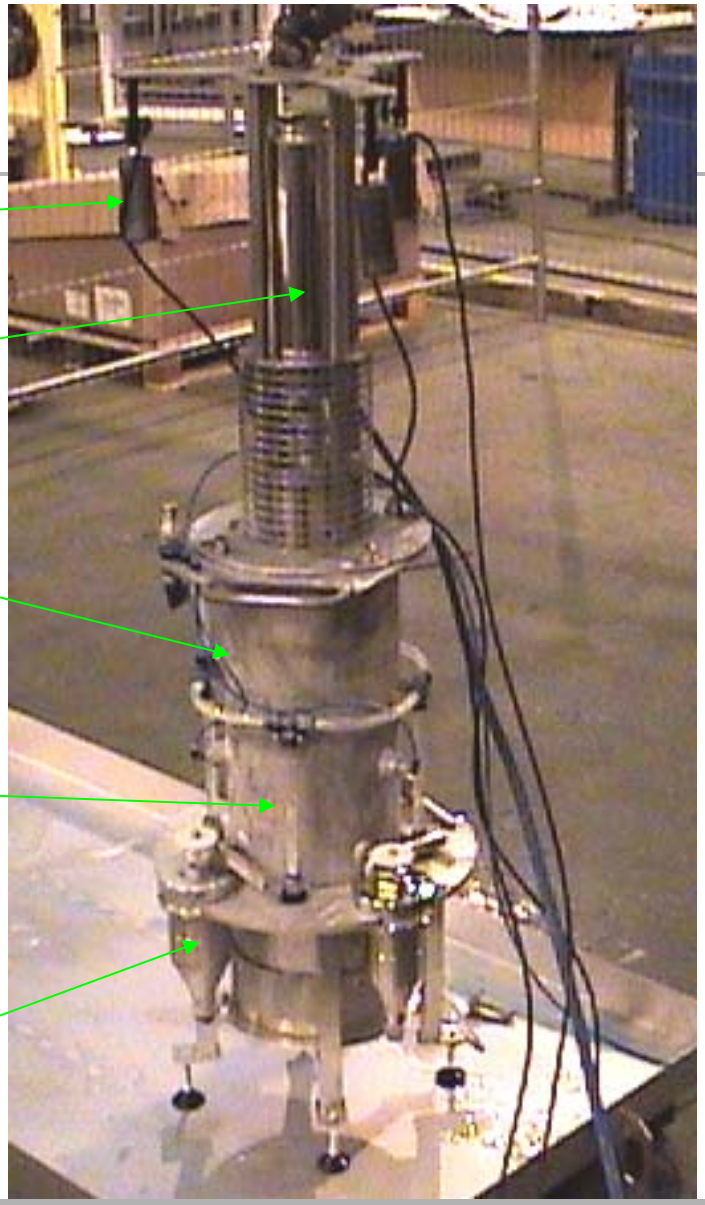
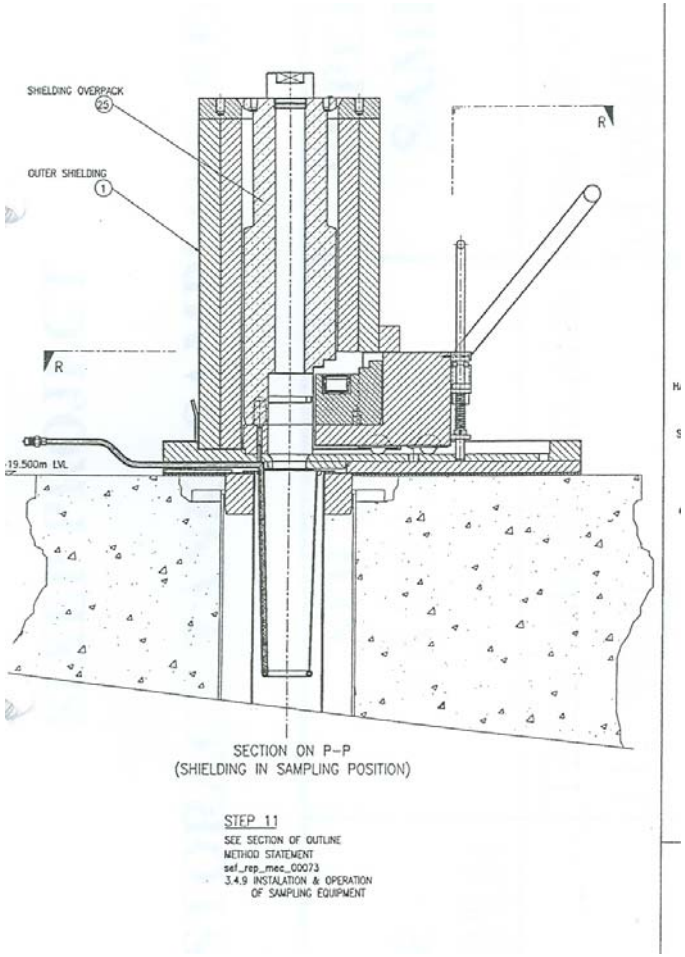
Test Materials

In-situ Experiments eg. Bell Jar

Developed to assess the amount of activity transferred from ILW sludge to the bulk liquor phase upon sludge disturbance.



In Situ Measurements
Troll 9000



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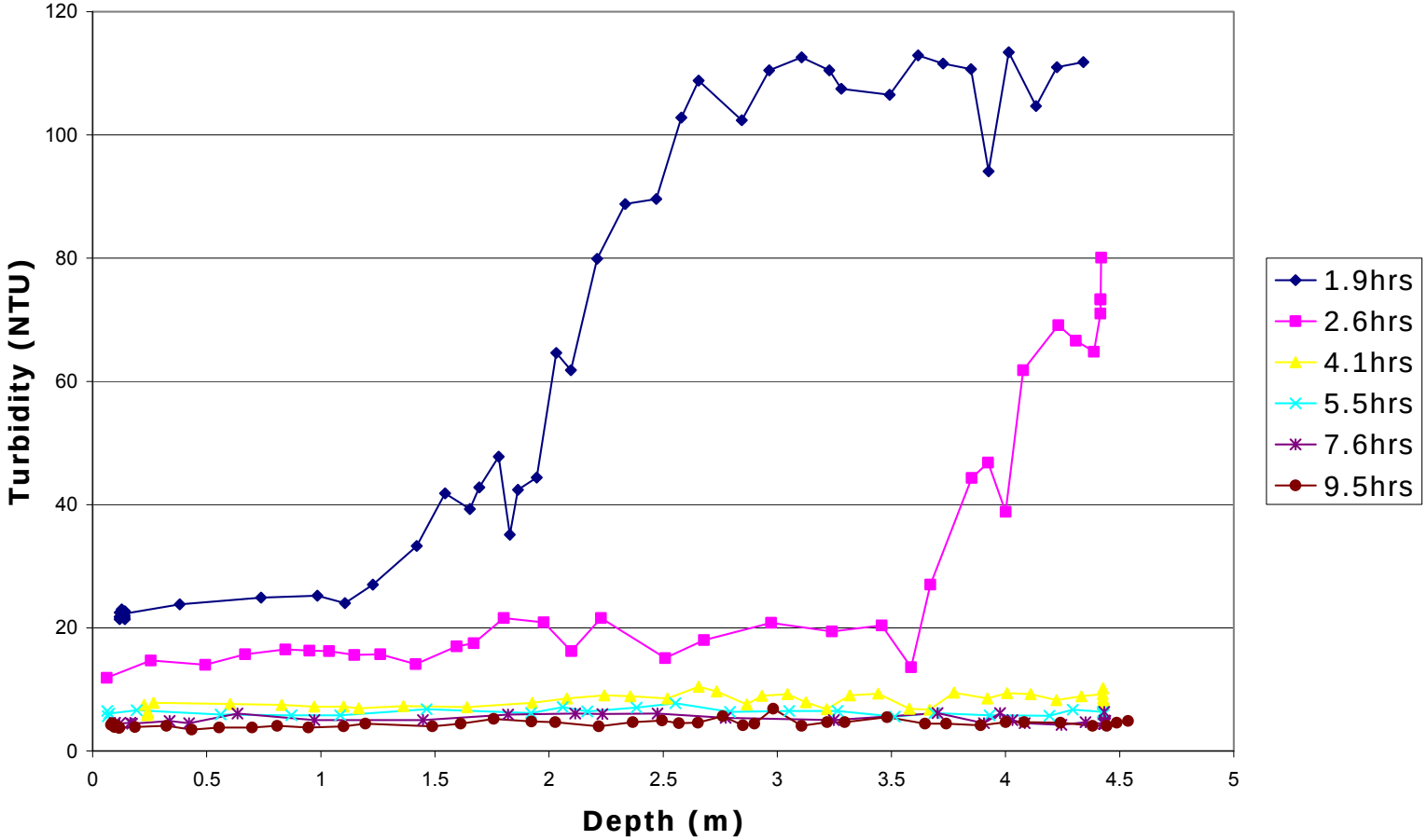
Turbidity Profile from Troll 9000



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Active Sludge Sampling

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Three men fishing

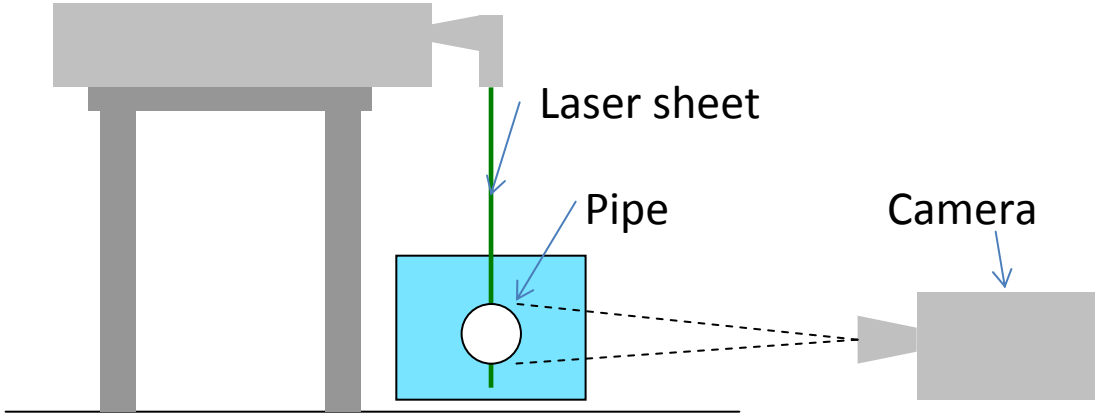
In situ Rheology

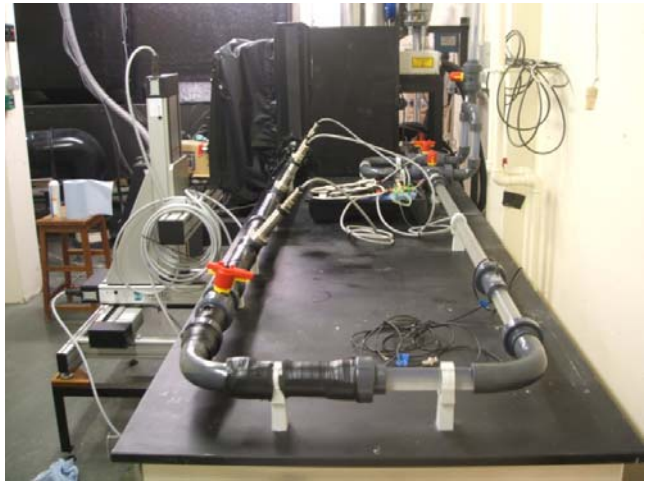
- Developing instrumentation to measure the rheological properties of slurries and pastes *in-situ* and continuously

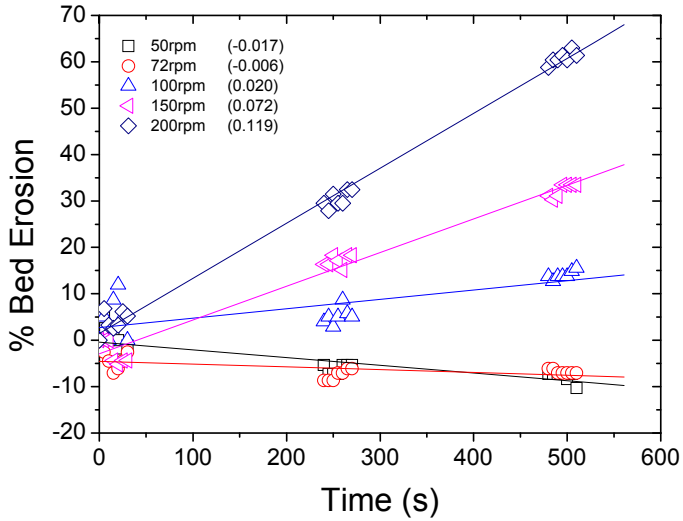
- Application in a wide variety of industrial sectors including Nuclear

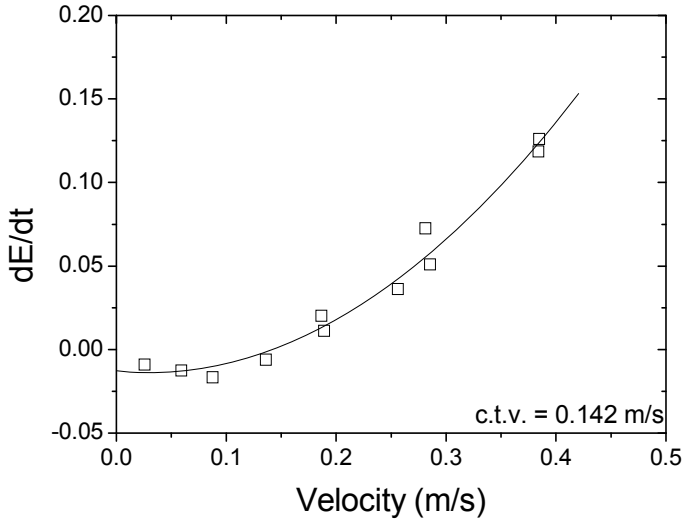
- Instrument characteristics:
  - Few mechanical parts
  - Low Cost
  - Portable
  - Compact design
  - Ease of operation

Sediment bed height as a function of time (Bed erosion) measured to determine the critical transport velocity. I.e. When bed erosion / time = 0. Image sequences where collected using a Particle Imaging Velocimetry (PIV) set-up.









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Management of solids

- Closely controlled process conditions
- Defined operating enveloped based on experience and lab work
- Techniques developed to deal with issues
  - Chemical dissolution
  - Invasive – High pressure jetting, low pressure washing deployment, pigs & rods, ultrasonics, etc
  - Computer modeling

Summary

- Overview of Plant and Processes
- Examples of solid formation, and issues
- Examples of characterisation and management of solids

Acknowledgement

- NDA and Site Licence Companies for funding the various studies
- Technical contribution: Steve Graham, David Harbottle, Simon Biggs, Tracy Ward, Mike Quayle, Donna McKendrick, Jeremy Hastings, Graham McKay, Andy Fellerman, Steve Thomson

- ~27 m3 of settled sludge transferred successfully from the fuel storage basin at the KE Reactor in preparation for D&D
- System/Equipment Problems caused delays in project execution
- Project Lessons Learned were compiled to relate the nature of the problems and how the problems were overcome

KBC-34834
Revision 0
EDC #: HNF-EDC-07-35133

KBC Project Hose-In-Hose Transfer Project Lessons Learned

Project No: A21 Document Type: TR Program/Project: KBC

G. A. Hofferber
Vista Engineering Technologies, LLC

J. C. Akers
M. Boger
G. A. Leshikar
J. F. Thompson
Areva

B. F. Barrett
S. P. Burke
C. R. Miska
Fluor Hanford, Inc.

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FLUOR
P.O. Box 1000
Richland, Washington

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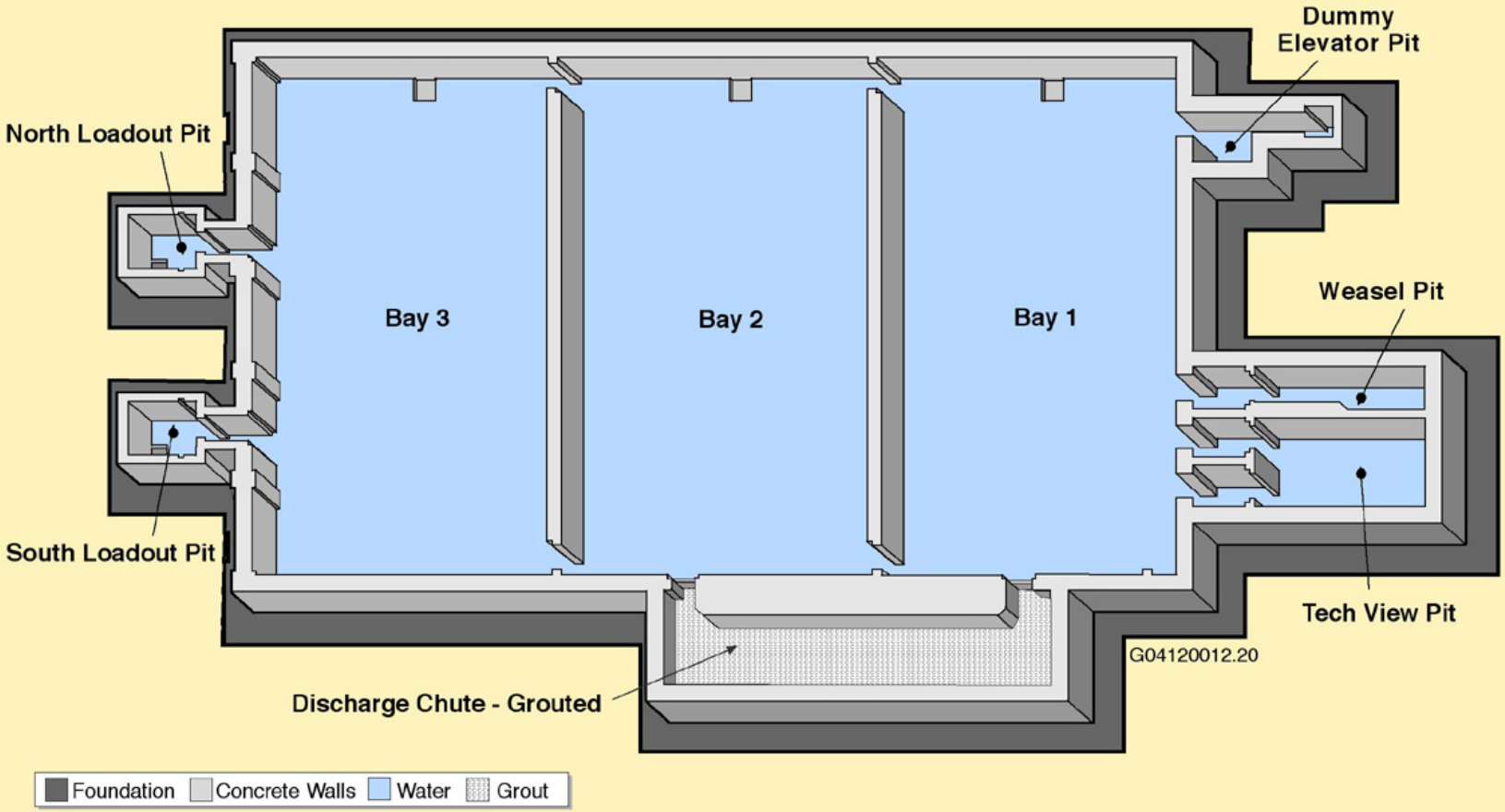
Content

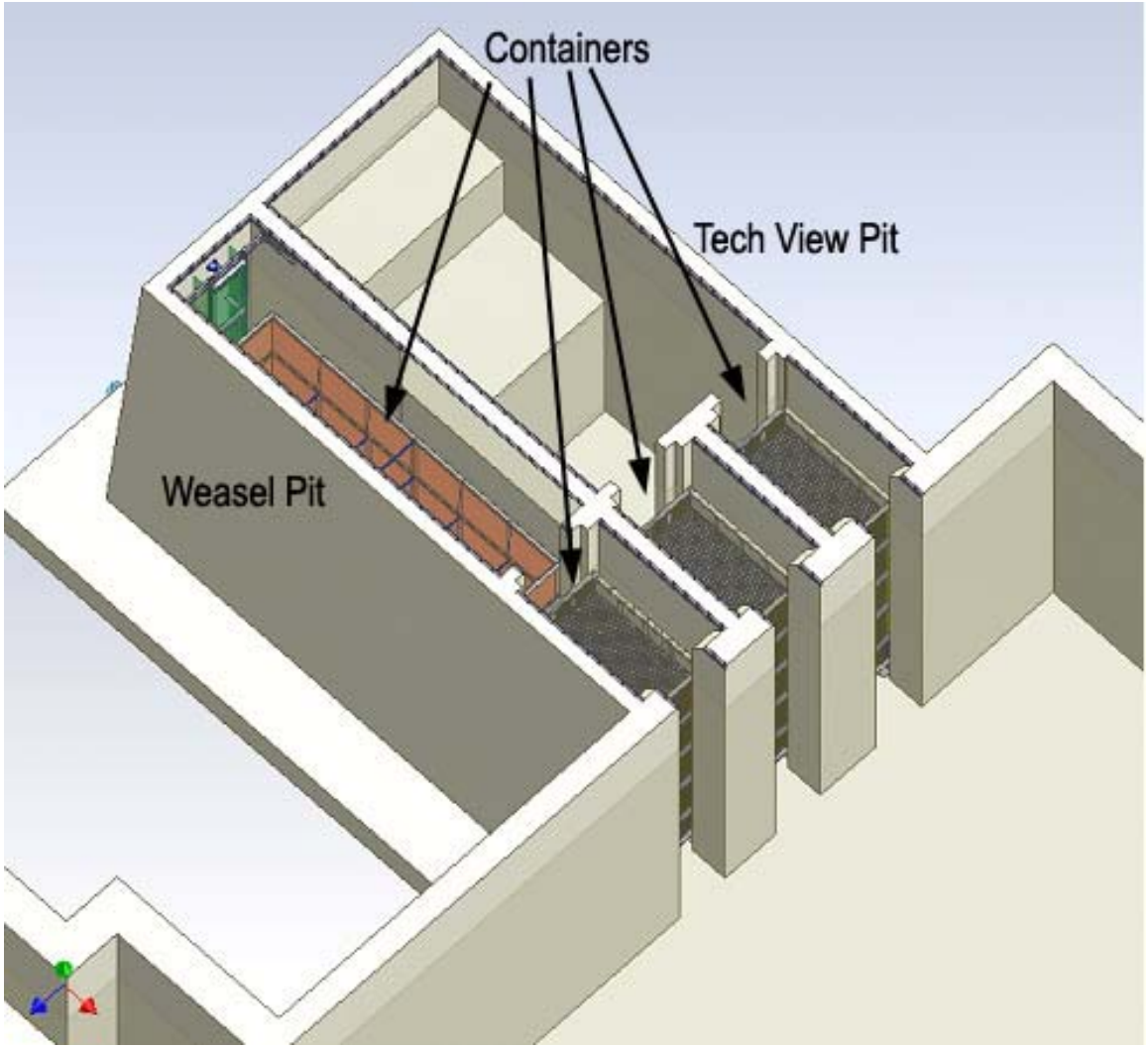
- Facility Description and History
- Sludge Composition
- Project Summary / Schedule
- Retrieval
  - Configuration Issues
  - Foreign Material in Sludge
- Transfer
  - Pump Fouling
  - Pump Wear
- Future K-Basin sludge handling activities

video

Schematic Overview of K East Storage Basin



Containers in KE



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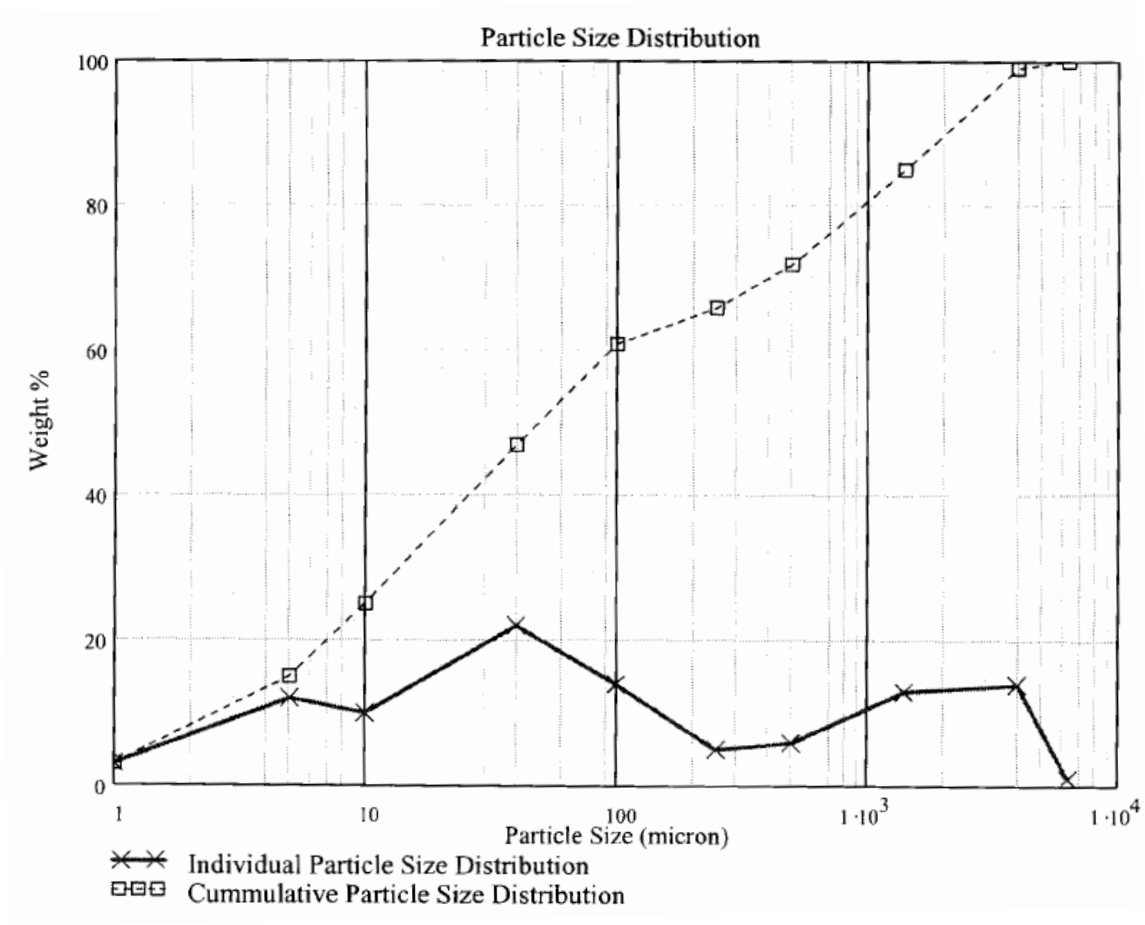
Sludge Composition

- Products of Fuel Corrosion during storage
  - Uranium Oxides
  - Uranium Metal pieces
  - Sand (blown in)
  - Spall from Concrete Basin Walls
- Insoluble
- Particle size distribution: < ¼”



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- Target slurry concentration ~2%weight
- Target system flow rate 64 gpm / 15 ft/sec
    - Driven by 1/4” diameter sG 19 particle
- Nominal Transfer hose ID = 1.25”
- Bottom Suction from KE container
- Dilution Control Automatic based upon solids % metering

- 7 centrifugal pumps in series – VFD Controlled (independently)
    - Suction Pump – Underwater in KE Basin
    - First Booster – Underwater in KE Basin
    - Four Above Ground Booster Pumps in Containment
    - Small Booster – Underwater in KW Basin
- Double Mechanical Seal w/ pressurized barrier fluid
- Installed Spare Pump in each station

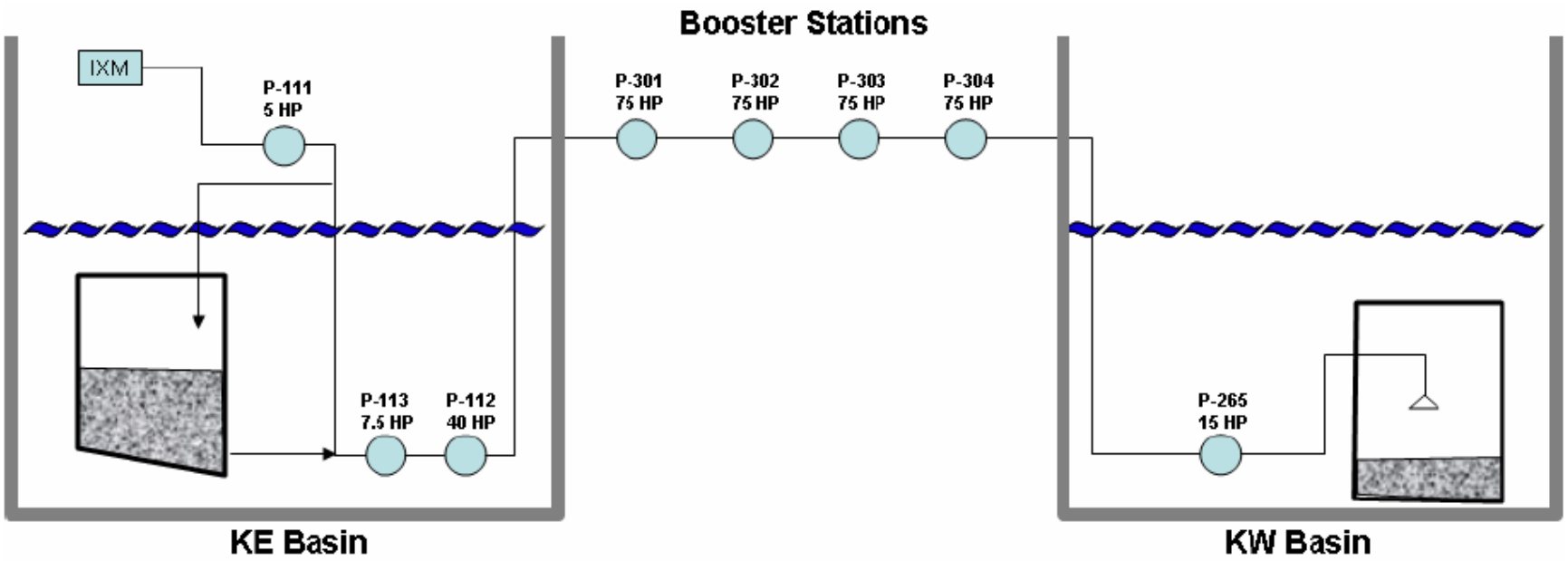

Figure 2-1 Simplified diagram of the HIH system

Testing

- Testing for slurry critical velocity and wear rates of hoses/fittings (Simulants)
- Sub-scale testing for centrifugal pump wear (Simulants)
- Full-scale Retrieval testing (Simulants)
- Integrated test of 4 Booster Stations (water only)
- Full-scale System Acceptance testing (water only)

Then… System Operation Begins

		SAT/SUN	MON	TUE	WED	THU	FRI	Percent Complete
Oct	14	off	OPERATIONS START CON-101	CON-101	FOCUS Day	Control Valve Slipping		4%
	21	off	<<<<<<<<<	<<<<<<<<<	FPSR DRY RUNS	>>>>>>>>>	>>>>>>>>>	4%
	28	off	<<<<<<<<<	<<<< FPSR Readiness Assessment >>>>			>>>>>>>>>	4%
Nov	4	off	P-265 Electrical Relay Problem		CON-101	Seal Pressure/Level Low - Re-seat		4%
	11	CON-101	CON-101	Indications of CON-101 plugging at tank outlet			FPSR	6%
	18	CON-102	CON-102	Conduit Nut found/ cleared in P-113	FPSR	Holiday		6%
	25	Additional blockage indicated - Back flush lines/pumps in KE - Found compression sleeve in P-113 discharge spool						6%
Dec	2	Install coarse In-line strainer	CON-102	Seal Pressure/ Level Low	CON-102	Recover from CON-102 Blockage		8%
	9	New coarse In-line strainer	CON-102	Difficulty with CON-102 retrieval. Attempted flushing.		Modified Dilution Strategy (secondary)		9%
	16	off	CON-102	CON-102	Resolved Locked Rotor at P-113		NCO training	10%
	23	off	Holiday		KW Crane work	Attempted Insertion of alternate coarse strainer		10%
	30	off	Holiday	CON-102	CON-102	Seal Pressure/ Level Low		16%
Jan	6	off	CON-102	CON-102	Resolved Locked Rotor at P-113	CON-102 First sign of noise (vibe) from P-302		19%
	13	Troubleshooting of P-302 vibration Aligned for use of P-332 (spare pump in booster station 2) Installed strainers in KE between P-113 and P-112						19%
	20							
	27	Disassemble P-302 spools Borescope investigation Aligned for P-331,333 / Installed Vibration transducers on pumps						19%
Feb	3							
	10							
	17	off	* Lowered Dilution Lance * Short in BS2 pump	CON-102	Establish Vibe limits for pump operation		CON-102	20%
	24	CON-102	CON-102	First indication of P-112 leak	Use air to find P-112 leak	Replace P-112		22%
Mar	3	Replace P-112		CON-102	CON-102	Realign for CON-101		25%
	10	CON-101	CON-101	CON-101	CON-101 dP issues with strainers	CON-101 First P-265 locked rotor	off	36%
	17	off	CON-101	CON-101	CON-101	P-265 unable be restarted	CON-101 P-265 bypassed	39%
	24	Gas bottle rack installed at booster stations	Line up to CON-104	CON-104	CON-104	CON-104	off	59%
Apr	31	off	CON-104	CON-104	Found second hole in P-112	UT for booster pump wear		68%
	7	Install new P-112	no HIH		CON-104	CON-104	off	74%
	14	CON-104	CON-104 video and sparging	CON-104	Line up to CON-103	CON-103	Moved coarse strainer	82%
	21	Video CON-104	CON-103	CON-103	CON-103	CON-103	FOCUS DAY	89%
	28	off	CON-103	Line up to CON-104	CON-104	CON-104	off	90%
May	5	off	CON-104	FTS Preps	CON-104	Realign to KW CON 240	off	92%
	12	off	FTS Load	Install Claw strainer in CON-101	CON-101	CON-101	Video CON-101 and CON-102	94%
	19	Video CON-102	CON-101	Line up and transfer CON-102	CON-102	Line up and transfer CON-104	off	97%
	26	Video CON-102	Solids Meter Cable Broken	CON-104	CON-104	CON-104	off	100%

Legend		
Days not available to HIH Operations	HIH System Operated	HIH Maintenance, Repair, Preparation

Figure 2-2 Hose-In-Hose Operations Timeline

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3. Lessons Learned Fact Sheets 6
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   3.3 Wear Failure of HIH-P-112 14
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   3.12 Heat Trace System 50
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   3.14 Spares Analysis and Supply 56
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Content

- Facility Description and History
- Sludge Composition
- Project Summary / Schedule
- Retrieval
  - Configuration Issues
  - Foreign Material in Sludge
- Transfer
  - Pump Fouling
  - Pump Wear
- Future K-Basin sludge handling activities

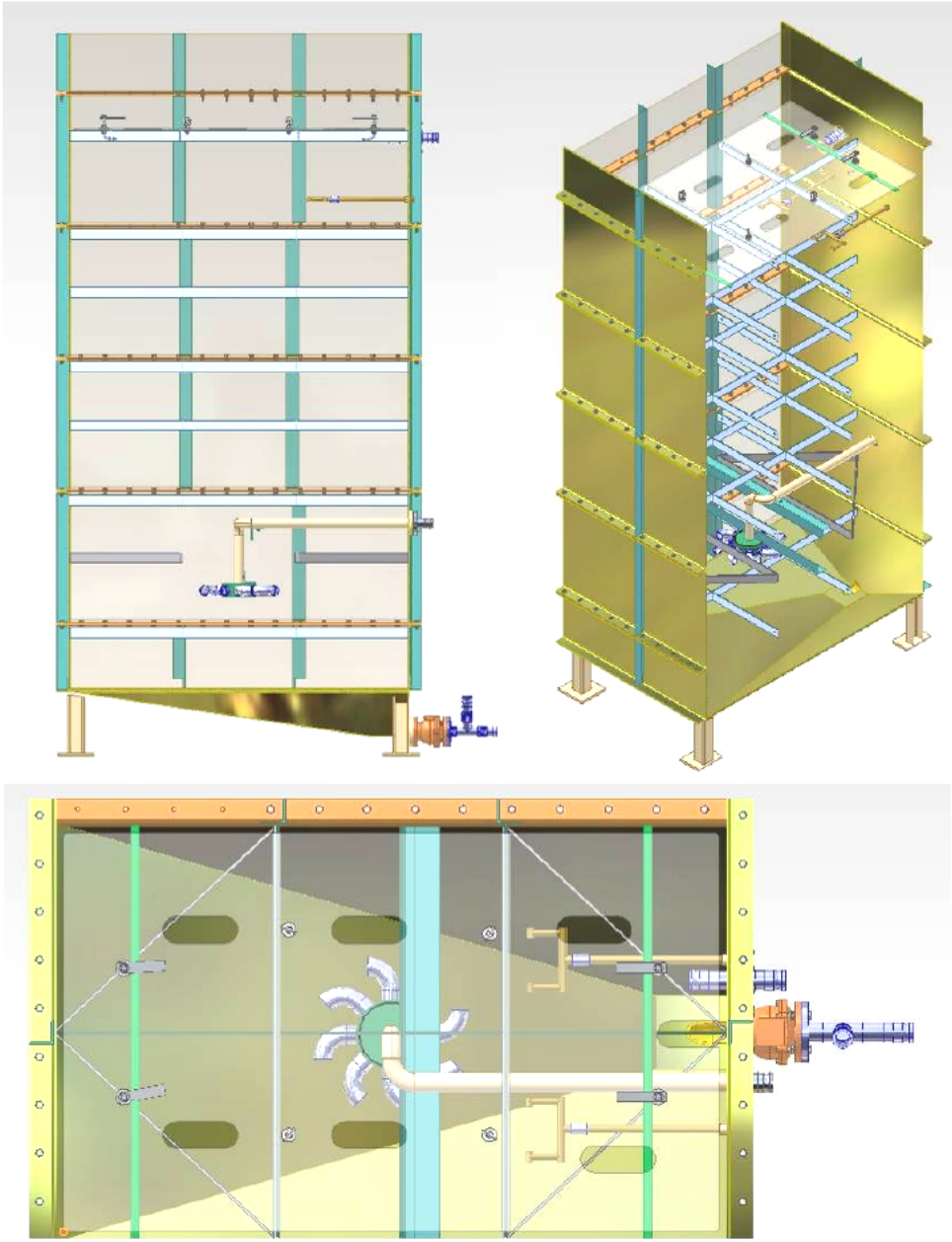

Figure 3-25 Side, Top, and Isometric Views of a Typical KE Container

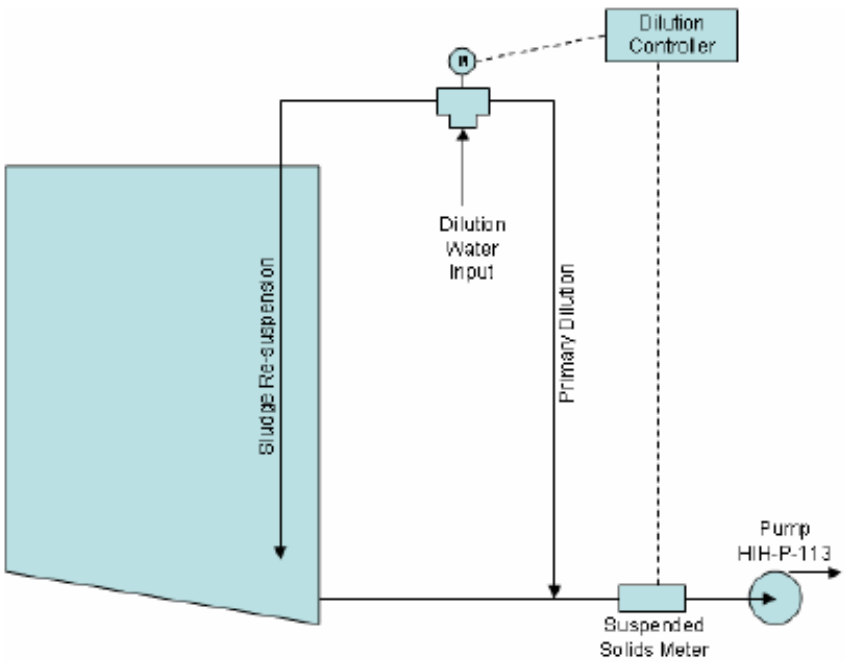

Figure 3-11 System Configuration Before and After Addition of In-line Coarse Strainer

- Construction Changes approved that moves dilution point further away from container (ease of operations issue)
- Foreign Material Inhibits suction line and fouls suction pump
- System trips due to insufficient suction pressure

Content

- Facility Description and History
- Sludge Composition
- Project Summary / Schedule
- Retrieval
  - Configuration Issues
  - Foreign Material in Sludge
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  - Pump Fouling
  - Pump Wear
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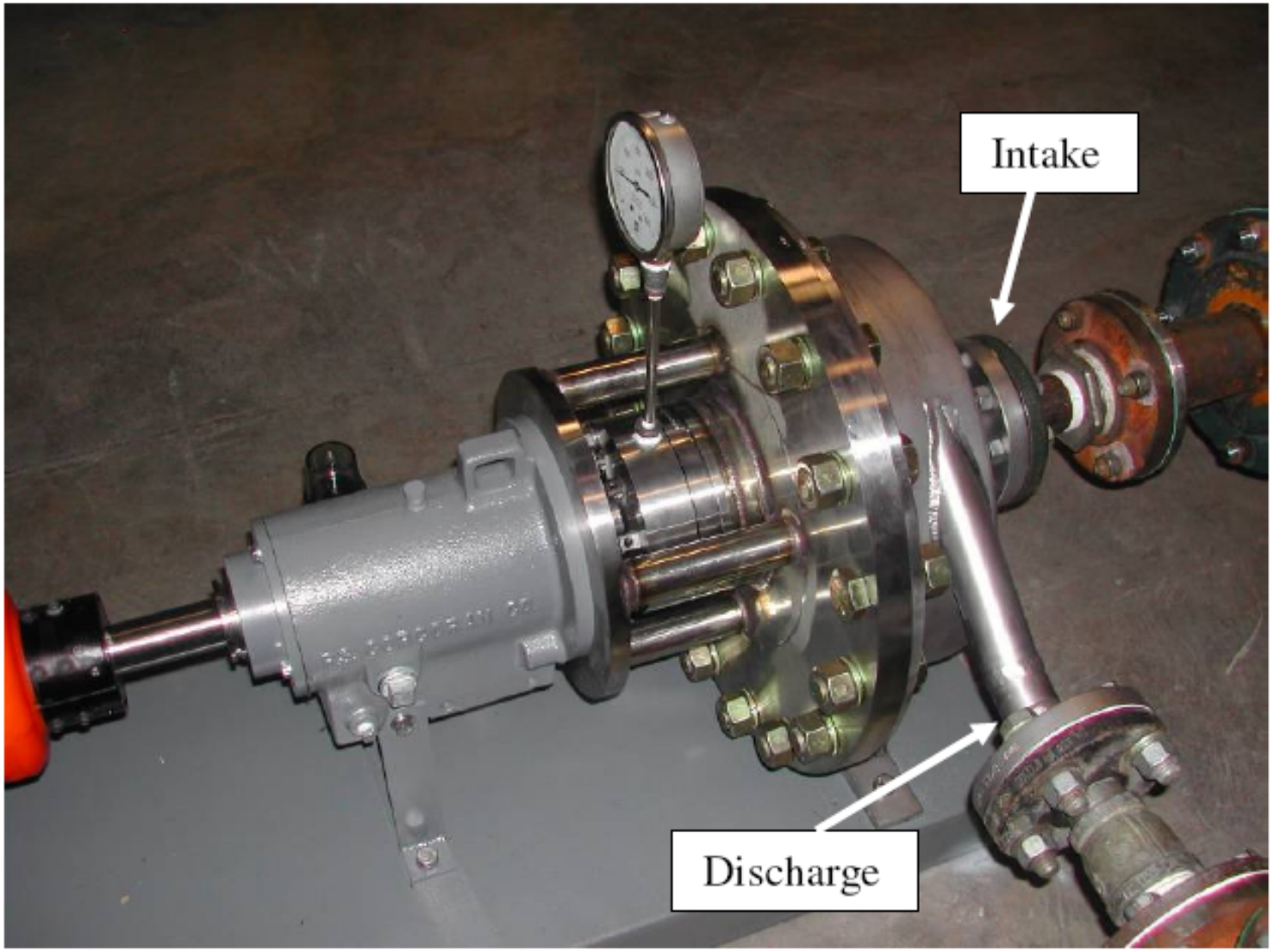

Figure 3-14 HIH Booster Pump

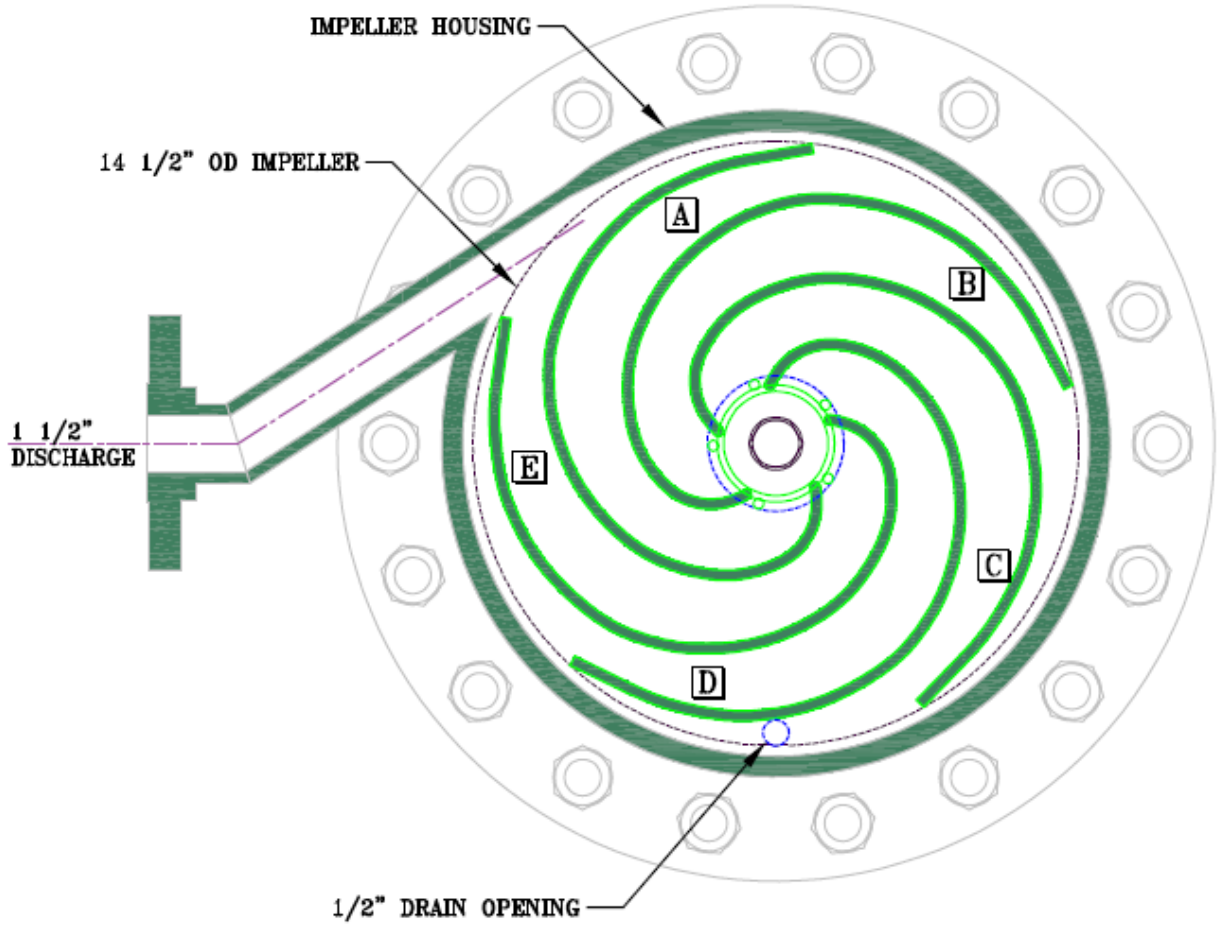

Figure 3-15 Map of R.W. Corcoran Impeller

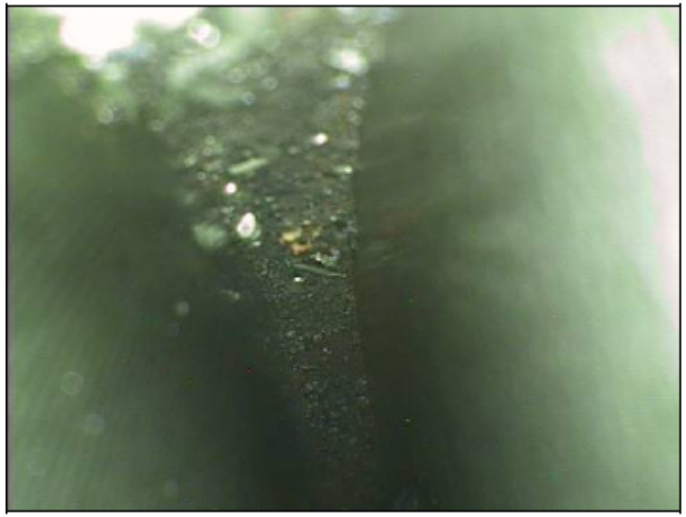

Figure 3-18 Impeller Cavity "A"

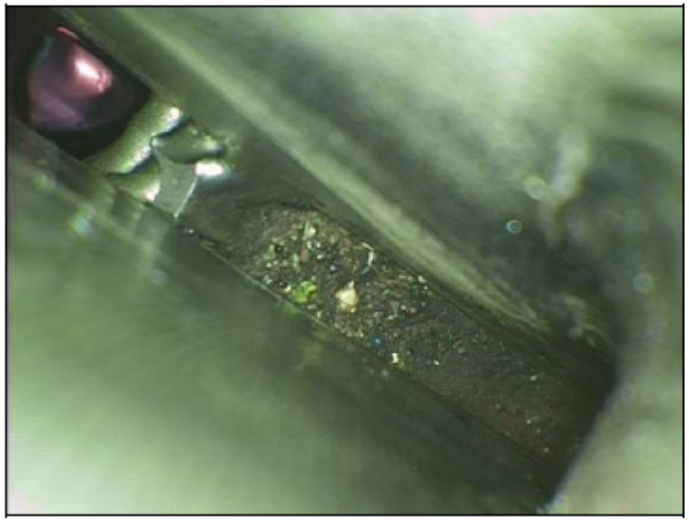

Figure 3-19 Impeller Cavity "E"

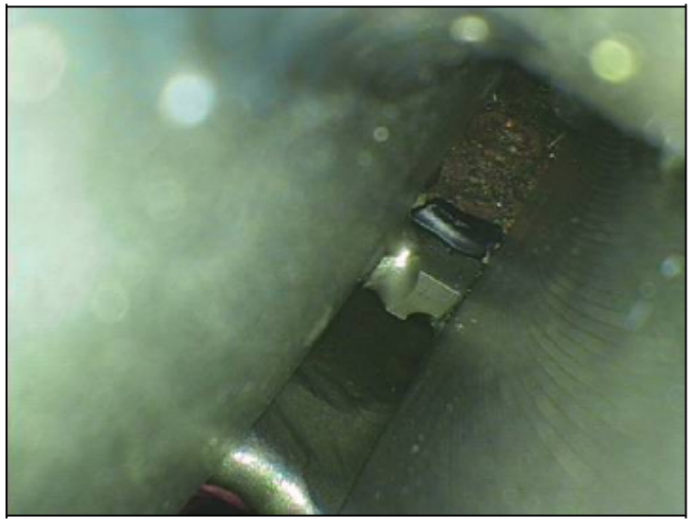

Figure 3-20 Impeller Cavity "C"

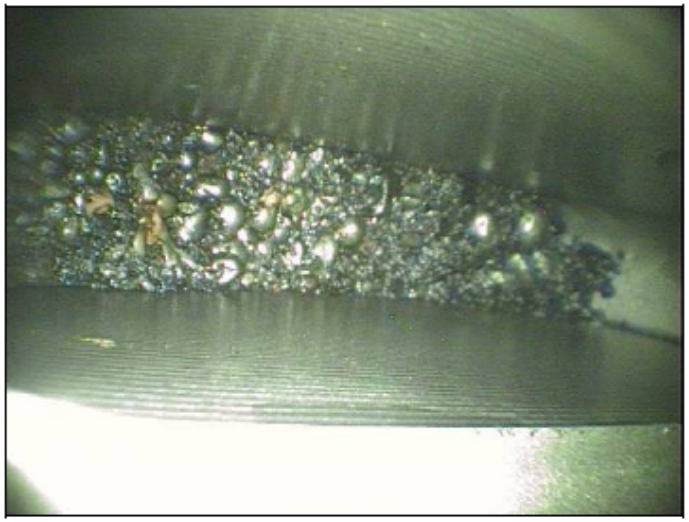

Figure 3-21 View From Impellor Eye

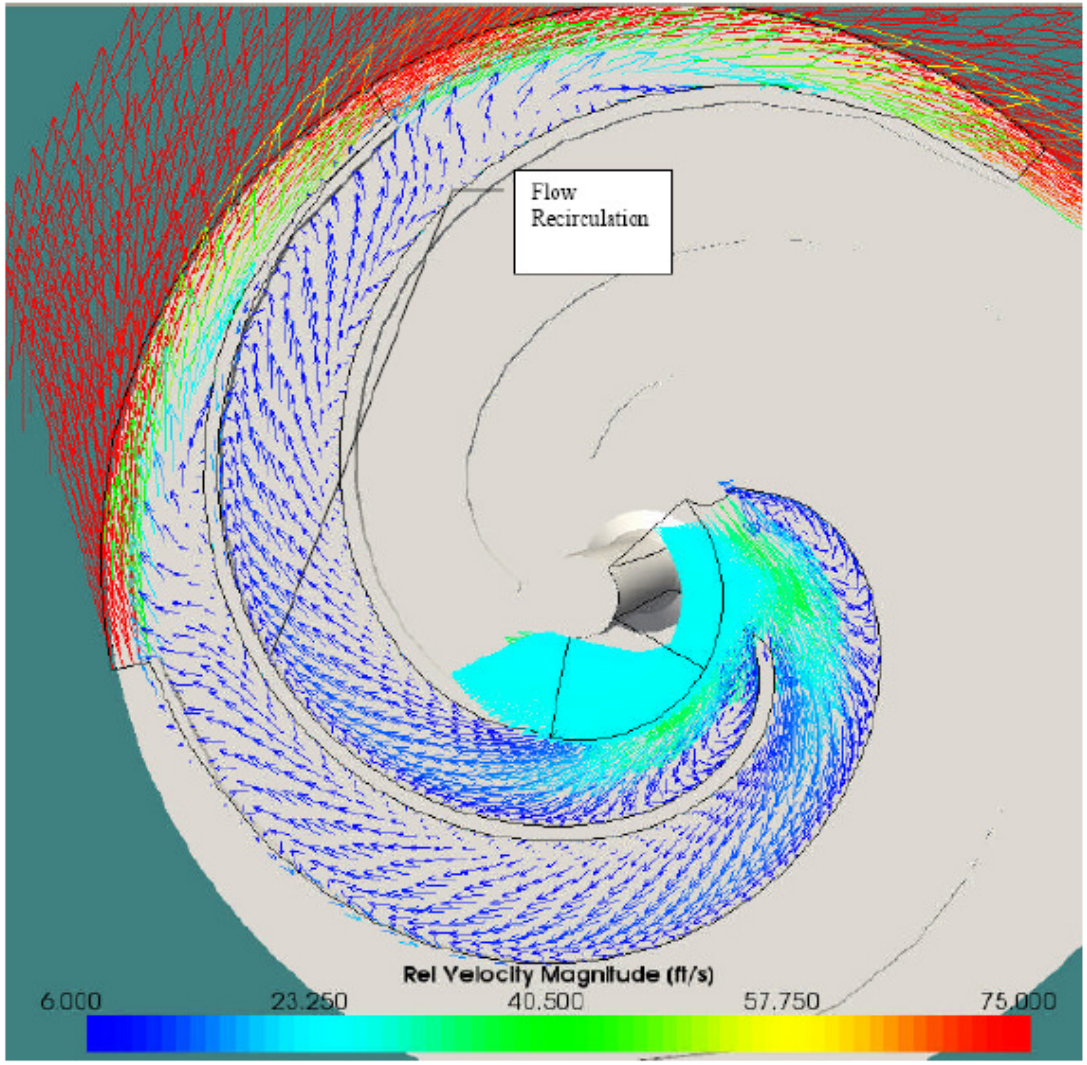

Figure 3-24 CFD-generated Relative Velocity Vector Diagram

225°
2-7
1-19
1-20
1-21

Test Results Drove Requirement for Monitoring of wear using UT thickness sensors fixed to pump and read remotely

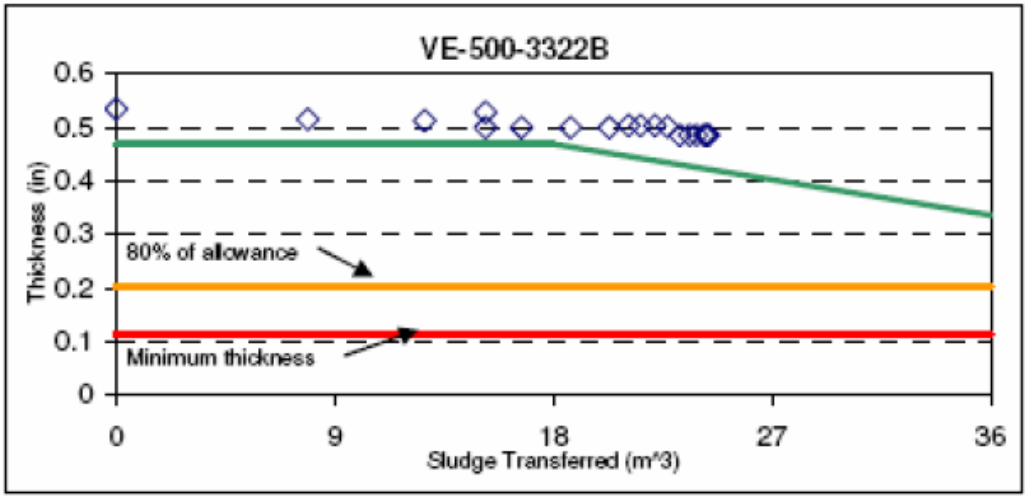

Figure 3-3 Wear at One Location on HIH-P-332

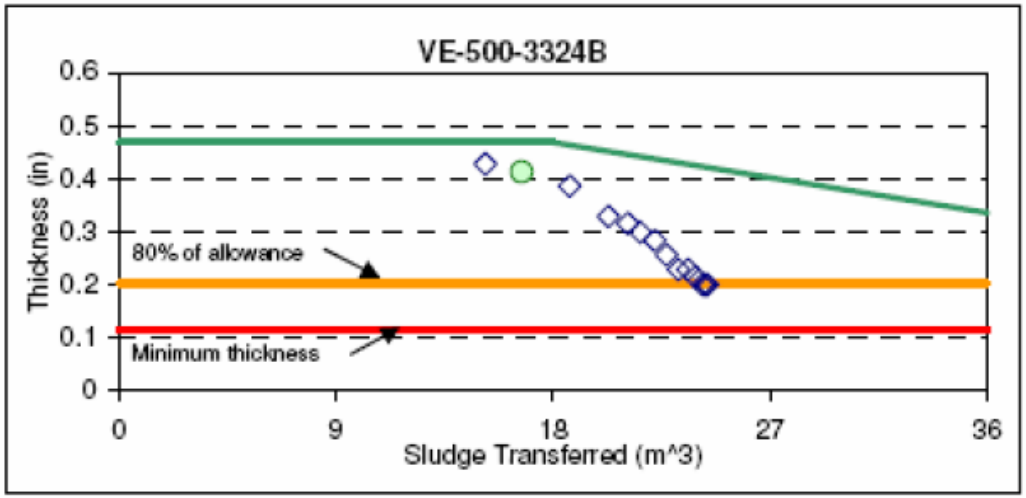

Figure 3-4 Worst Case Wear Location on HIH-P-332

Underwater Booster Pump experienced multiple wear failures

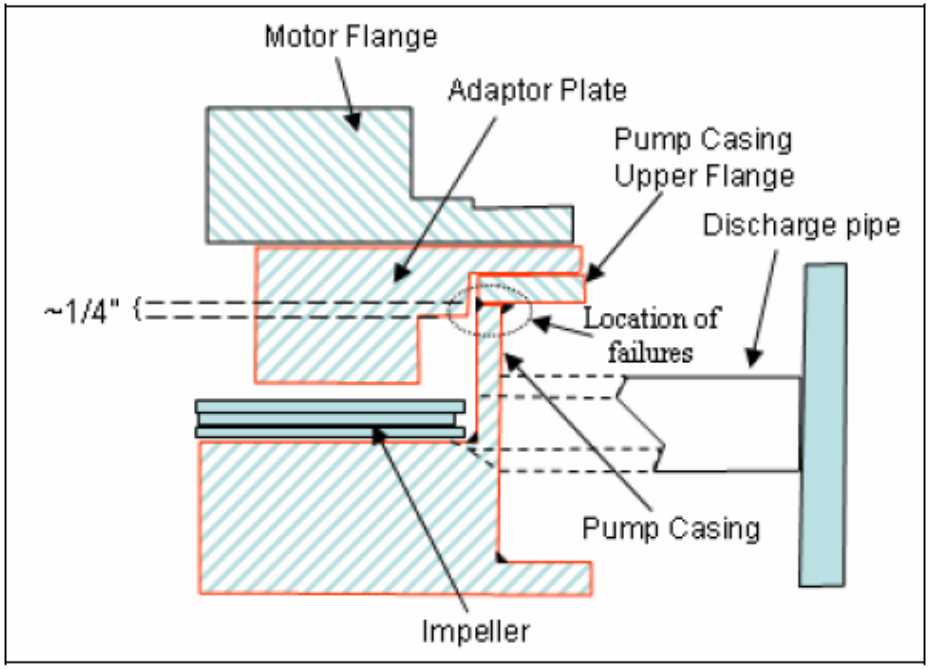

Figure 3-6 HIH-P-112 Cut-away View

Content

- Facility Description and History
- Sludge Composition
- Project Summary / Schedule
- Retrieval
  - Configuration Issues
  - Foreign Material in Sludge
- Transfer
  - Pump Fouling
  - Pump Wear
- Future K-Basin sludge handling activities

Future K-Basin sludge handling activities

- Completed Sludge Vacuuming in the K WEST basin as well
- KE and KW floor and Pit Sludge is containerized in KW
- Additional streams held in water treatment components in KW
- Sludge Treatment Project is in development to retrieve and treat the containerized sludge streams for disposal
- KE is being de-watered and prepared for D&D

On the Formation and Treatment of Trisodium Phosphate Plugs

J. S. Lindner, Institute for Clean Energy Technology

R. K. Toghiani, Swalm School of Chemical Engineering and ICET

V. K. Raju, currently at Boehringer Ingelheim Roxane Inc

We gratefully acknowledge support from the U. S. Department of Energy Cooperative Agreement No. DEFCO106EW07040-06040310

Background and Motivation

- Hanford Saltwell Supernatant Transfers
  - 0.4-5gpm 3 inch mild steel heat traced pipe
  - Re=dV$\rho/\mu = 180–1800$
  - Junction Boxes untraced
  - Plugs from SX-104, U-103, BY-102 $2.4 million impact
- Actual SX-104 Supernatant Analysis
  - Herting PLM Cloudy at 25°C, Gel at 23°C
  - Steen [PO_4] =0.044M

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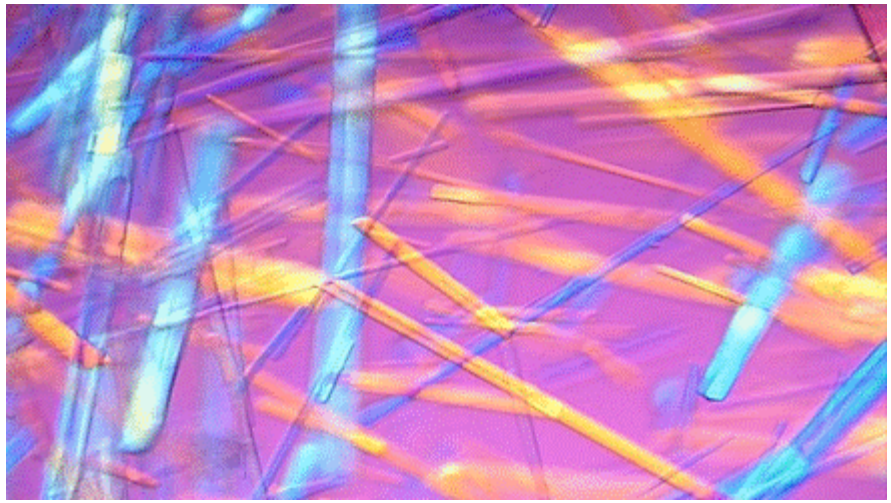
Background and Motivation

- Investigate Plug Formation SX-104 Surrogate
  - Construct Lab-Scale Flow Loop based on Re
  - Develop Operating Envelope, V, T, [PO_4]
- Examine unplugging options



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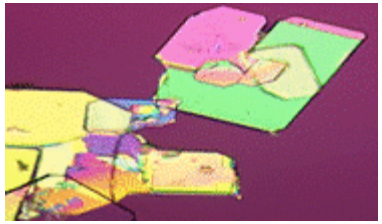
Solubility Studies and Database Development



- Environmental Simulation Program (ESP, OLI Systems Inc.) - aqueous thermodynamic electrolyte model for solid-liquid equilibria (SLE)
- Selected at Hanford (and SRS) as a predictive engineering tool
  - reduce costs of analytical characterization
  - identification of compositions that could result in difficulties during pretreatment and retrieval operations
  - Flowsheet Development – Unit Operations
- Data gaps found for many chemical systems contained in Hanford wastes

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Validation Issues

- Quality of the fundamental thermodynamic data
- Direct comparisons to experimental results
- Evaluation against other models
- Code will never be fully validated
  - interactions between all possible component combinations
- Focus on predominant systems

Development Path

- Comparison of simulation results to Hertings' core salt cake dissolution experiments
- Fundamental solubility experiments – Database Development
- Direct comparisons to other experimental results (FIU tall column experiments)
- Code will never be fully validated
  - interactions between all possible component combinations

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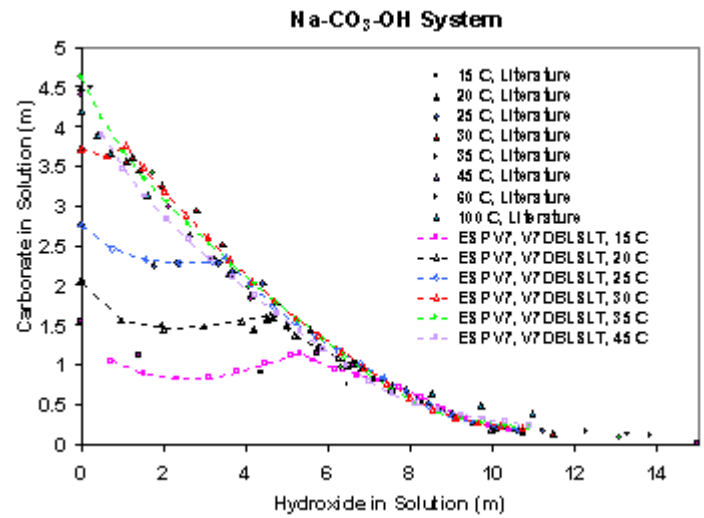
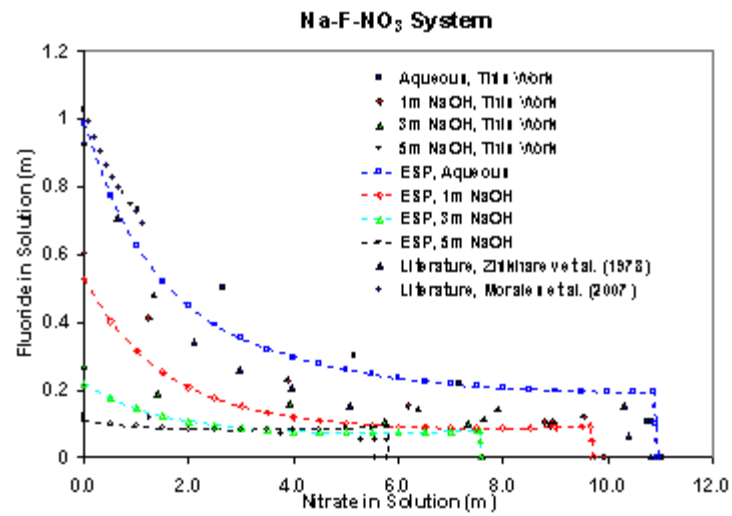
Regression Fits and Database Development

$Na\text{-}F\text{-}NO_3$	$Na\text{-}NO_2\text{-}CO_3$	$Na\text{-}SO_4\text{-}PO_4$
$Na\text{-}F\text{-}SO_4$	$Na\text{-}NO_3\text{-}CO_3$	$Na\text{-}Al\text{-}NO_3$
$Na\text{-}F\text{-}PO_4$	$Na\text{-}NO_2\text{-}SO_4$	KNO_3
$Na\text{-}PO_4\text{-}NO_3$	$Na\text{-}NO_3\text{-}SO_4$	$CsNO_3$
$Na\text{-}F\text{-}PO_4\text{-}NO_3$	$Na\text{-}SO_4\text{-}CO_3$	



- DBLSLTDB double salt database for use in ESP 6.5
- Attempted to port to ESP version 6.7
- Ported to ESP version 7.0 V7DBLSLT
- After fitting the data for 3 different versions of ESP initiated subcontract with OLI Systems Inc.
- Data will now be in a <u>permanent</u> repository
- Release from OLI Systems Inc., 1st Qtr 2008



Development of Surrogate

SX-104 Supernatant Surrogate	ORNL	Sample 5	Sample 8
moles Sodium salt			
Aluminate	1	1	1
Nitrate	7	7	7
Hydroxide	2	2	2
Phosphate	0.2	0.3	0.3
Carbonate	0.4	0.4	0.1
moles H2O	55.51	55.51	55.51

Laboratory Scale Saltwell Pumping Flow Loop

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Temperatures and Pressures Observed Following Activation of the Heat Exchanger

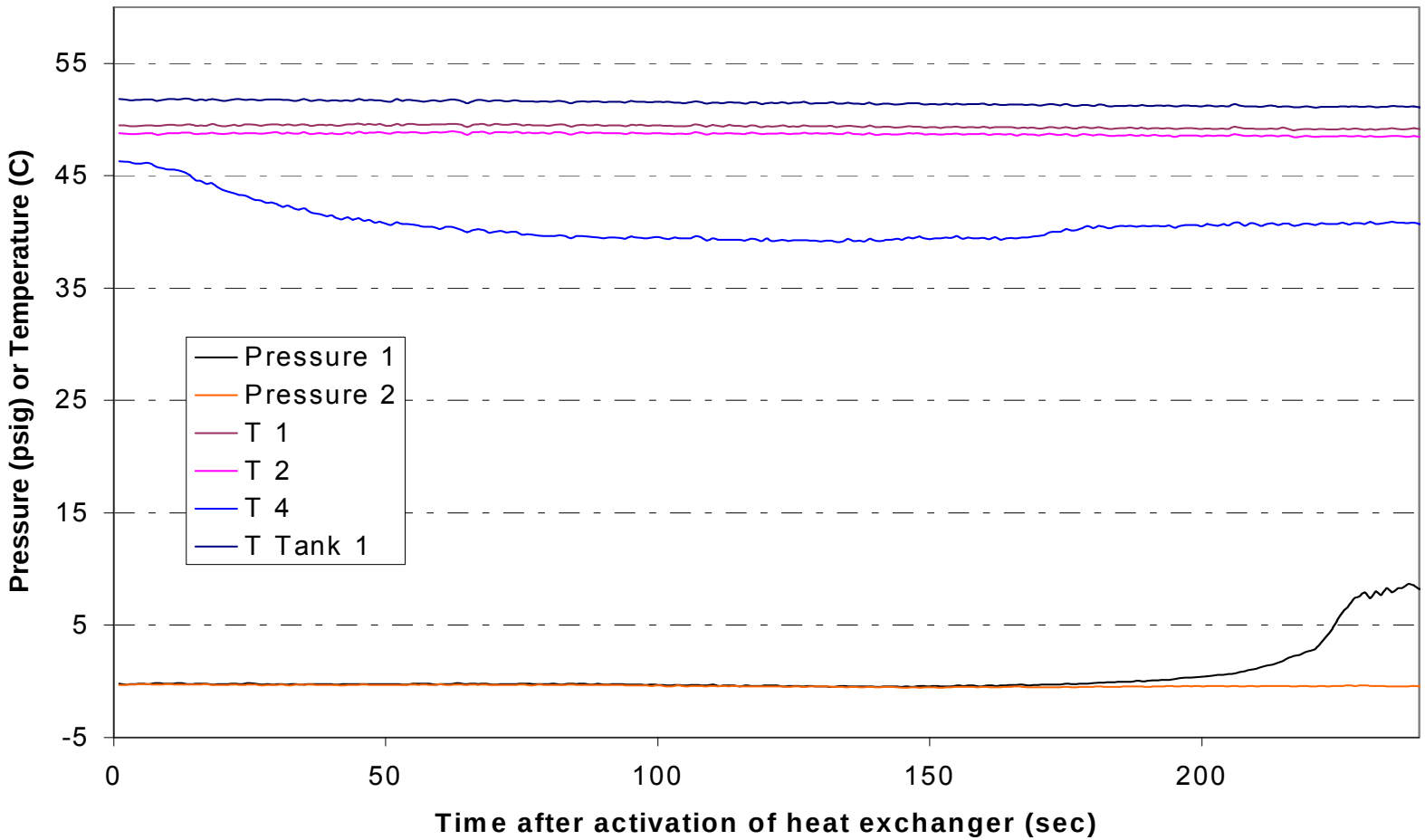
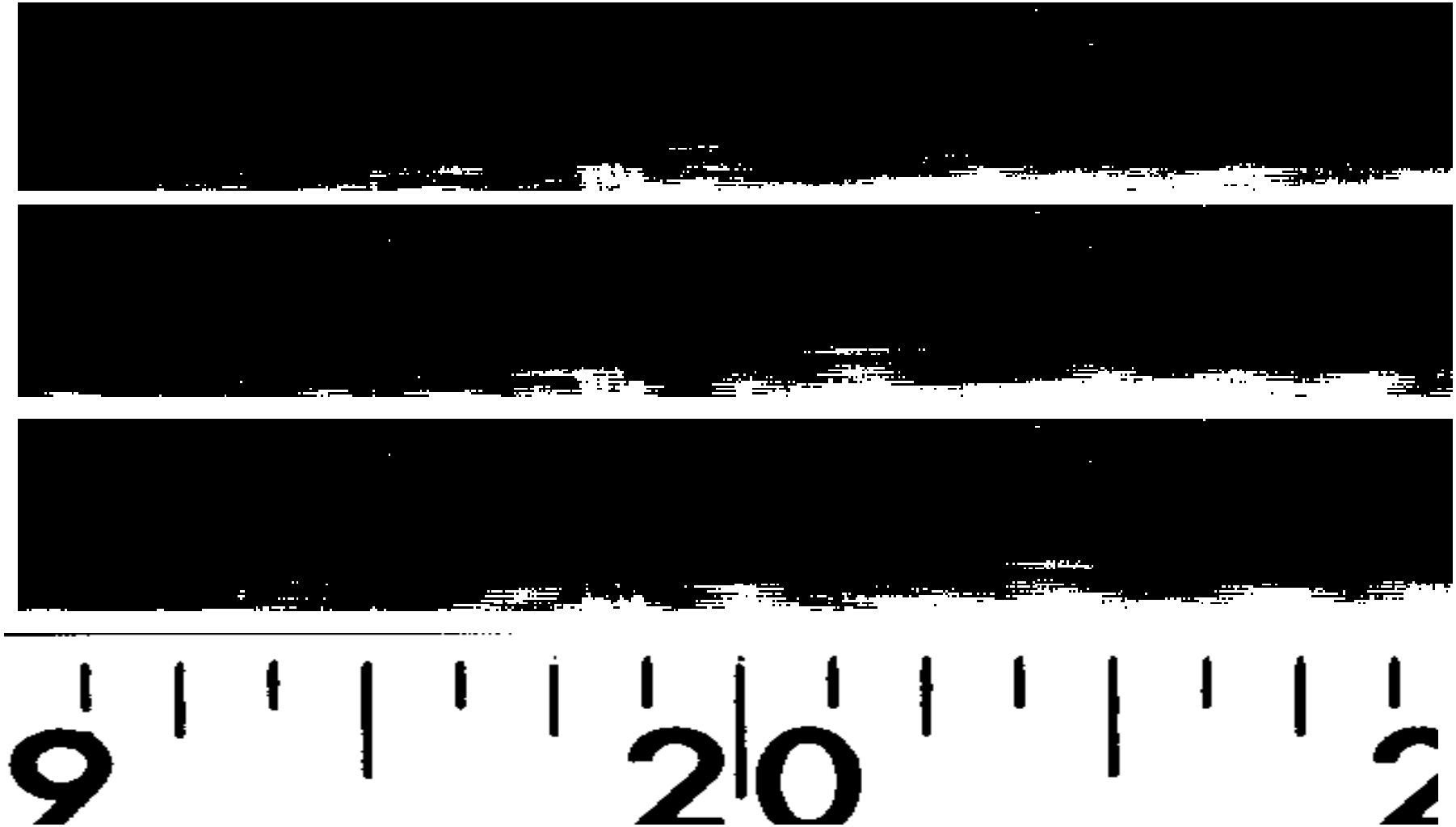


Image Sequence of Particle Growth



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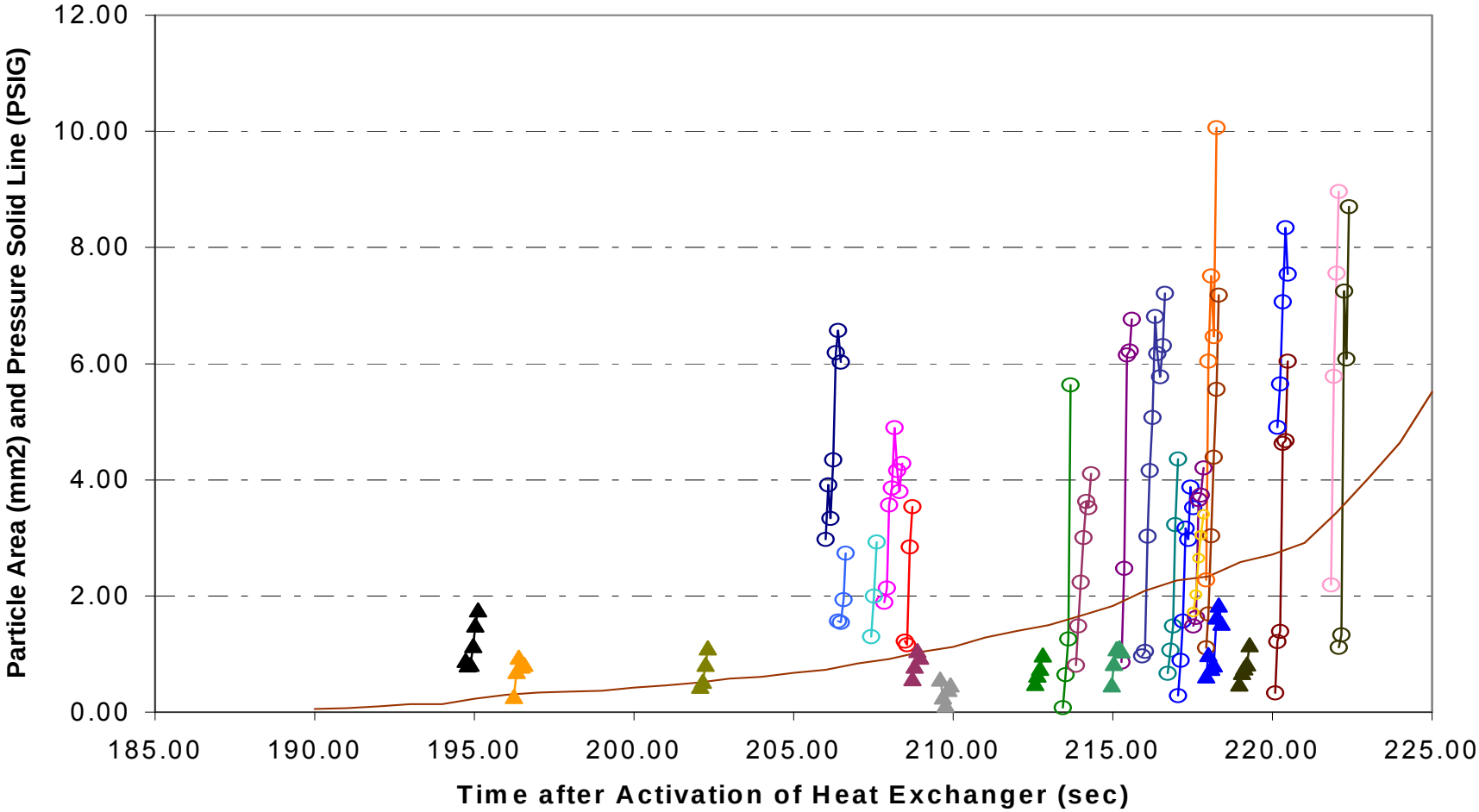
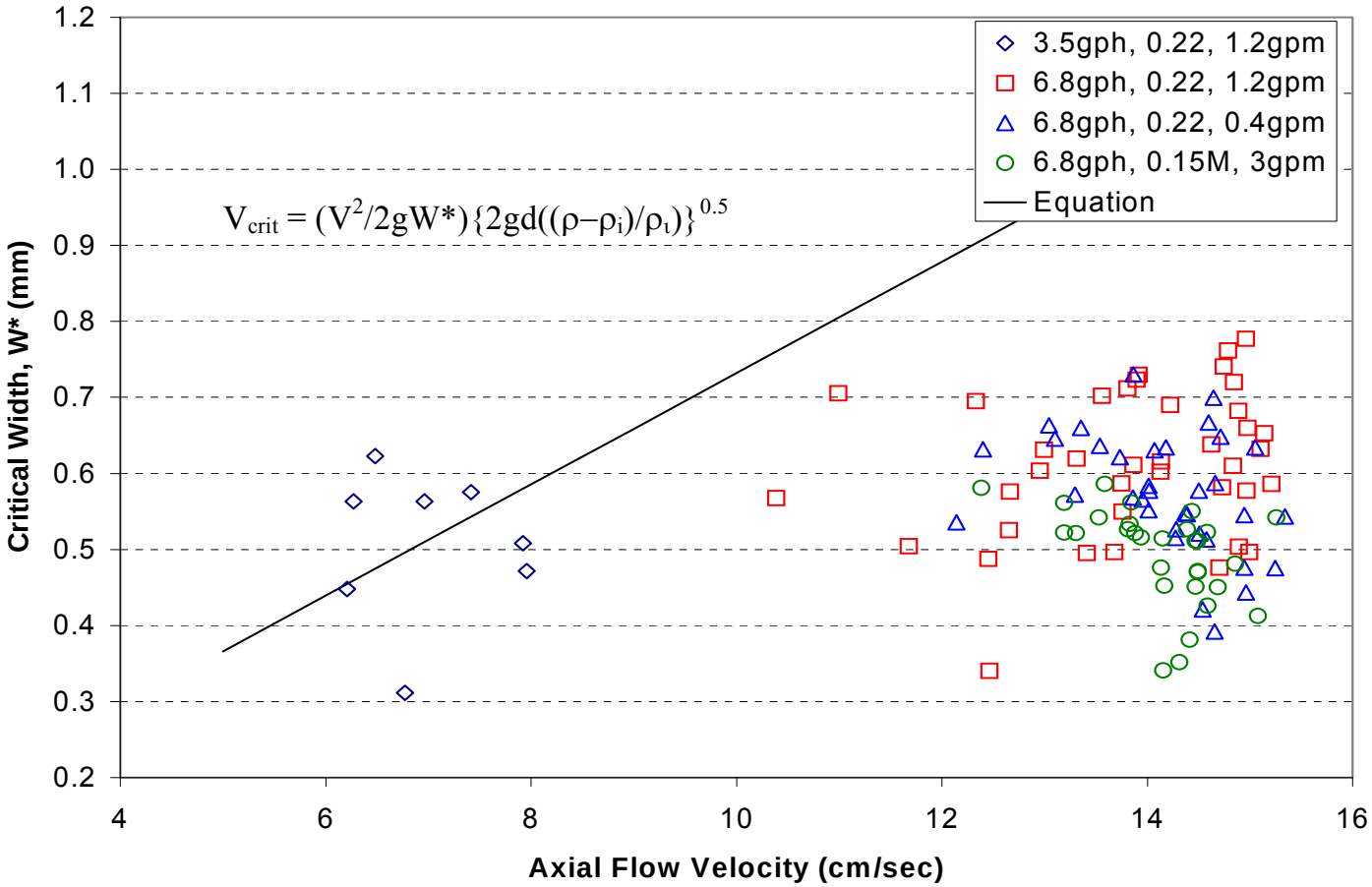
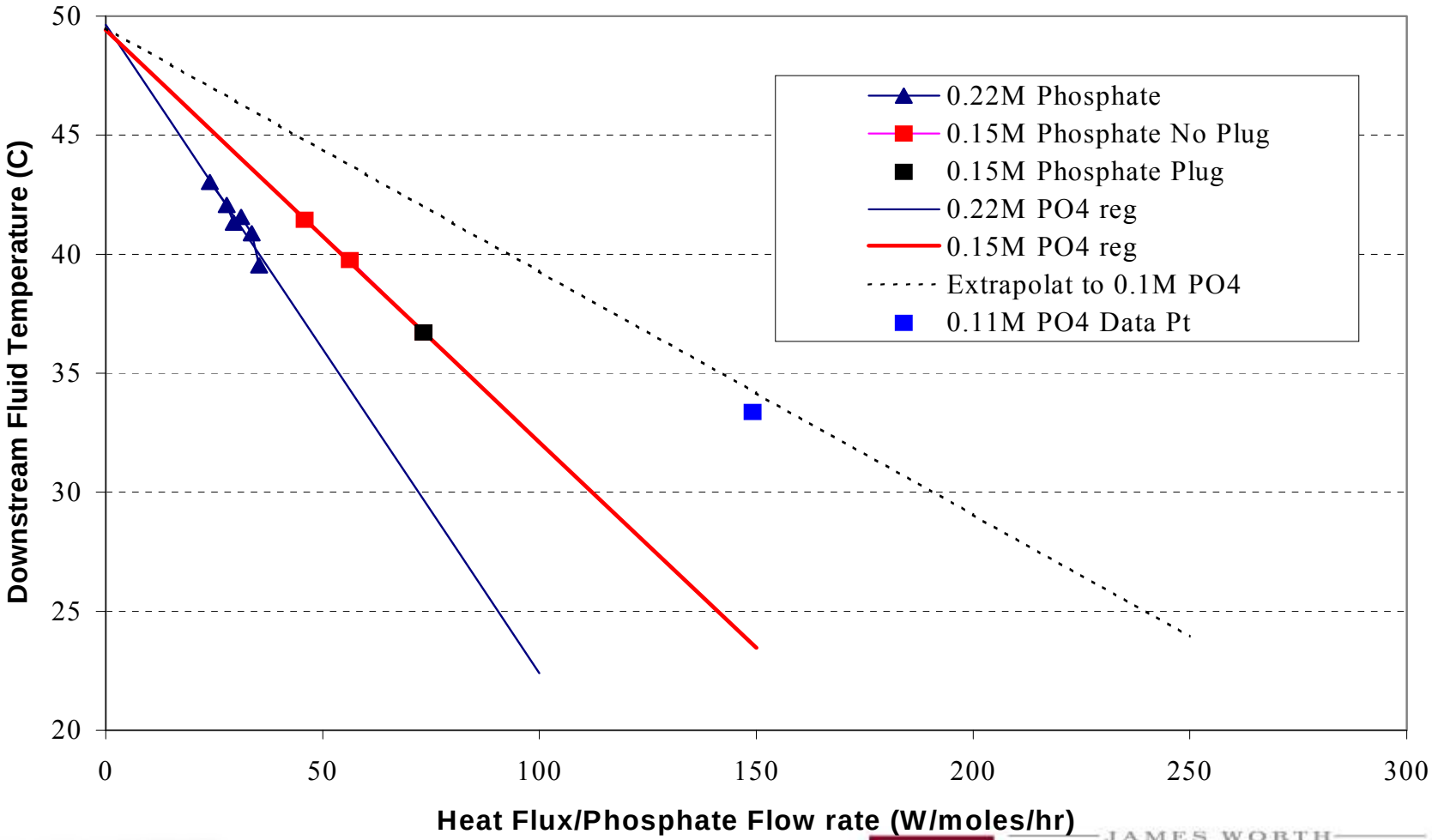
Single Particle (triangles) and Agglomerate (open circles) Areas and Growth



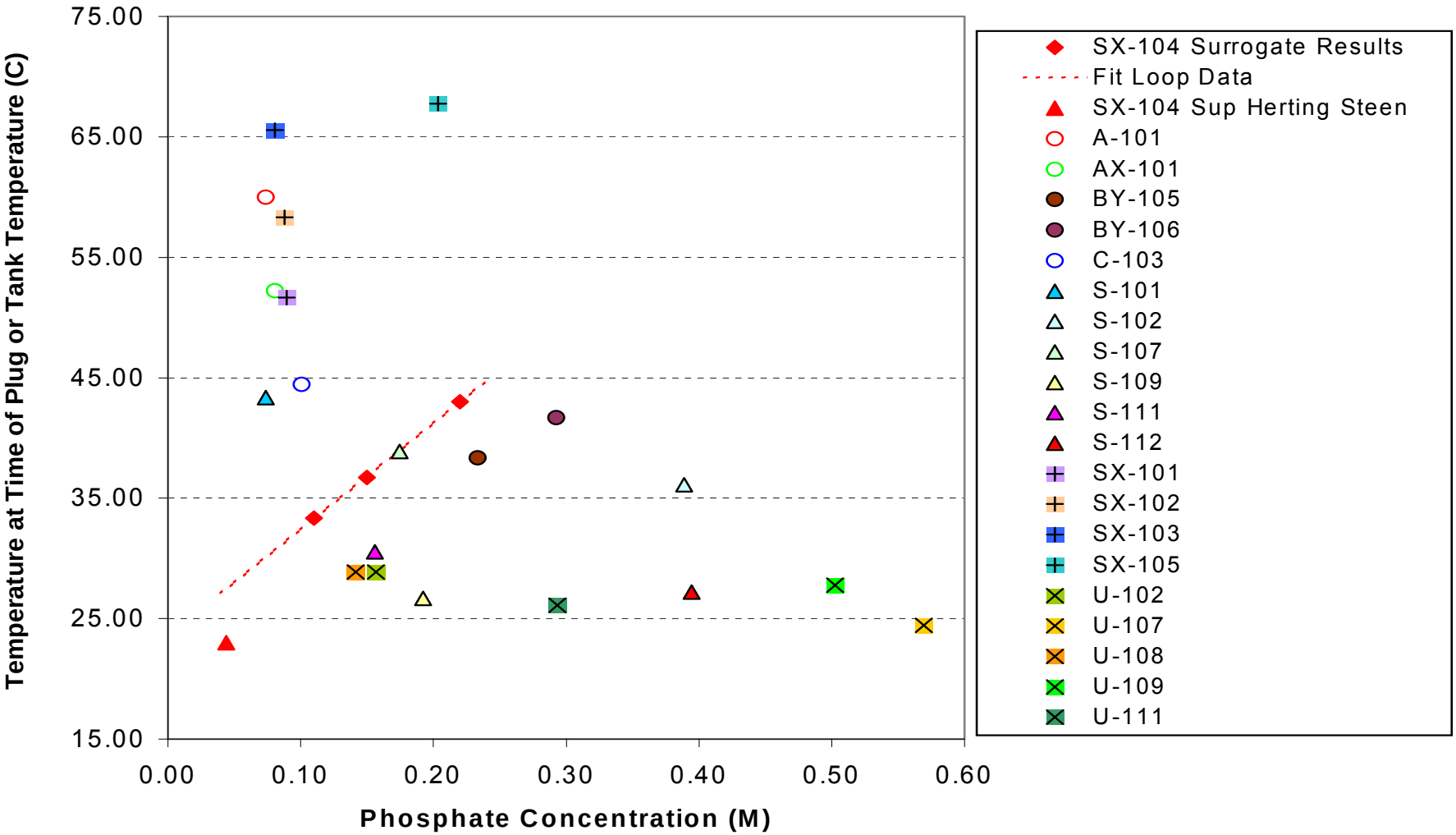
Measured Single Particle and Calculated Critical Deposition Widths



Plug Formation Depends on Phosphate Loading and Cooling Rate



Loop Data Extrapolated to Actual SX-104 SW Supernatant Non-Stabilized SST Waste Temperatures and [PO_4] (BBI)



Key Findings for Interim Stabilization Transfers

- Growth Rates of single and agglomerate sodium phosphate dodecahydrate particles can approach 2 and 18mm^2/sec respectively.
- Bed development commences when the particles or agglomerates exceed a critical size and settle.
- Plug formation can occur at temperatures as high as 43°C.
- An operating envelope for waste transfers based on the phosphate loadings in the tanks and temperature drops that may be encountered during routing has been advanced.

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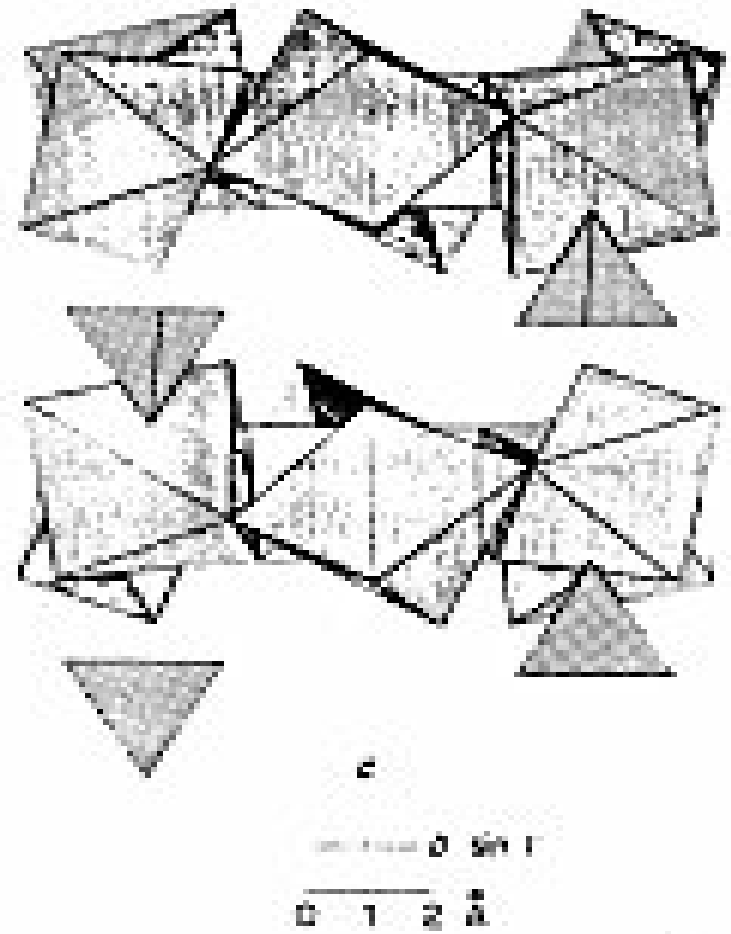
$Na_3PO_4 \cdot 12H_2O$ Crystal Structure

Tillmans, E., & W. H. Baur, Inor. Chem, 9, 1957 (1970).

- Sheet with composition of $Na(OH_2)_4$ sharing corners to form rings with each ring connected to other rings by common edges
- Connections between sheets from hydrogen bonds from the water molecules to the phosphate groups located between the sheets
- Disrupt the hydrogen bonding fracture the structure



On the use of Carbonate Anion for Phosphate Plug Dissolution

- Previous Work - "A novel reaction between carbon dioxide and trisodium orthophosphate dodecahydrate shown by photoelectron spectroscopy and X-ray diffraction,"
  Belton, P. S. et al., J. Inor. Nucl. Chem., 43, 614 (1981).

- $\mathbf{CO_{2(g)} + Na_3PO_4{\cdot}12H_2O_{(s)} \rightarrow Na_2HPO_4{\cdot}7H_2O_{(s)} + NaHCO_{3(s)} + 4H_2O.}$

- Unique reaction between a gas and a solid forming two solids.

- Phosphate plugs formed in test loop allowed same 2-hour residence time followed by addition of xM Na_2CO_3 solution.

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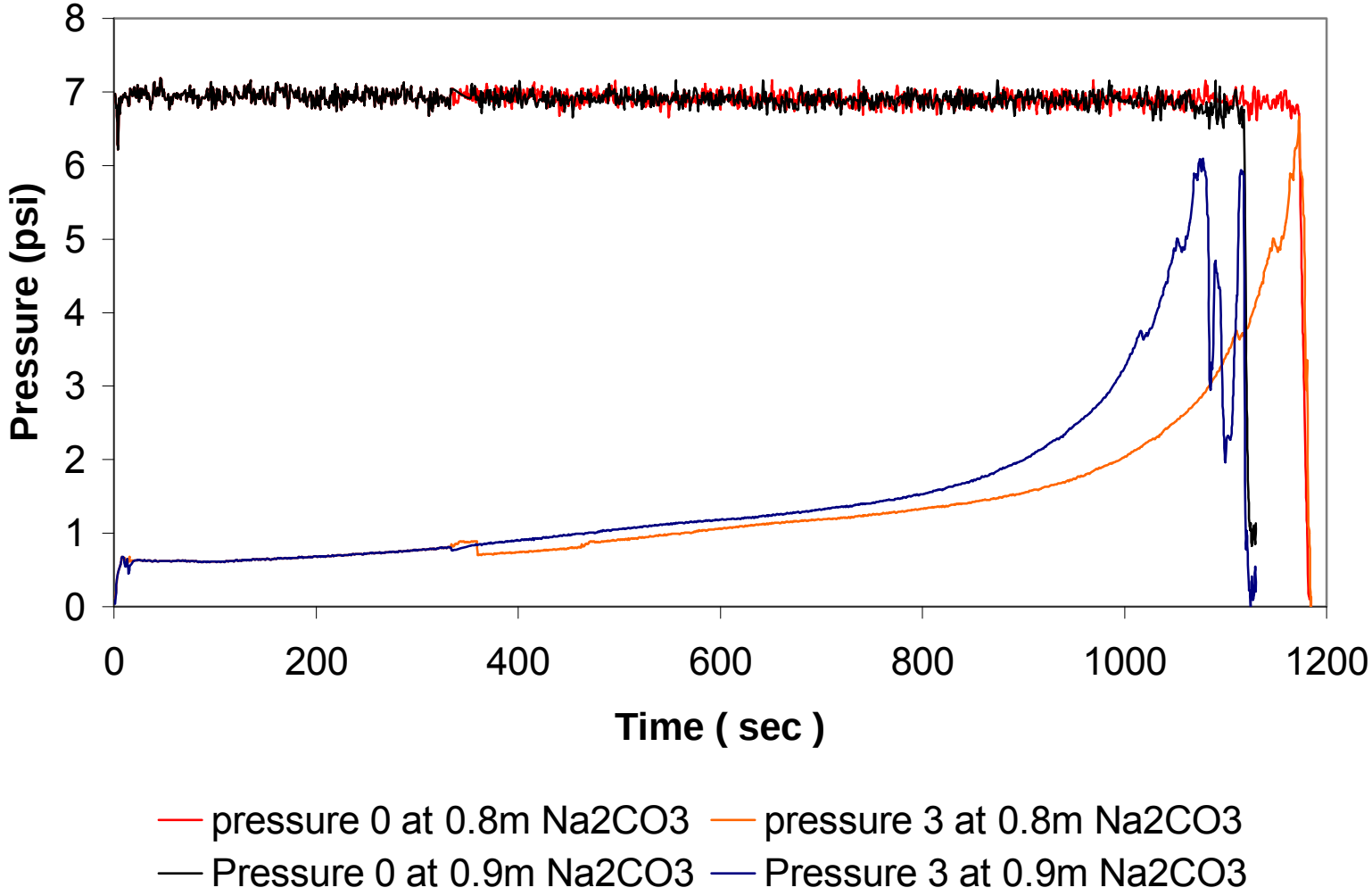
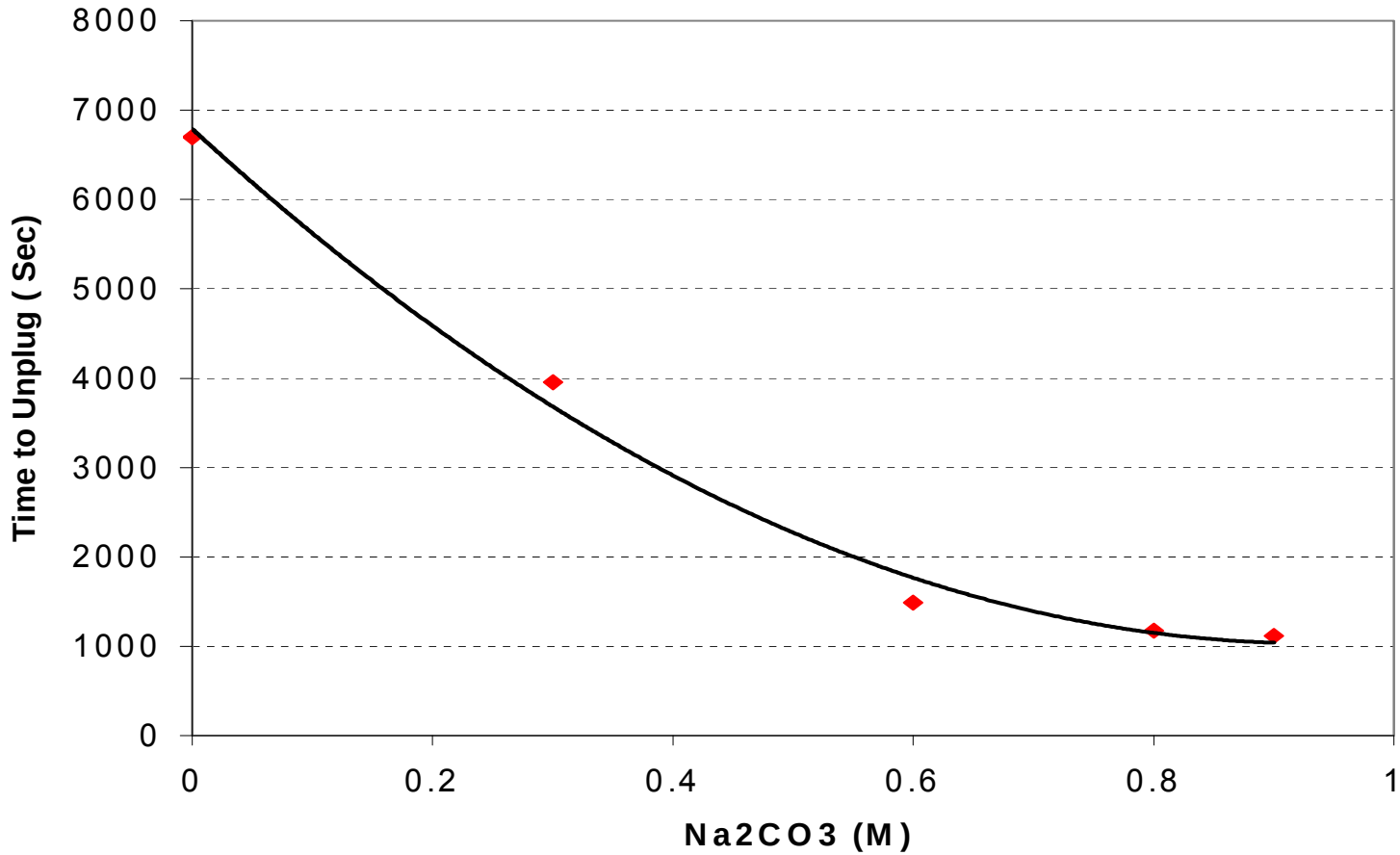
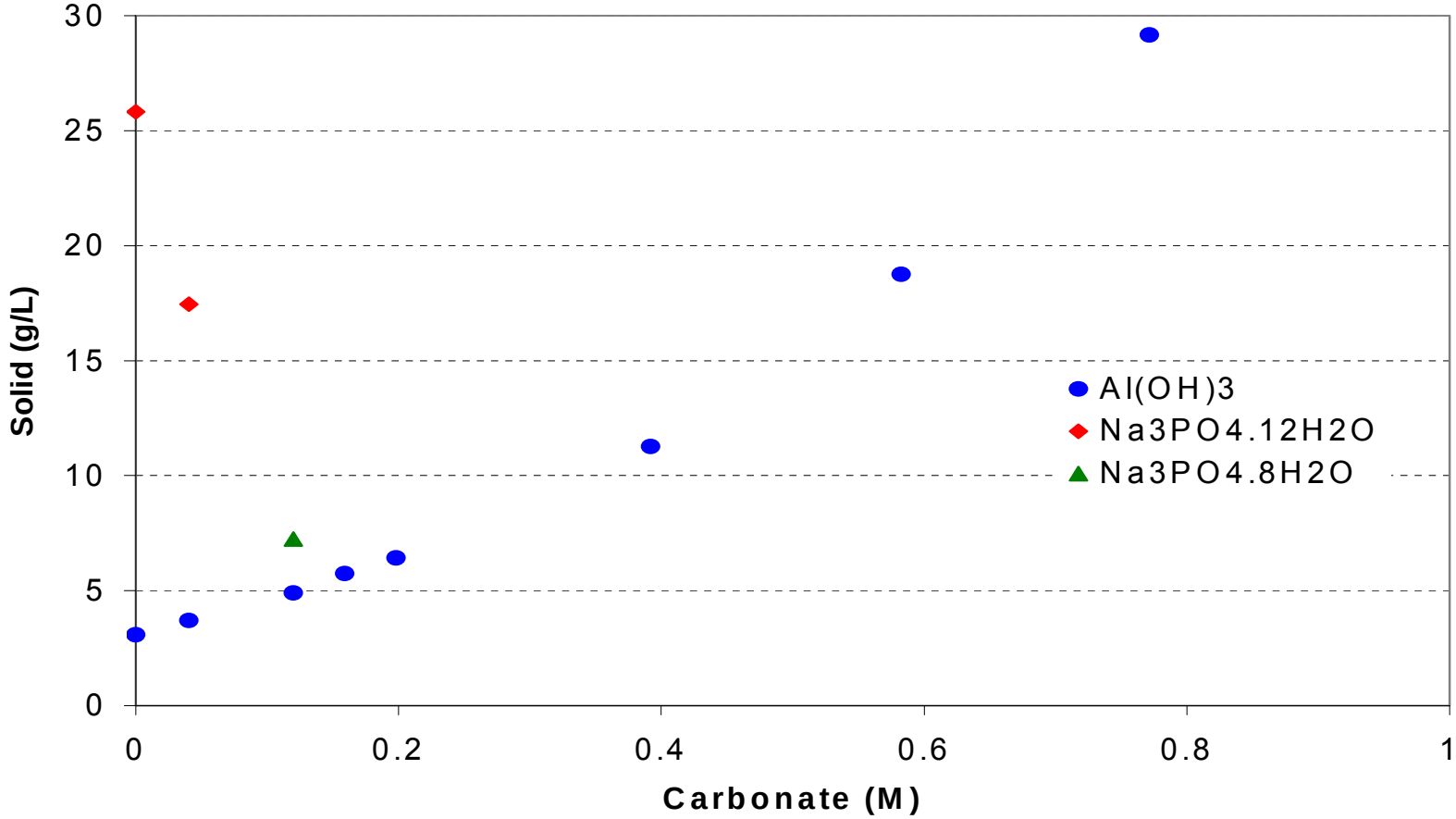
Experimental Observation of Unplugging



Reaction of $Na_3PO_4 \cdot 12H_2O$ with Na_2CO_3 solutions results in less time needed for unplugging than using water alone



Solids predicted by ESP for the reaction of $CO_{2(g)}$ with $Na_3PO_4 \cdot 12H_2O$ in SX-104 surrogate



Observations regarding the use of CO_3^{-2} for destroying $Na_3PO_4 \cdot 12H_2O$ Plugs

- Dissolution of a phosphate plug took approximately 1/7th the time with a 0.9M Na_2CO_3 solution compared to with water alone.
- The addition of carbonate causes reaction with the $Na(OH_2)_4$ sheet resulting in an increase in the $NaCO_3^{-1}$ concentration and the destruction of hydrogen bonding.
- ESP simulations at carbonate concentrations less than ca. 0.1M indicate that the $Na_3PO_4 \cdot 12H_2O$ converts to $Na_3PO_4 \cdot 8H_2O$. Further carbonate addition dissolves all phosphate solids.
- Sprite™ works.

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Imaging and Tomography at Sellafield in the UK

Dominic Rhodes & Tim Tinsley
Nexia Solutions

nexiasolutions

Nuclear expertise intelligently applied

Date: 16/01/08

Non-intrusive monitoring for nuclear applications

What can non-intrusive monitoring deliver?

- Process Monitoring
  - Control
  - Safety
- Process Diagnostics
  - Optimisation
- Better understanding of the process
  - Predictive power
  - Improved modelling

Imaging and Remote Sensing Capability

Which Techniques Should I use?

- What do you want to know?
  - Changes in density, radiation, conductivity, phase, chemical composition, etc…
- How much information do you need?
  - A point measurement, full 3D scanning, or something in between…
- Access to the process?
  - Does it need to be remote, completely intrusive, etc…
- Is the process/unit dynamic?

Imaging and Remote Sensing Capability

Which Techniques are Available

Modality	*Temporal Resolution*	*Spatial Resolution*	*Ease of deployability*
MRI	Excellent	Excellent	Poor
PET	Moderate	Good	Poor
PEPT	Excellent	Excellent	Poor
SPECT	Excellent	Good	Poor
Electrical	Good/Excellent	Moderate	Good
Acoustic	Good	Moderate	Excellent
X ray CT	Good	Good	Moderate
OCST	Excellent	Moderate	Moderate

Imaging and Remote Sensing Capability

Which Techniques are Available

Modality	*Delivered Information*	*Sensor/detector arrangement*
Radiation (transmission)	Attenuation coefficient (density)	Radiation source and detectors
Radiation (emission)	Location of radioactive material	Radiation detectors
Acoustic	Acoustic impedance (reflection, scattering)	Acoustic transducers
Electrical	Conductivity or permittivity distribution	Metallic sensor electrode arrays
MRI	Spectroscopic distribution of material	Induced current in antennae
Optical	Chemical species distribution	Diode Lasers, fibres and photodiodes (transmission)
Radiation (Naturally Occurring)	Location of high atomic number materials	High energy radiation detectors

Case Study 1

Gamma Applications on Vitrification Test Rig (VTR) and others.

Active and Passive applications.

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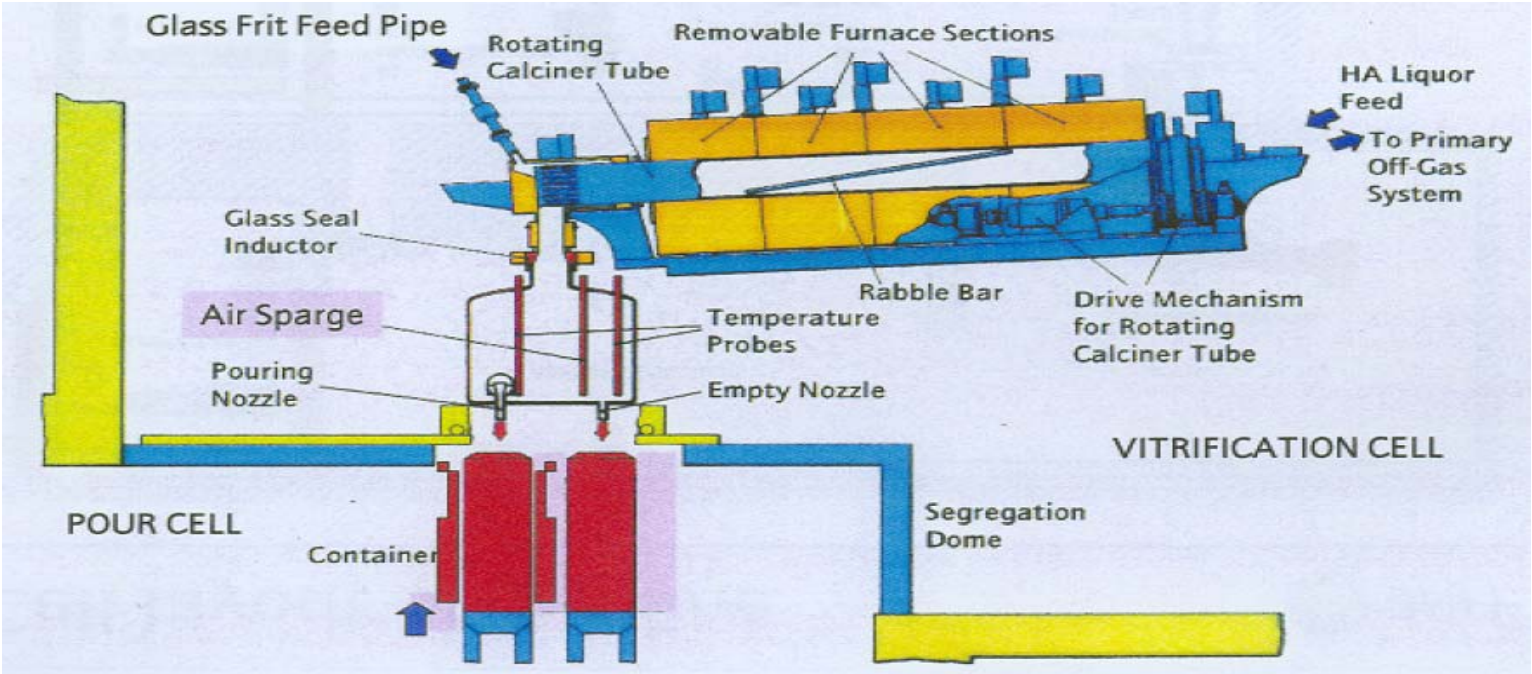
Nuclear expertise intelligently applied

Date: 16/01/08

Imaging and Remote Sensing Capability

Case Study: Vitrification Test Rig

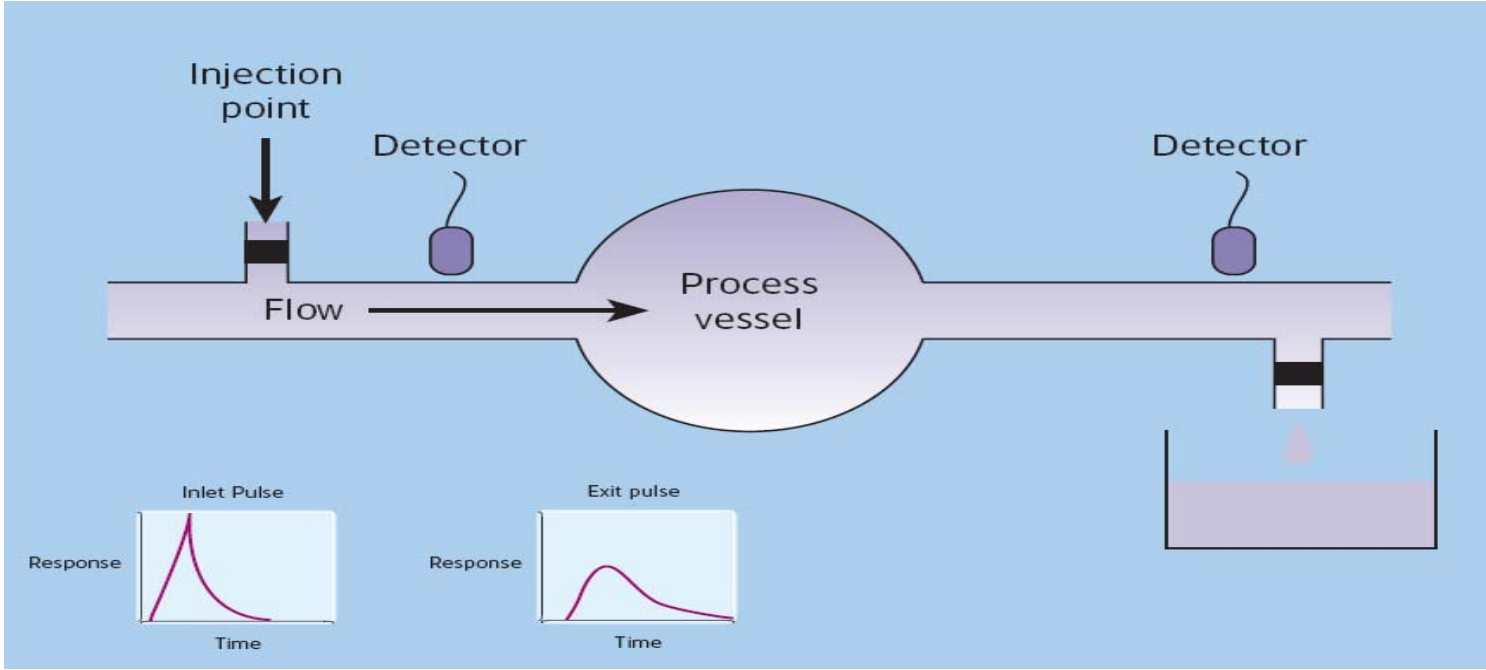
Residence Time of Calciner



Imaging and Remote Sensing Capability

Case Study: Vitrification Test Rig

Residence Time of Calciner



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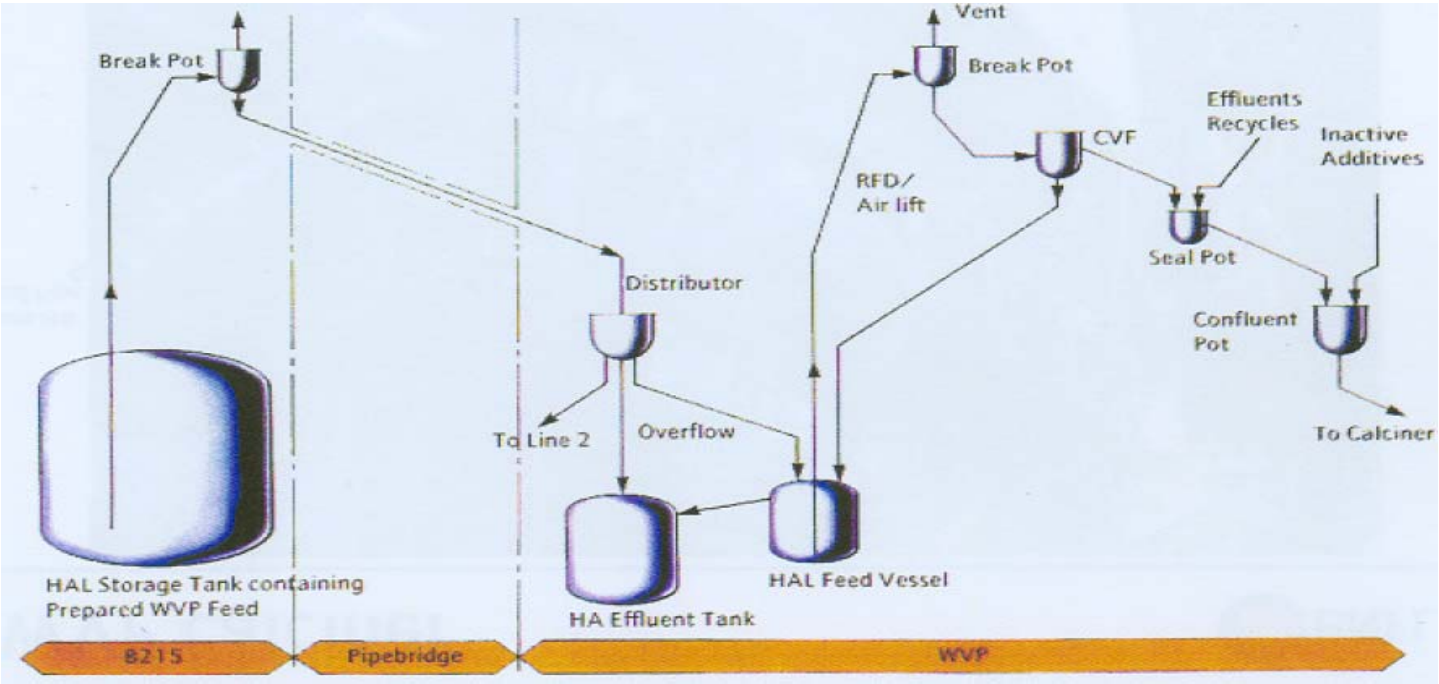
Imaging and Remote Sensing Capability

Case Study: Vitrification Test Rig

Non-invasive Level Detection



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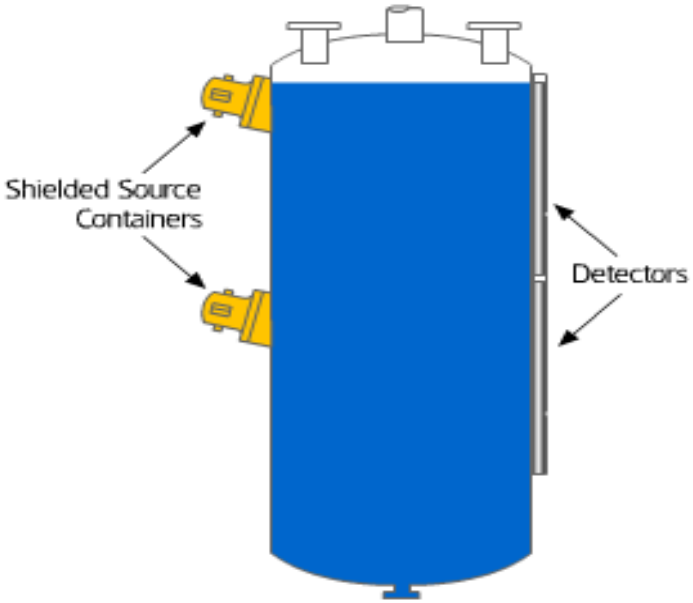
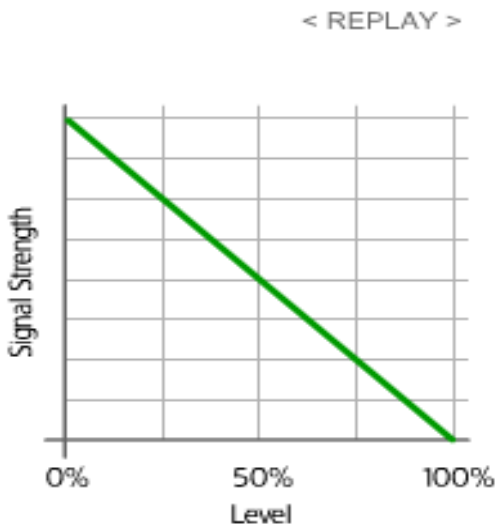
Imaging and Remote Sensing Capability

Case Study: Vitrification Test Rig

Non-invasive Level Detection





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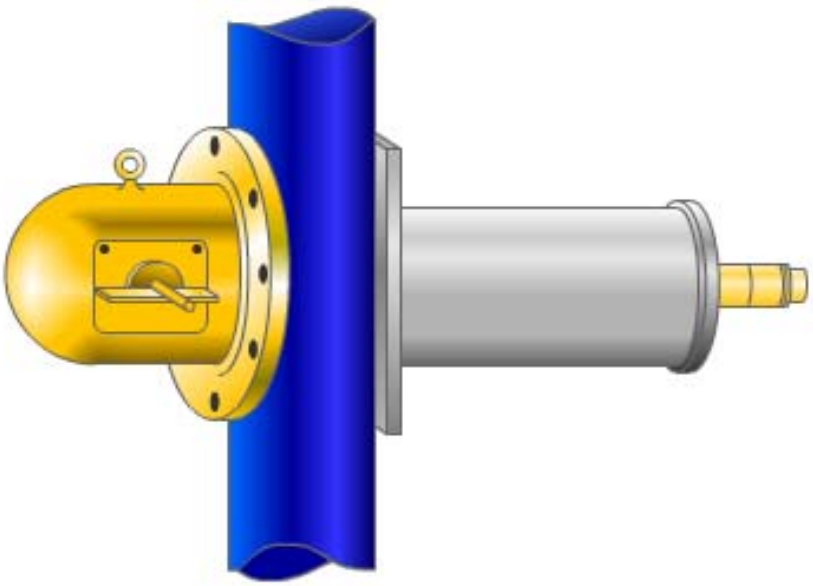
Imaging and Remote Sensing Capability

Case Study: Vitrification Test Rig

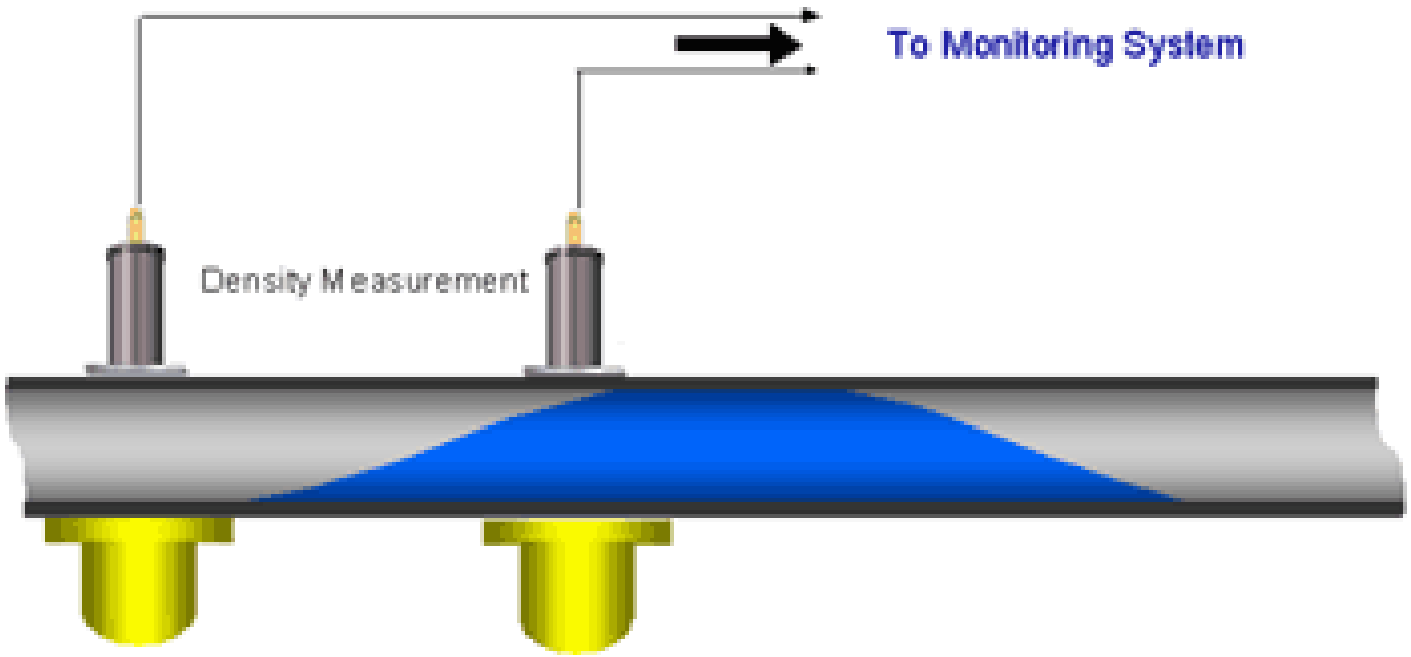
Density metering for the off-gas dust scrubber



Imaging and Remote Sensing Capability

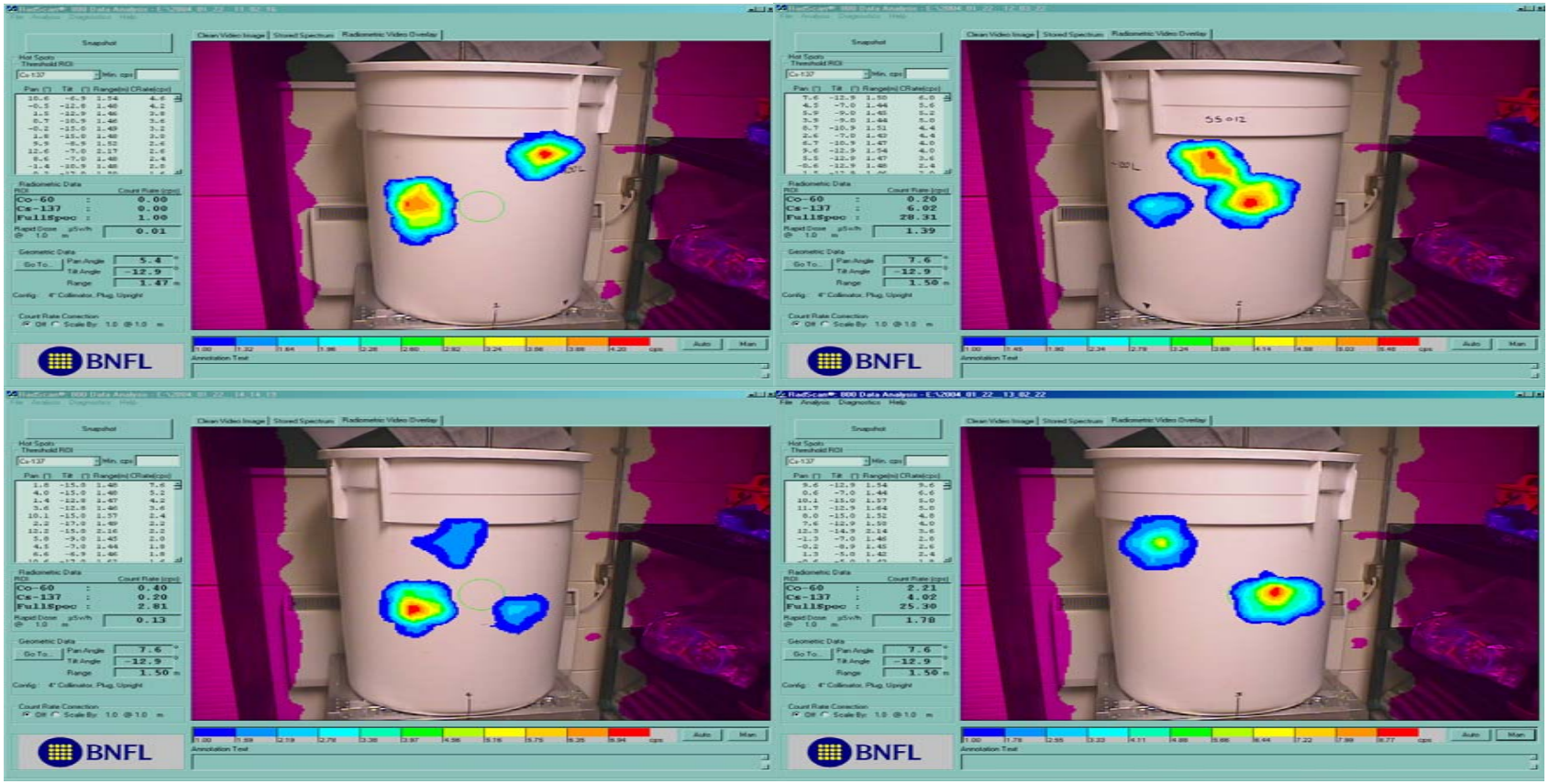
Case Study: Pipeline applications

Slug monitoring



Radiation Tomography Test

Radiation Survey



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Tomographic Reconstruction

Case Study 2

Acoustic and Electrical detection of gas core inside a nuclear reprocessing precipitation reactor for heavy metals.

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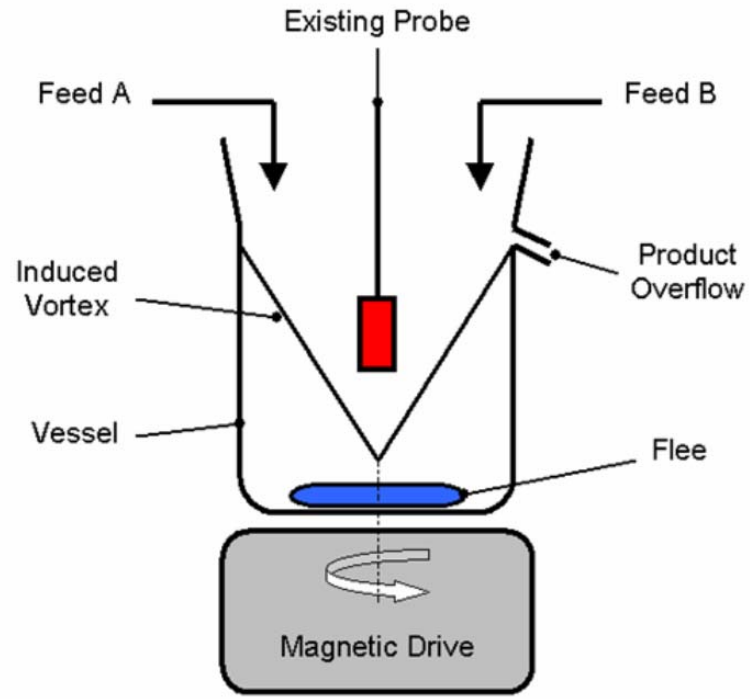
Acoustic Process Monitoring

What do we want to know

- Size of vortex indicates residence time for the reactor
- Can be used as a control point
- Monitoring of start up and shut down
- Approach to steady state



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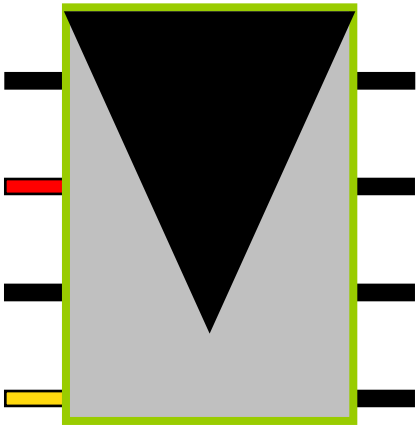
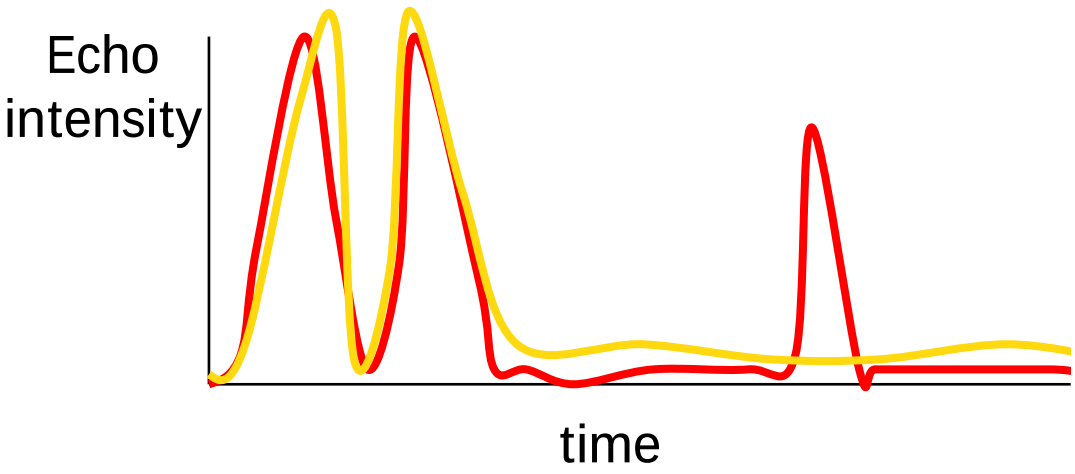
Acoustic Process Monitoring

Development of an acoustic monitoring system





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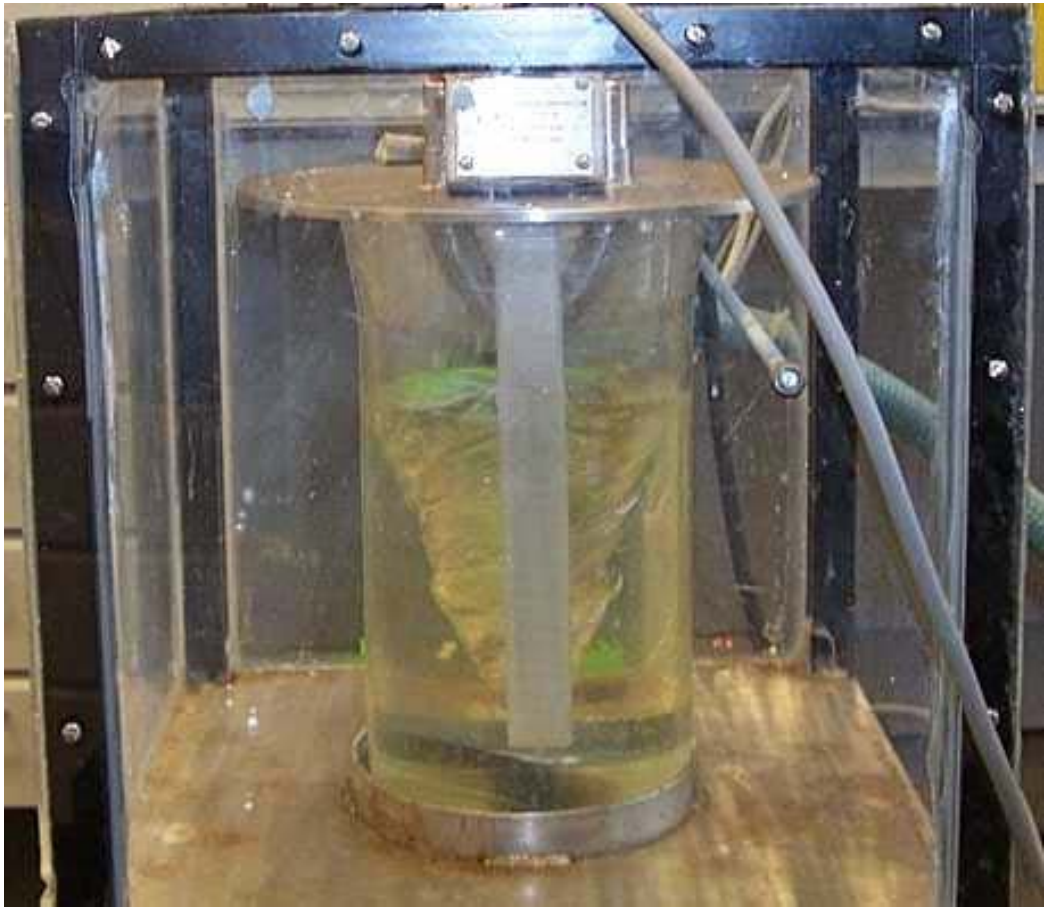
ERT Tomography: The Solution



Vortex Video

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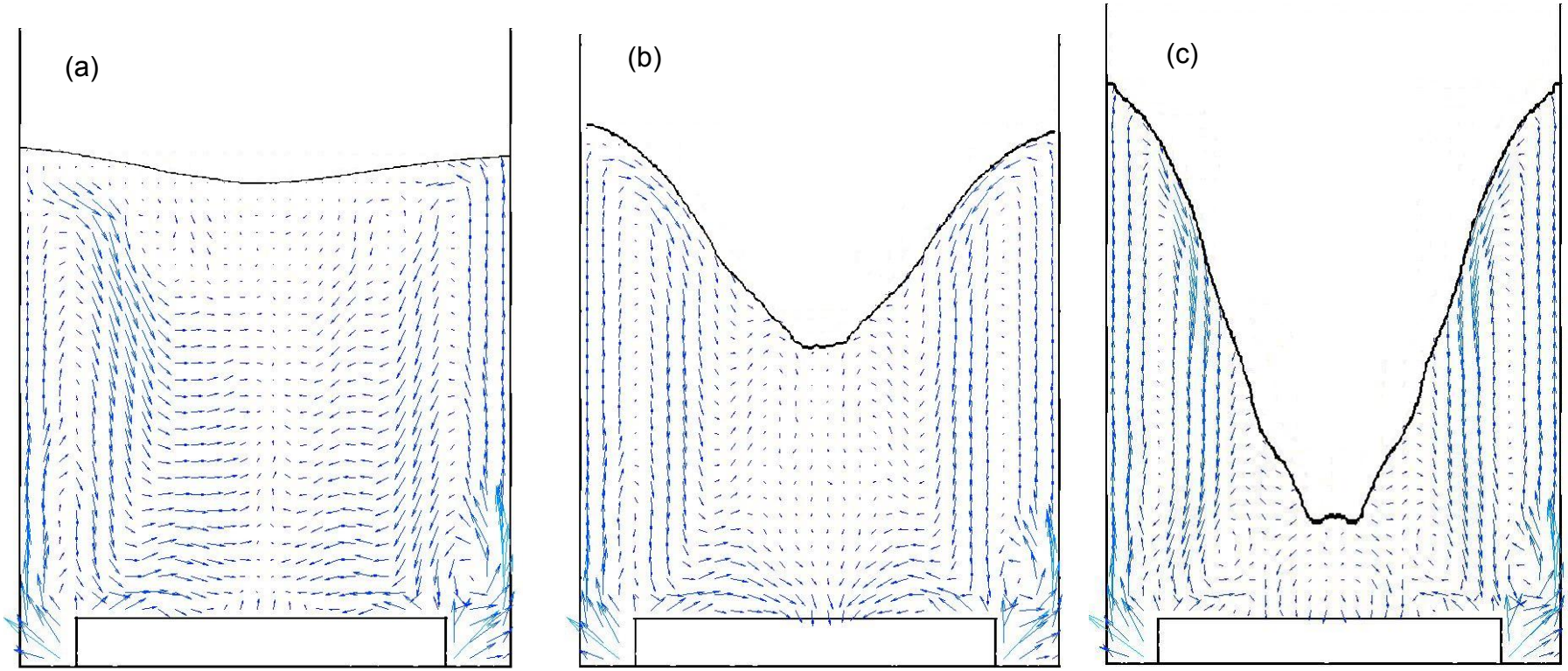
Predicted liquid surface profiles / flow fields



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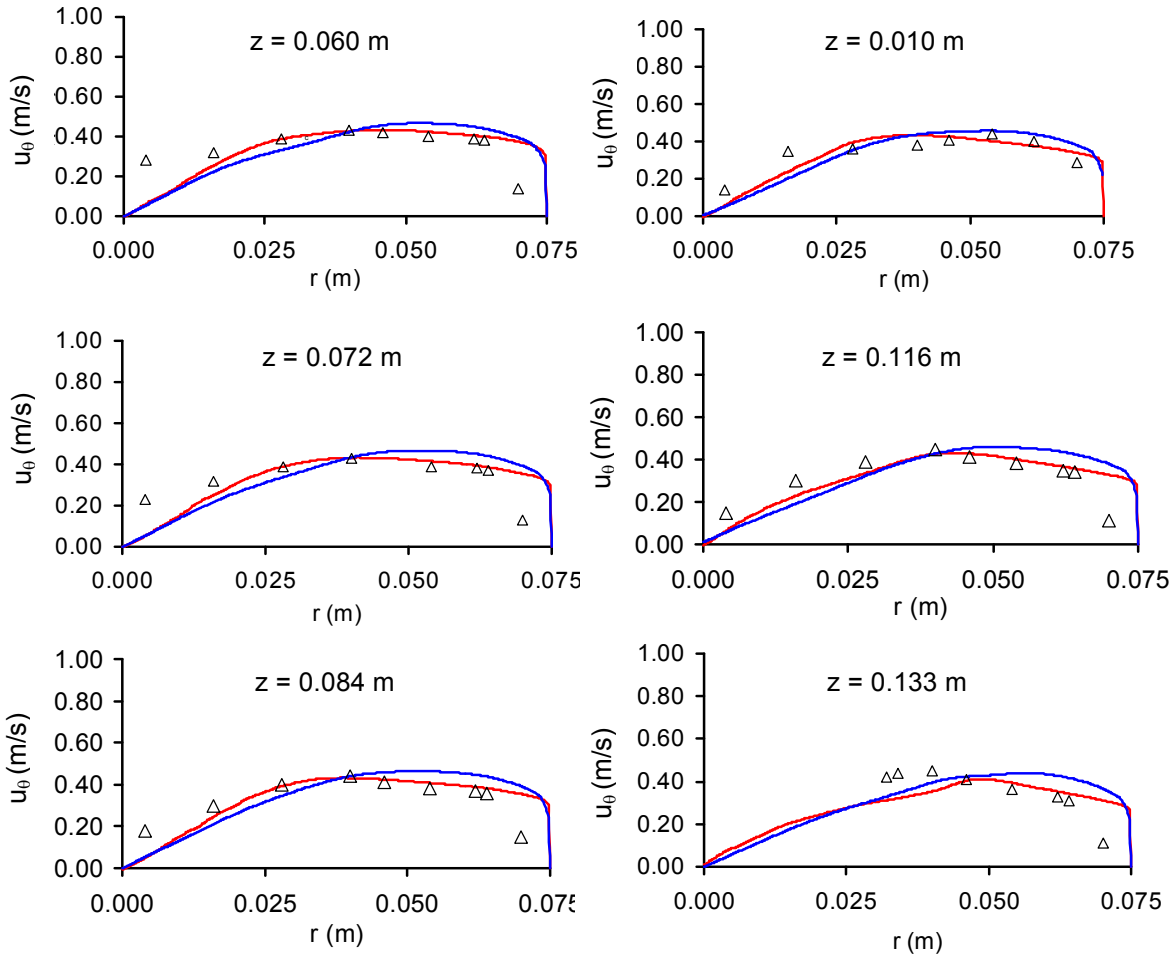
Comparison: Predicted vs Measured



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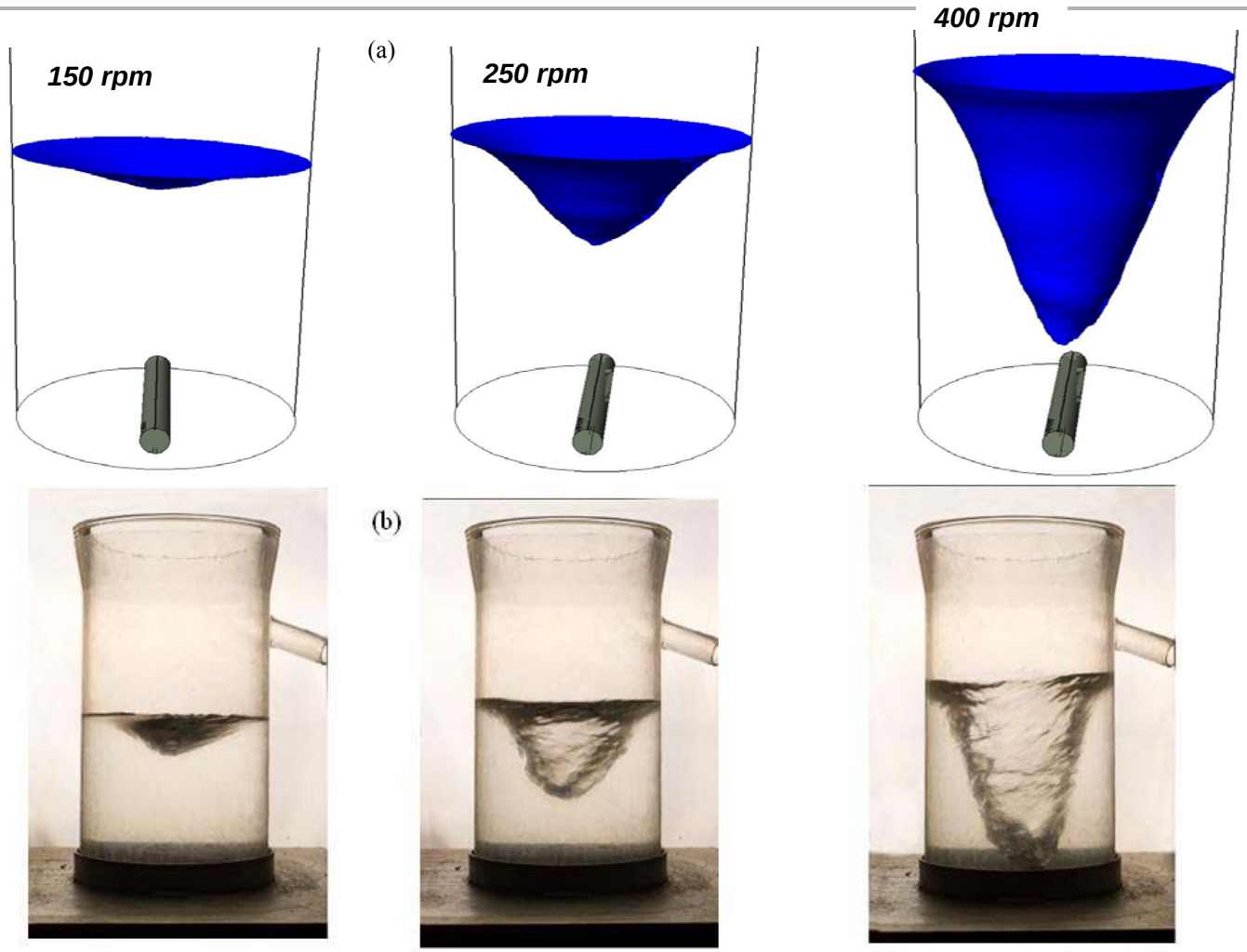
Validation



Case Study 3

Electrical imaging of solid liquid suspensions

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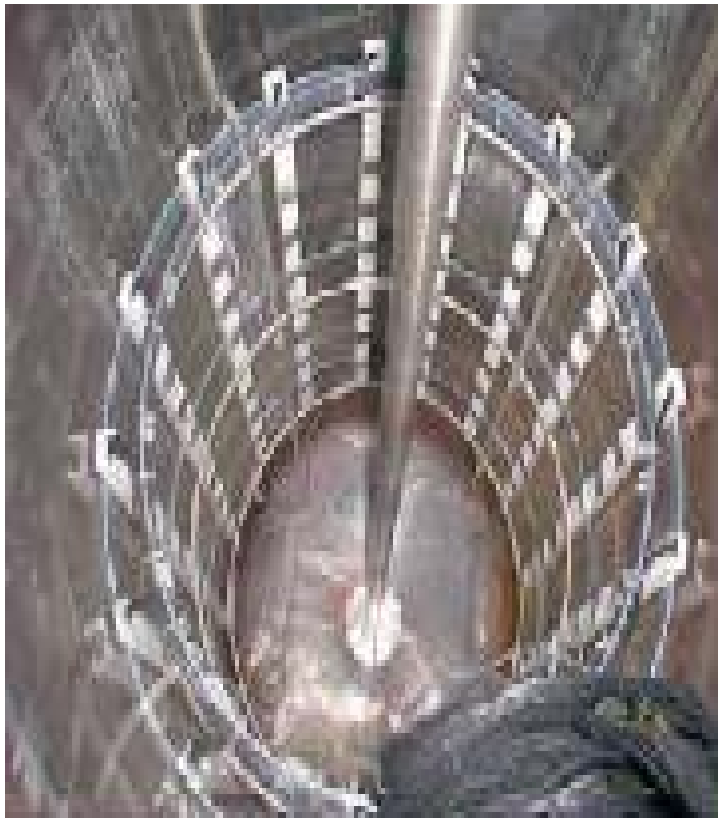
Date: 16/01/08

Electrical Imaging of Solid Suspensions

Ring Arrays



Linear Arrays

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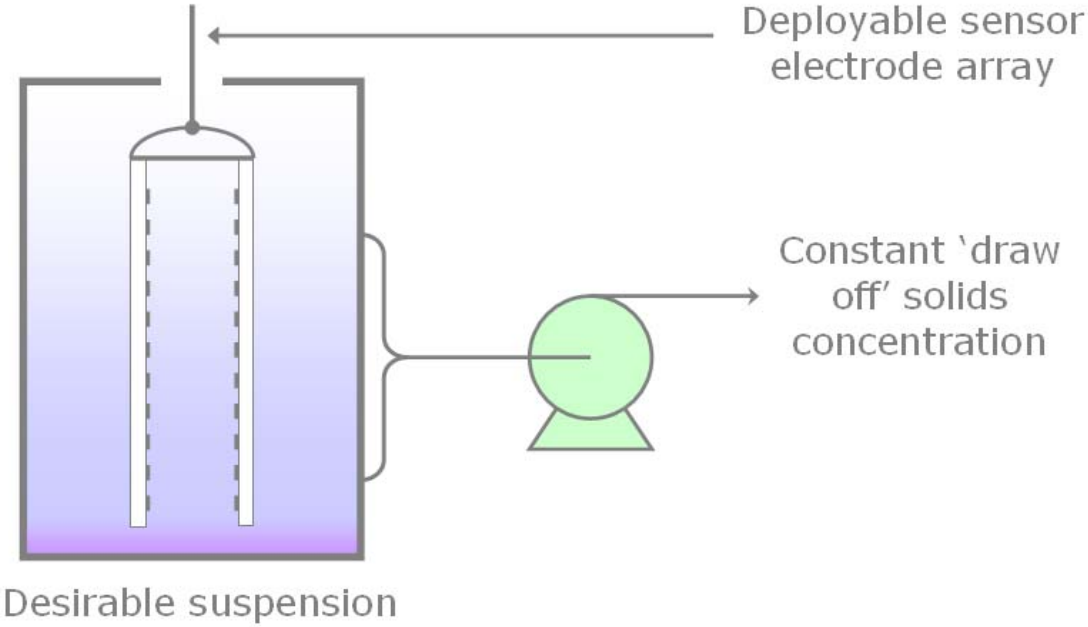
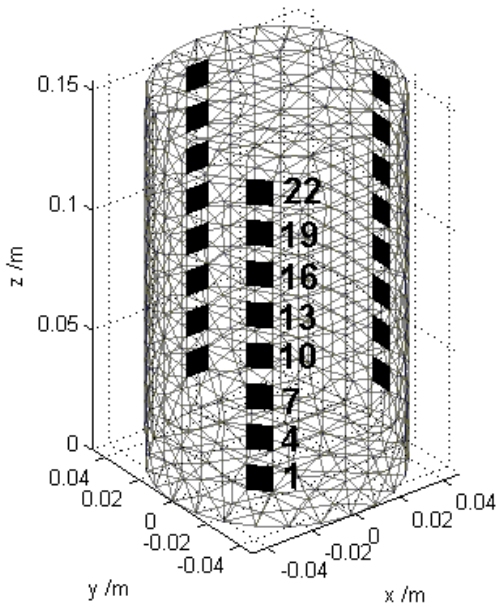
Electrical Imaging of Solid Suspensions

Linear Arrays





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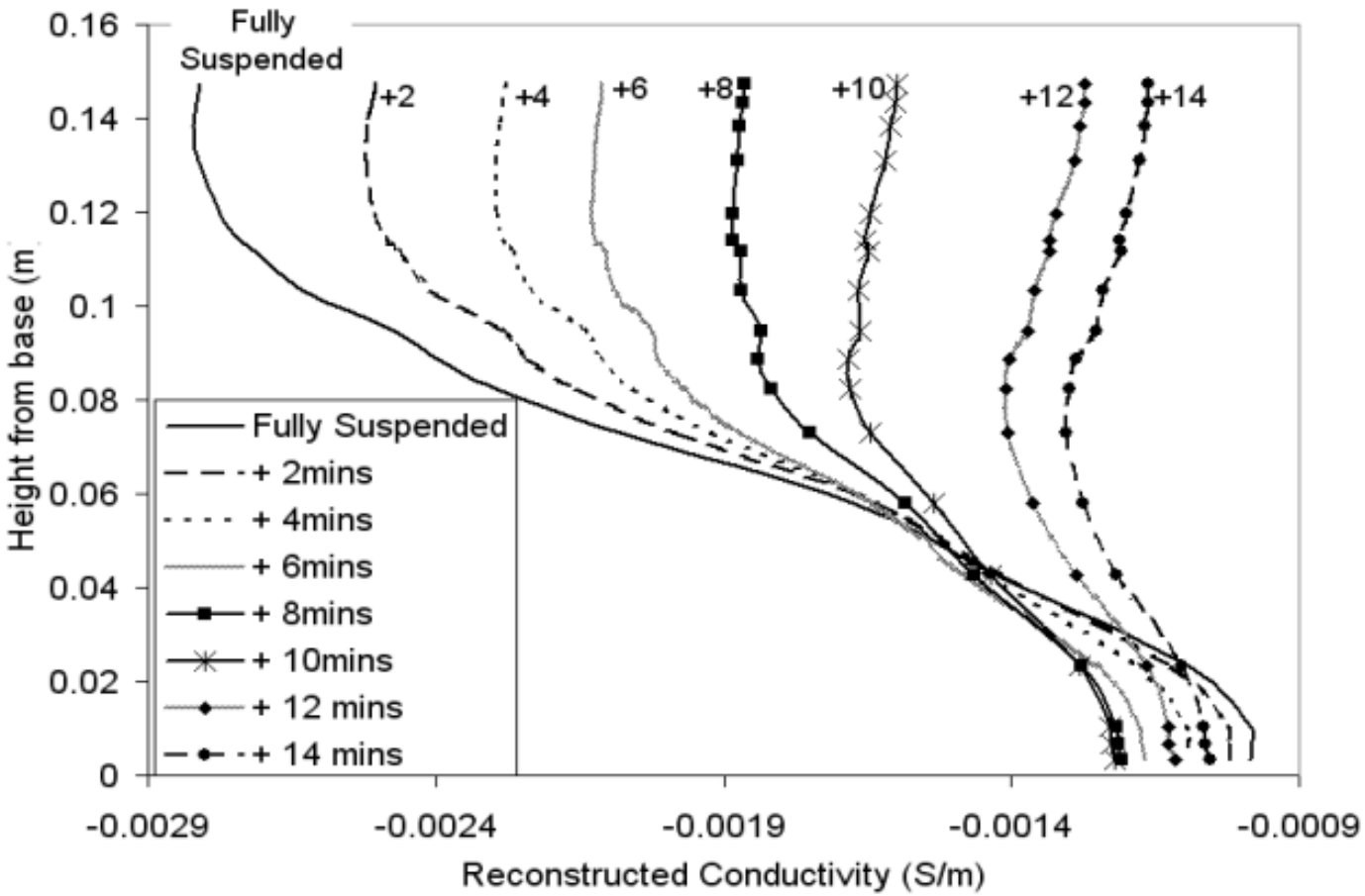
Electrical Imaging of Solid Suspensions



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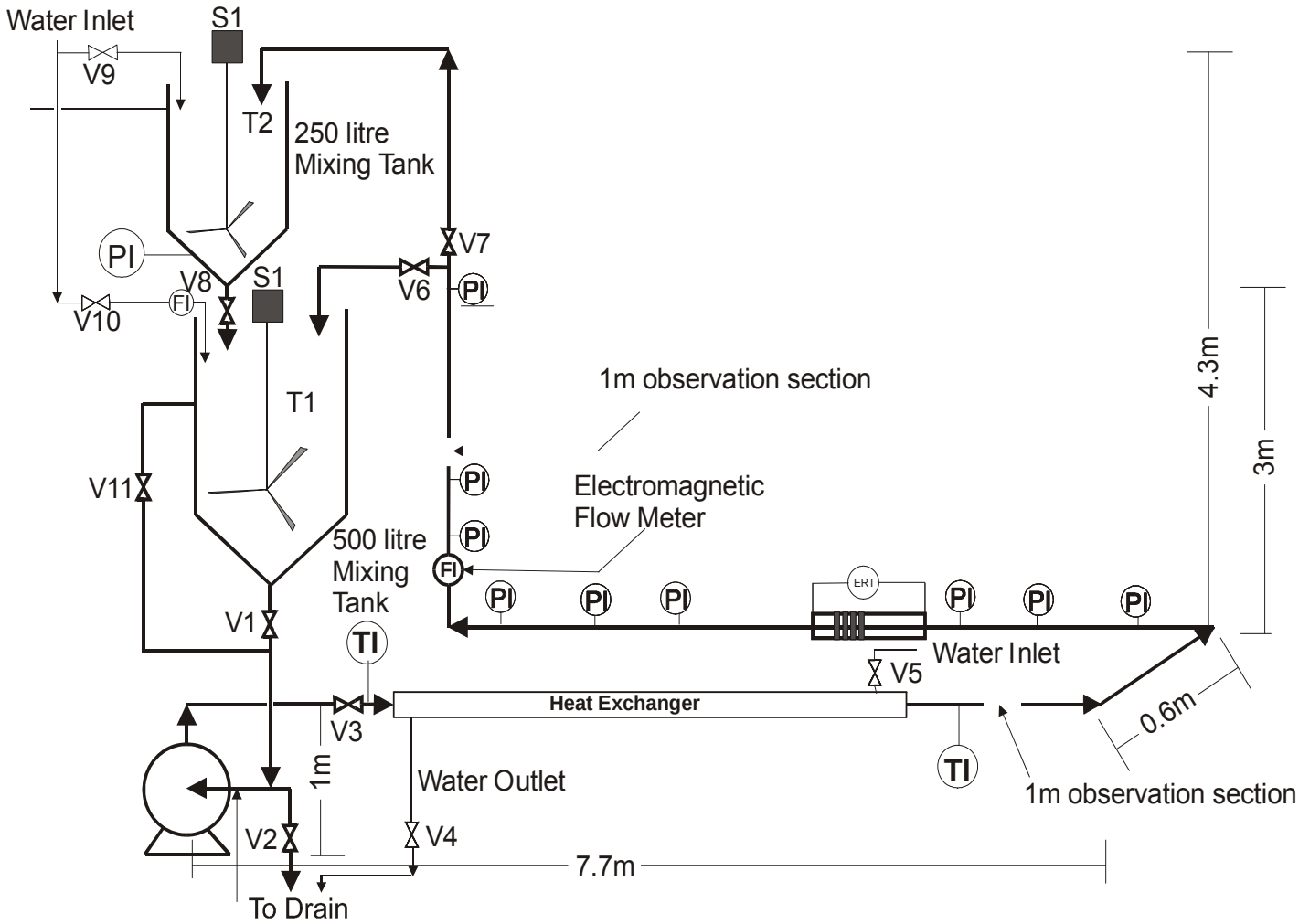
Typical Flow loop



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Case Study 4

Non Electrical Imaging

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Date: 16/01/08

What is RadBall?

RadBall is a deployable radiation mapping device which can **locate**, **quantify** and **characterise** radiation hazards from a single position.

Polymer plastic

+

Collimation sheath

=

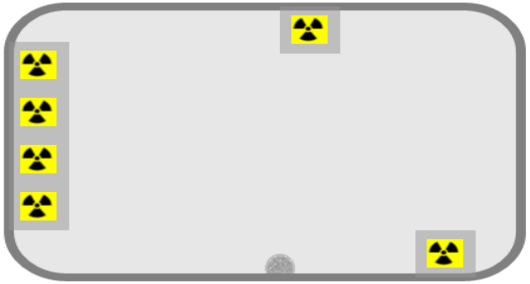
DEPLOYABLE RADIATION MAPPING DEVICE

RADBALL

What can it be used for?

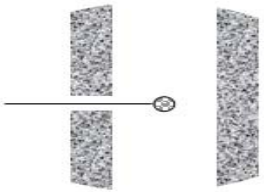
Nexia Solution will offer a radiation mapping service based on the RadBall device

- Radiation mapping in:
  - Active cells
  - Glovebox
  - Confined spaces
  - Hard to reach parts of the plant
  - Plants with high radiation levels



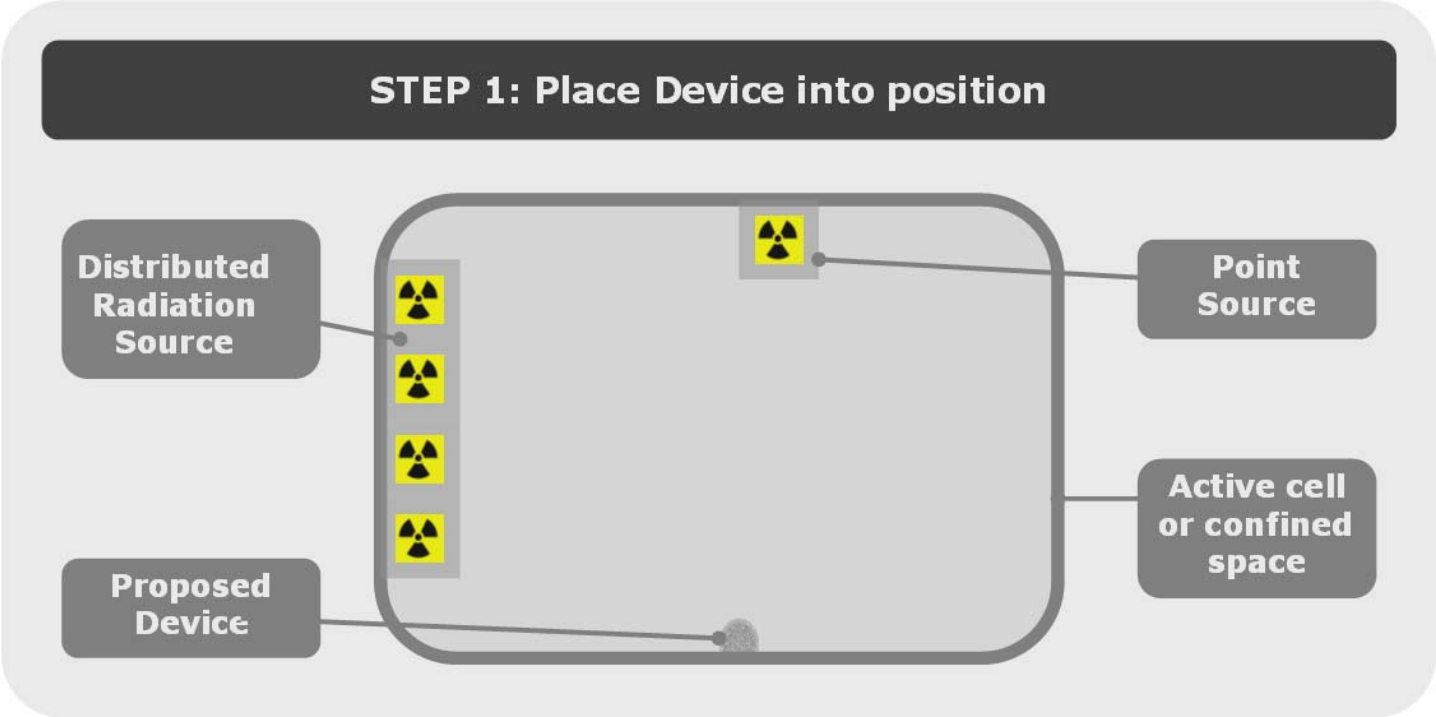
Cell/glovebox

- Quality control for decontamination efforts
  - Before and after surveys
  - Detection of untreated areas

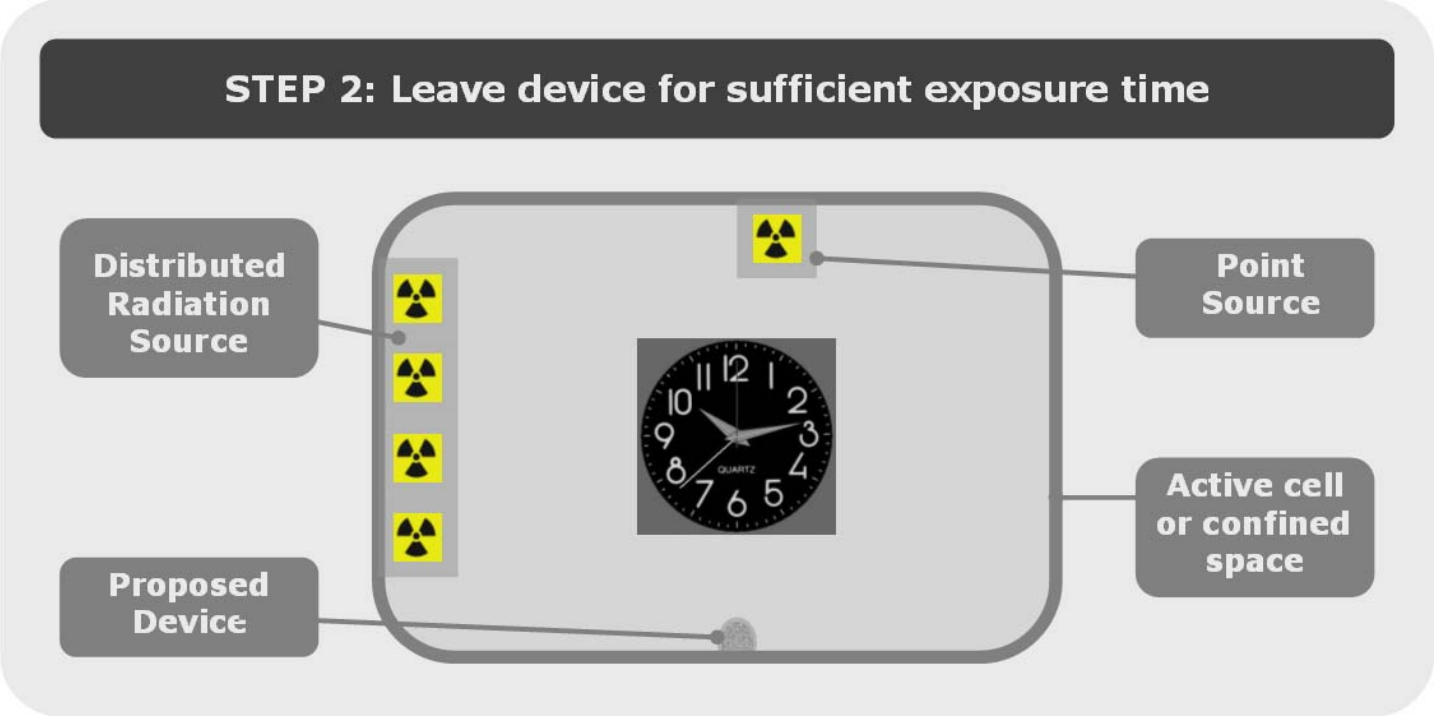


Through a drilled hole

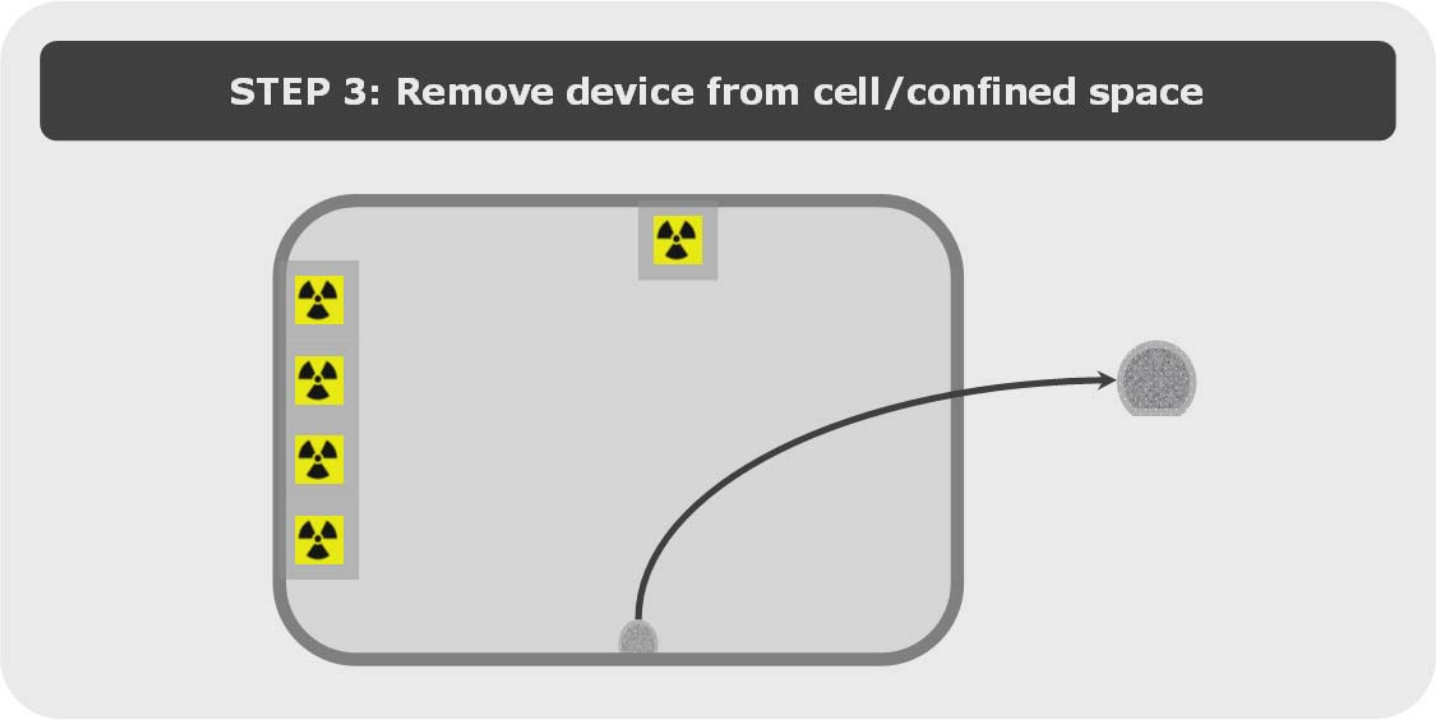
How does it work?



How does it work?

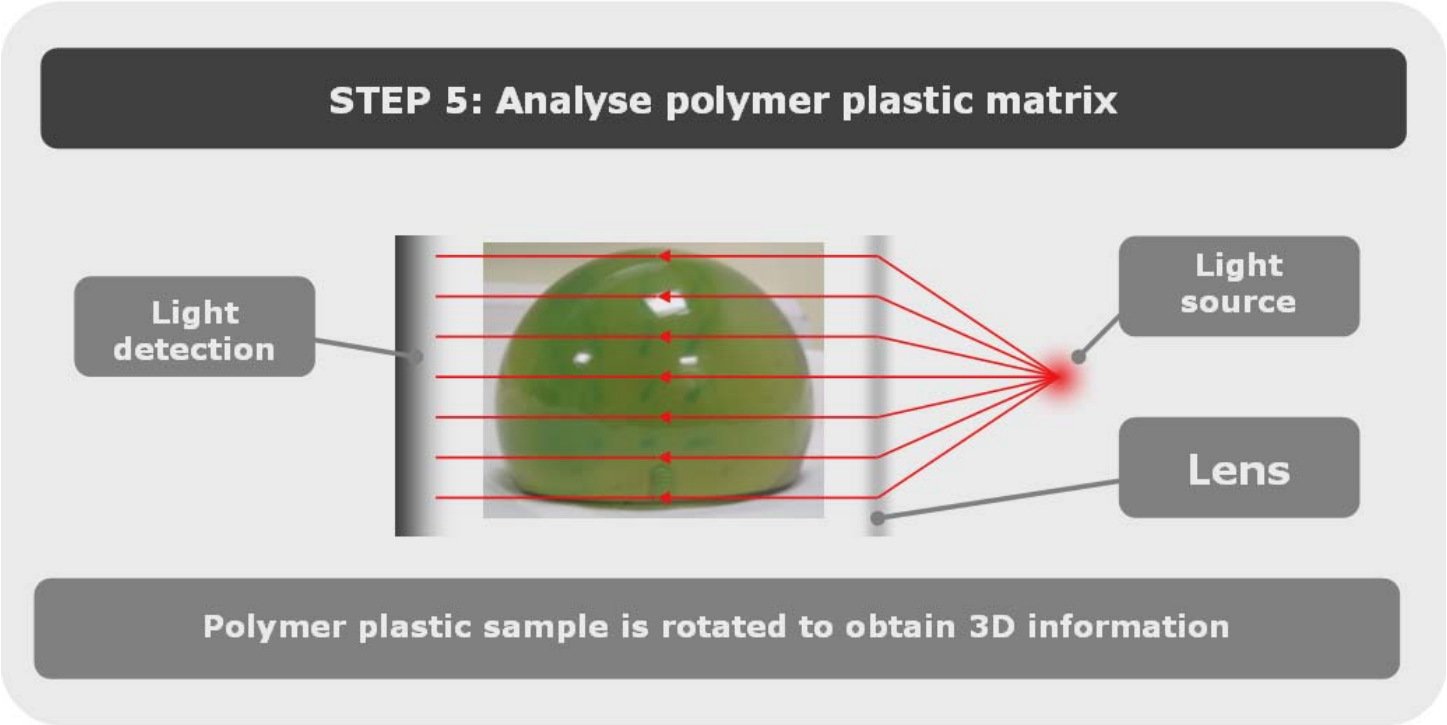


How does it work?



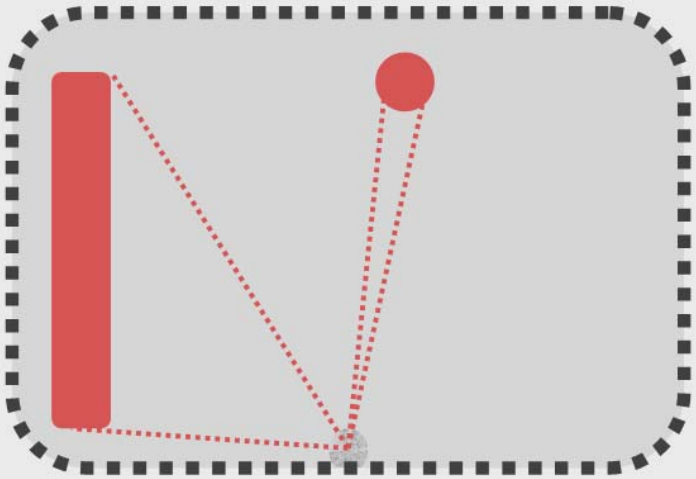
How does it work?

How does it work?



How does it work?

STEP 6: Radiation hazards located and characterised

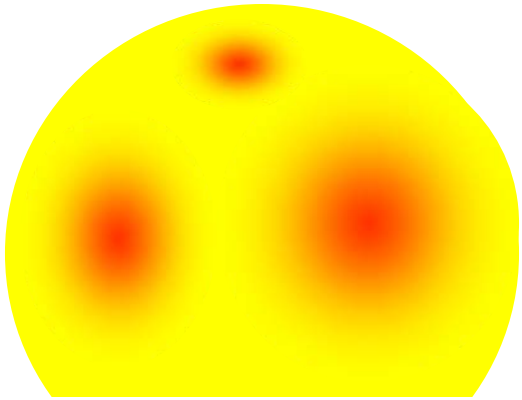


Radiation is located, quantified and characterised

What is the output (basic)?

BASIC OUTPUT:

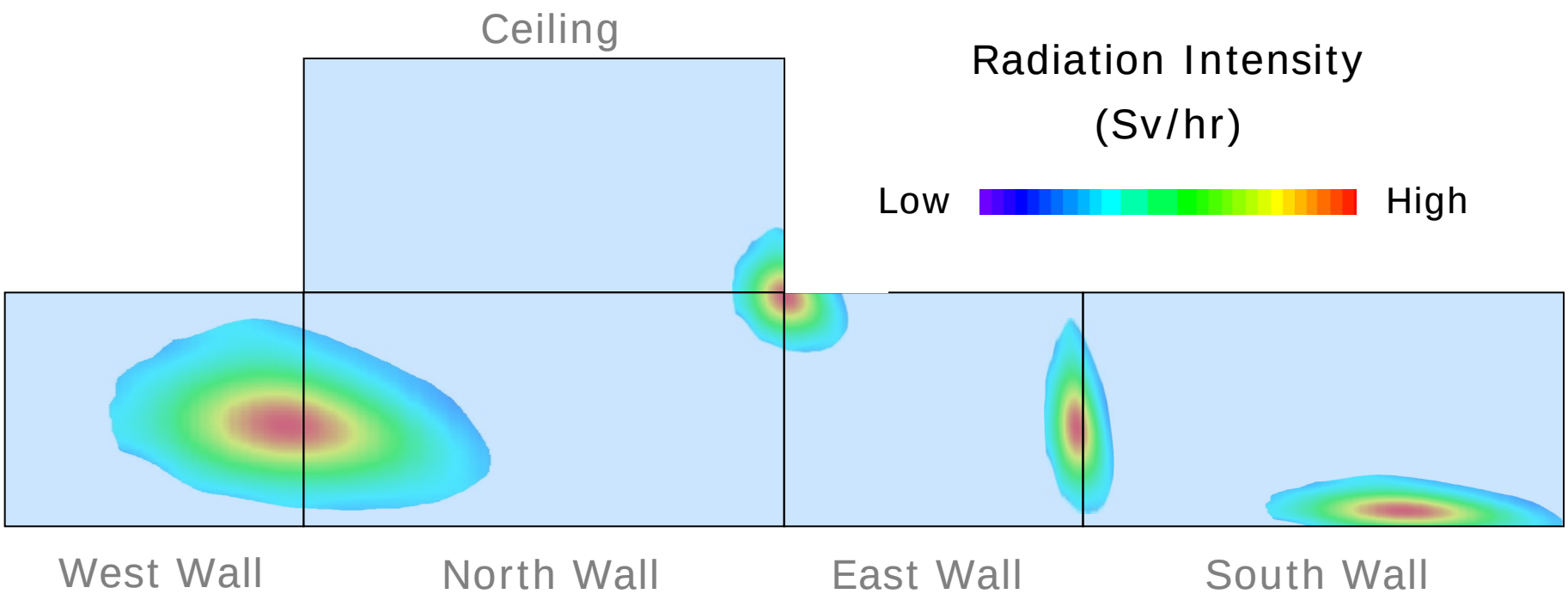
Radiation deposited on the device
From a particular direction
+
An estimate of the incident
radiation energy



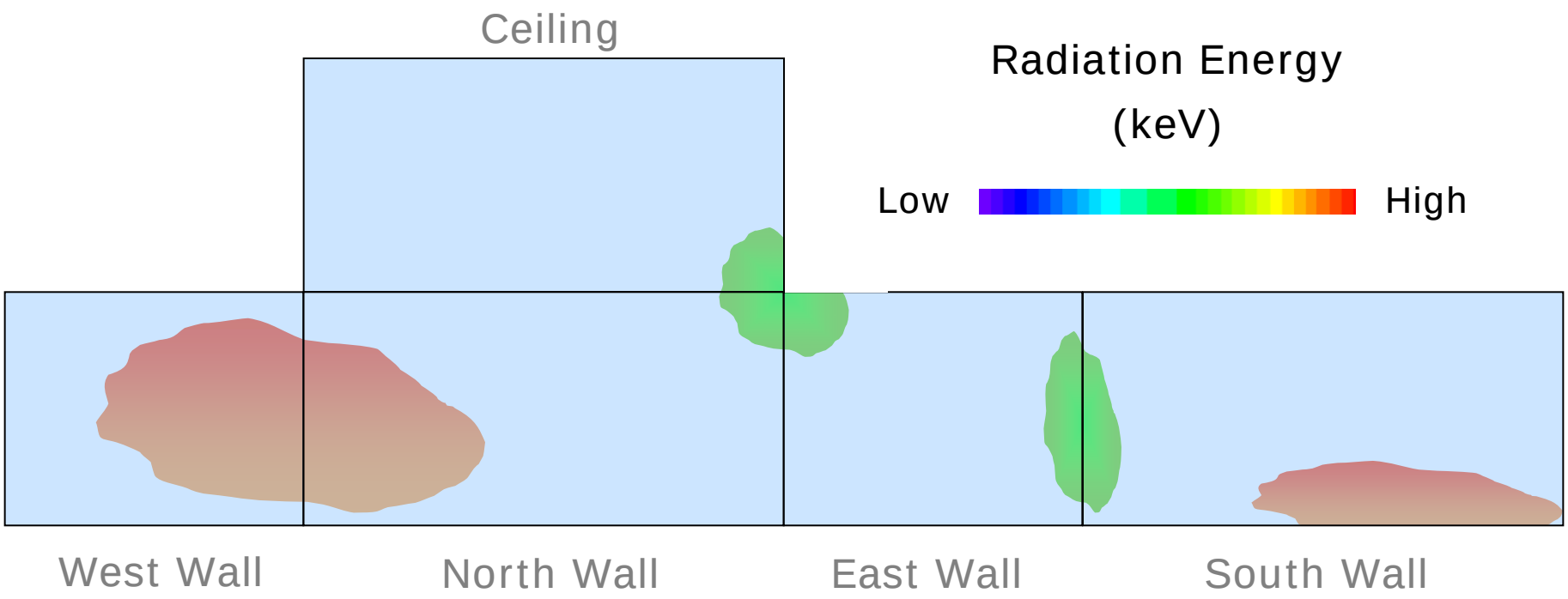
Radiation Intensity (Sv/hr)

Low High

What is the output (enhanced)?



What is the output (enhanced)?



Others Imaging Techniques

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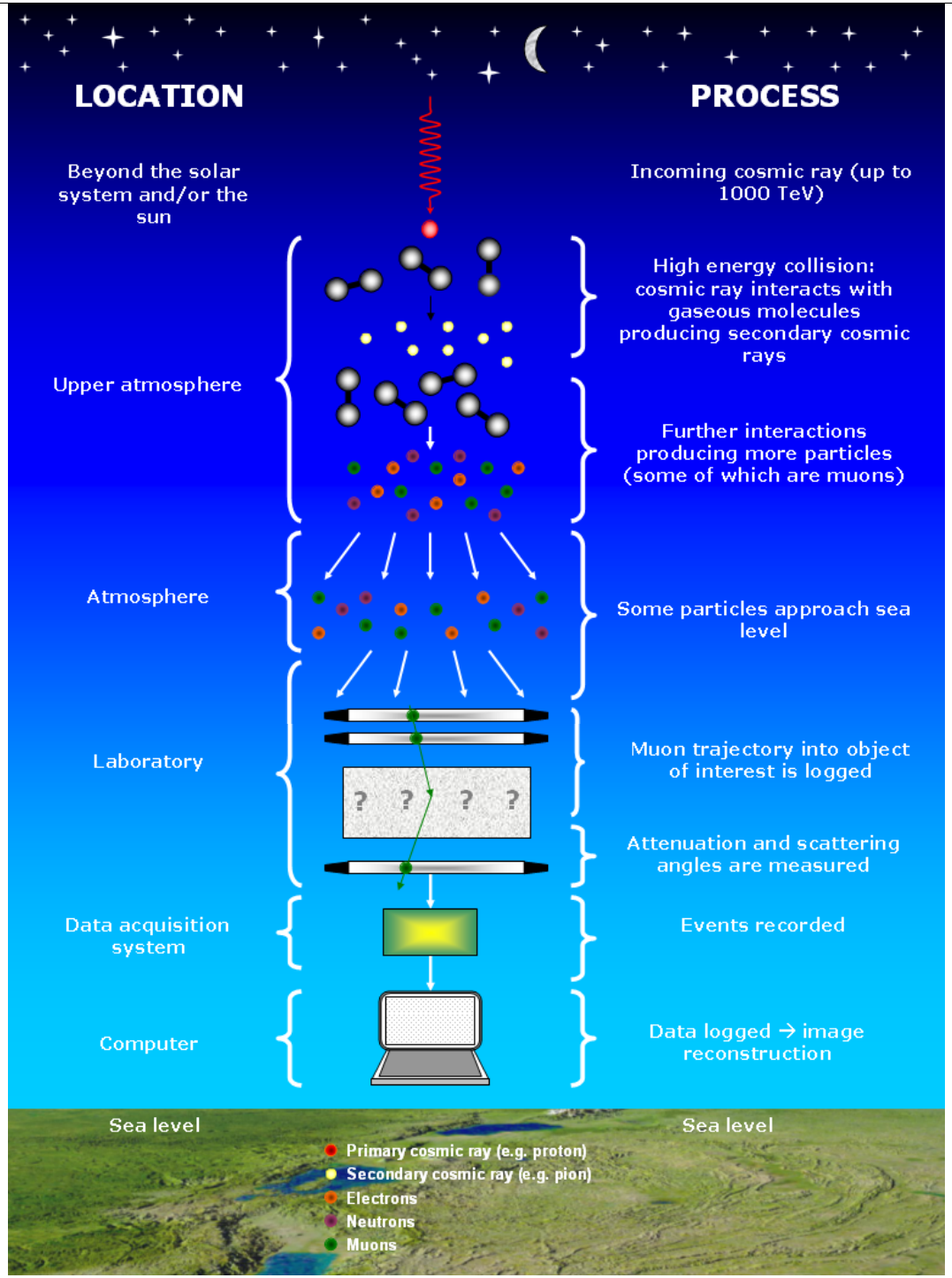
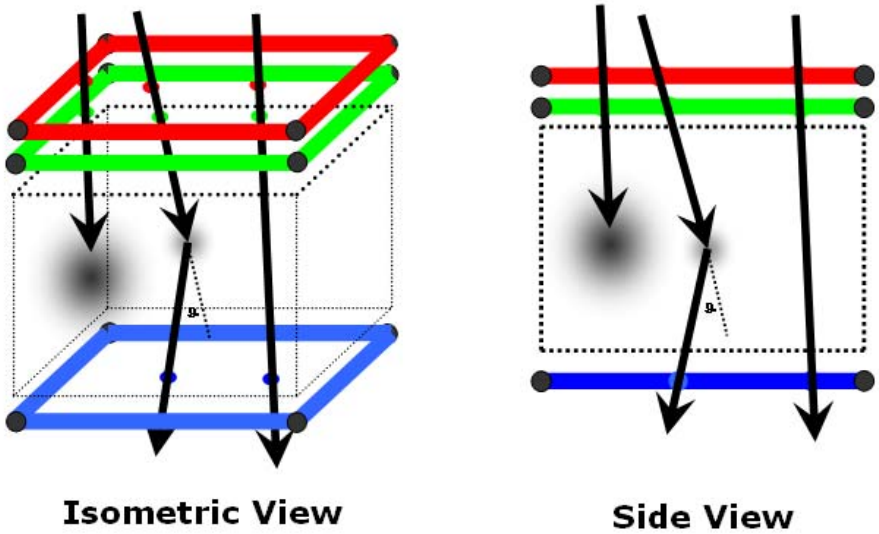

LOCATION
PROCESS
Beyond the solar system and/or the sun
Incoming cosmic ray (up to 1000 TeV)
Upper atmosphere
High energy collision: cosmic ray interacts with gaseous molecules producing secondary cosmic rays
Further interactions producing more particles (some of which are muons)
Atmosphere
Some particles approach sea level
Laboratory
Muon trajectory into object of interest is logged
Attenuation and scattering angles are measured
Data acquisition system
Events recorded
Computer
Data logged → image reconstruction
Sea level
Sea level
Primary cosmic ray (e.g. proton)
Secondary cosmic ray (e.g. pion)
Electrons
Neutrons
Muons

Imaging in the nuclear industry

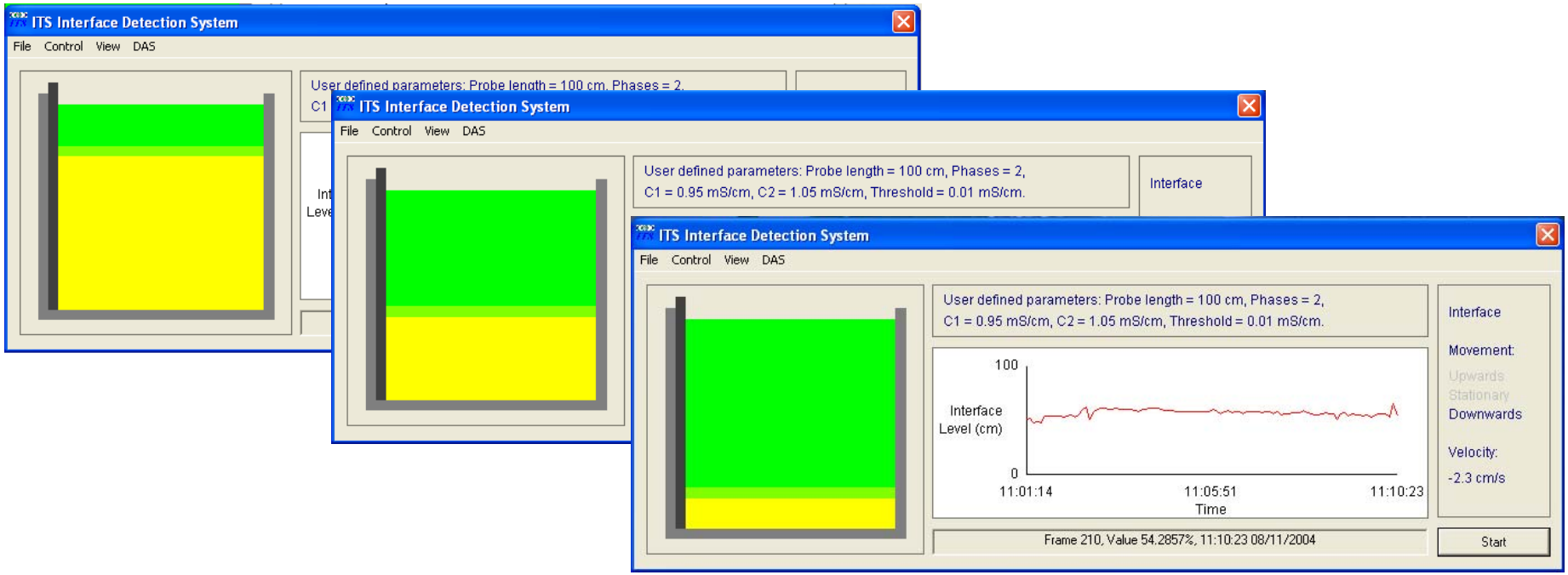
Prototype Muon Camera



The detector panels are placed around the domain of interest, preferably above and below. The muon trajectories are tracked into the domain of interest where they are either stopped, scattered or unaffected. This provides a means to locate, quantify and characterise the heavy metal (i.e. Plutonium and Uranium) contents of the vessel or silo through spatial muon flux and scattering angle measurements.

Imaging and Remote Sensing Capability

Case Study: Process Measurement, level detection during sludge transfer



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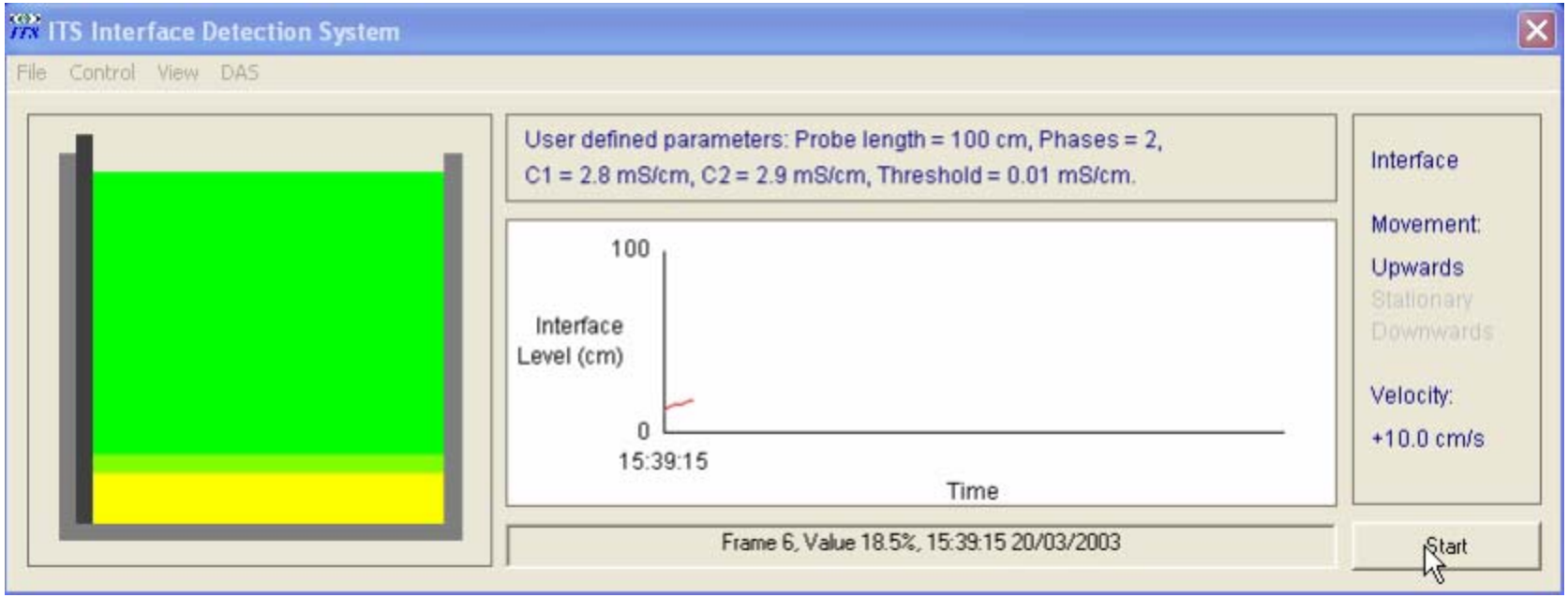
Interface Detection Video



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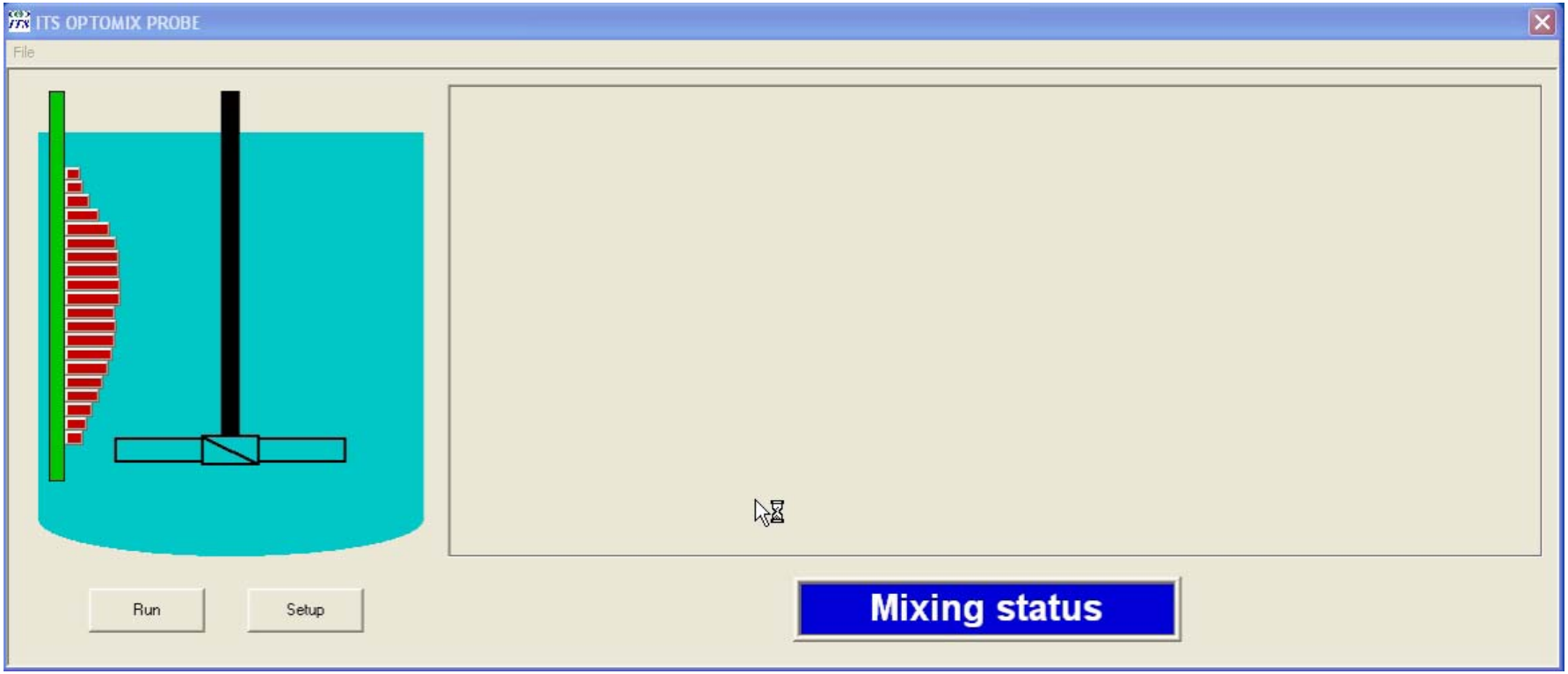
Mixing Video



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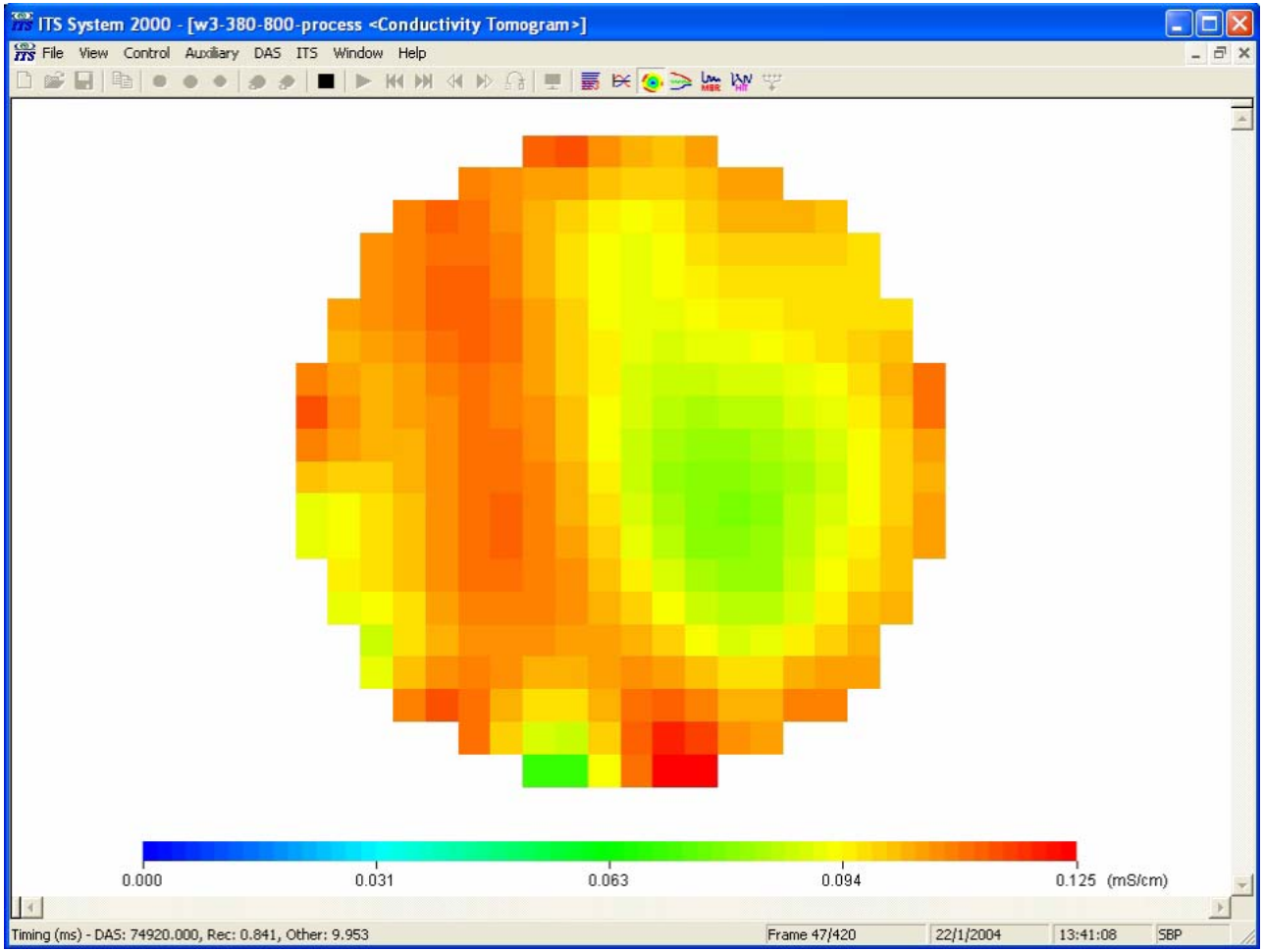
Filtration Video



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Drift Flux CFD

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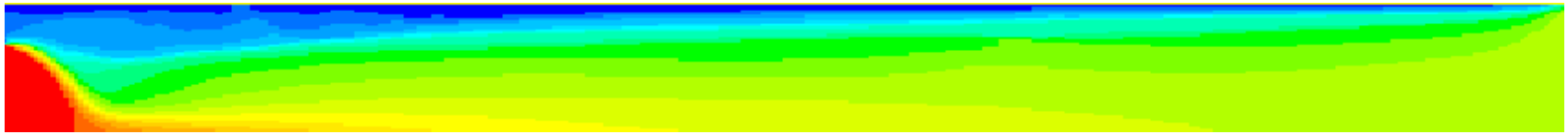
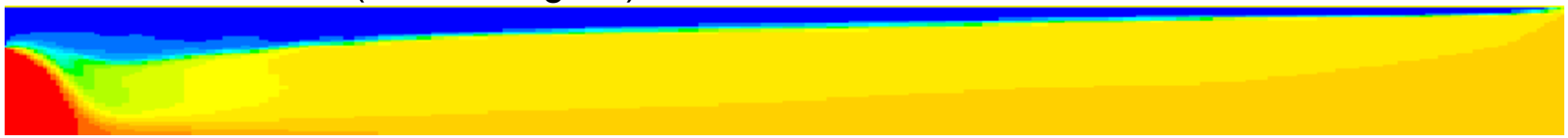
Date: 16/01/08

Glass Particles in Model Settling Tank



Total concentration (red ~ 19 kg/m^3)



Concentration in 20-30 μm range.

Concentration in 60-70 μm range.

Concentration in 90-100 μm range.

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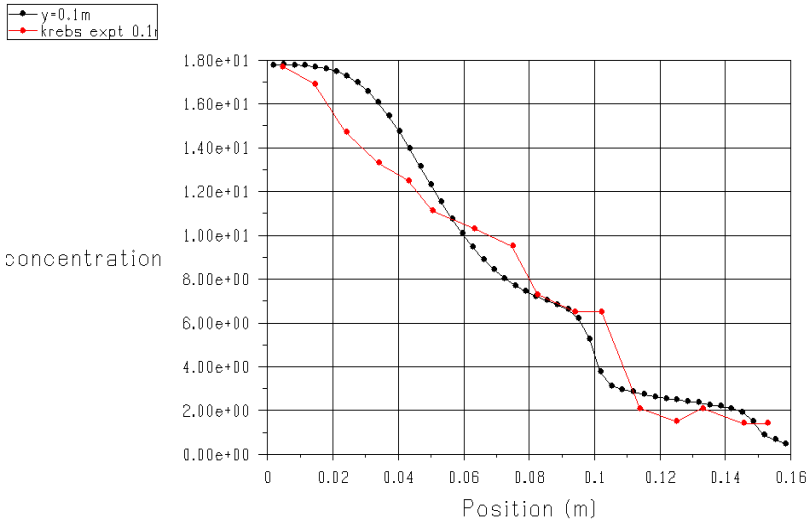
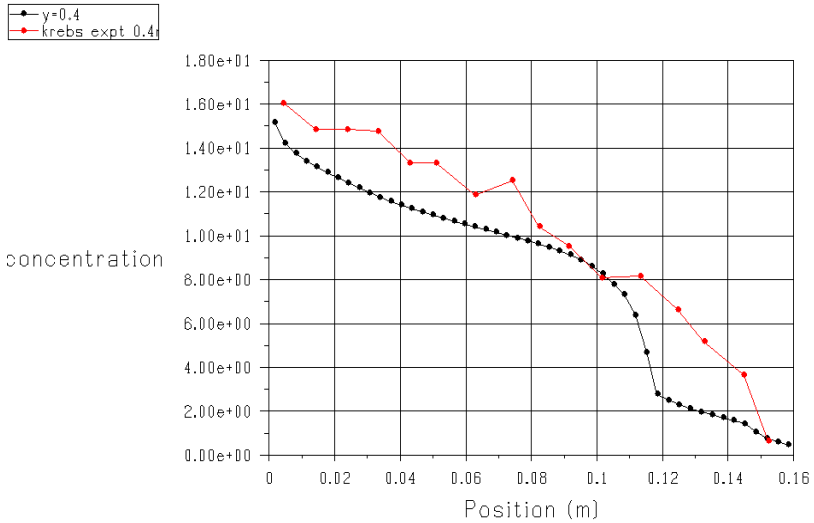
Concentration in Model Settling Tank





Total concentration at 0.1m along tank (left) and 0.4m along tank (right). Red is experiment, black calculated.

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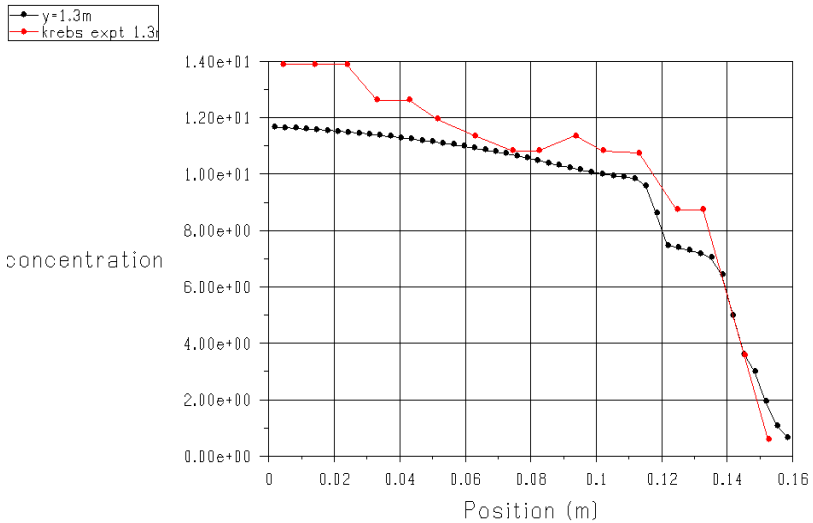
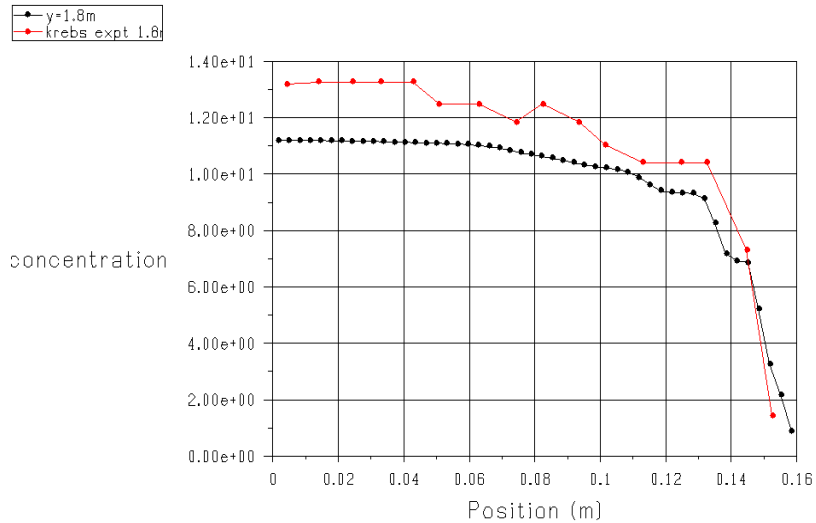
Concentration in Model Settling Tank





Total concentration at 1.3m along tank (left) and 1.8m along tank (right). Red is experiment, black calculated.

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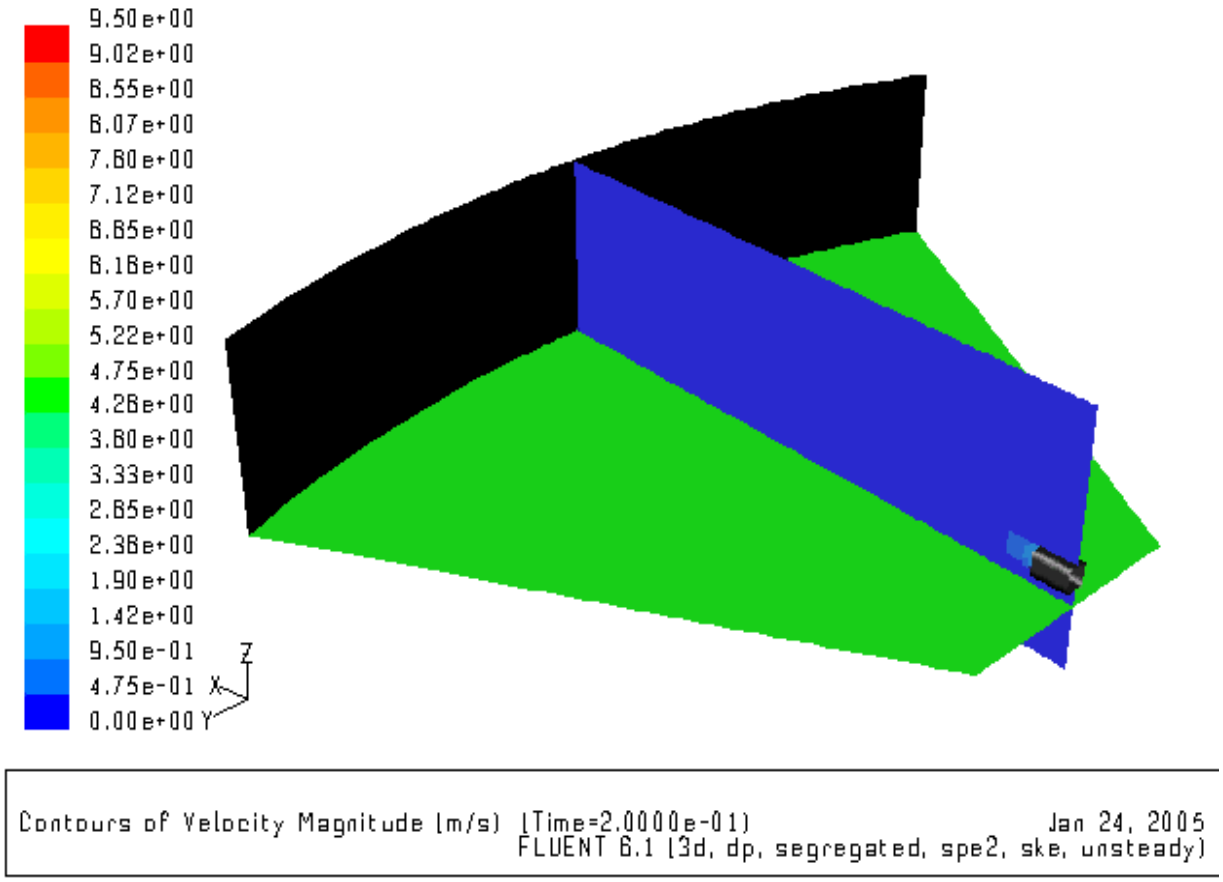
Glass Particles in Settling Tank - Scalars

Evolution of concentration over first 85 seconds.

Sludge Erosion (Horizontal Jet - SAGE226)



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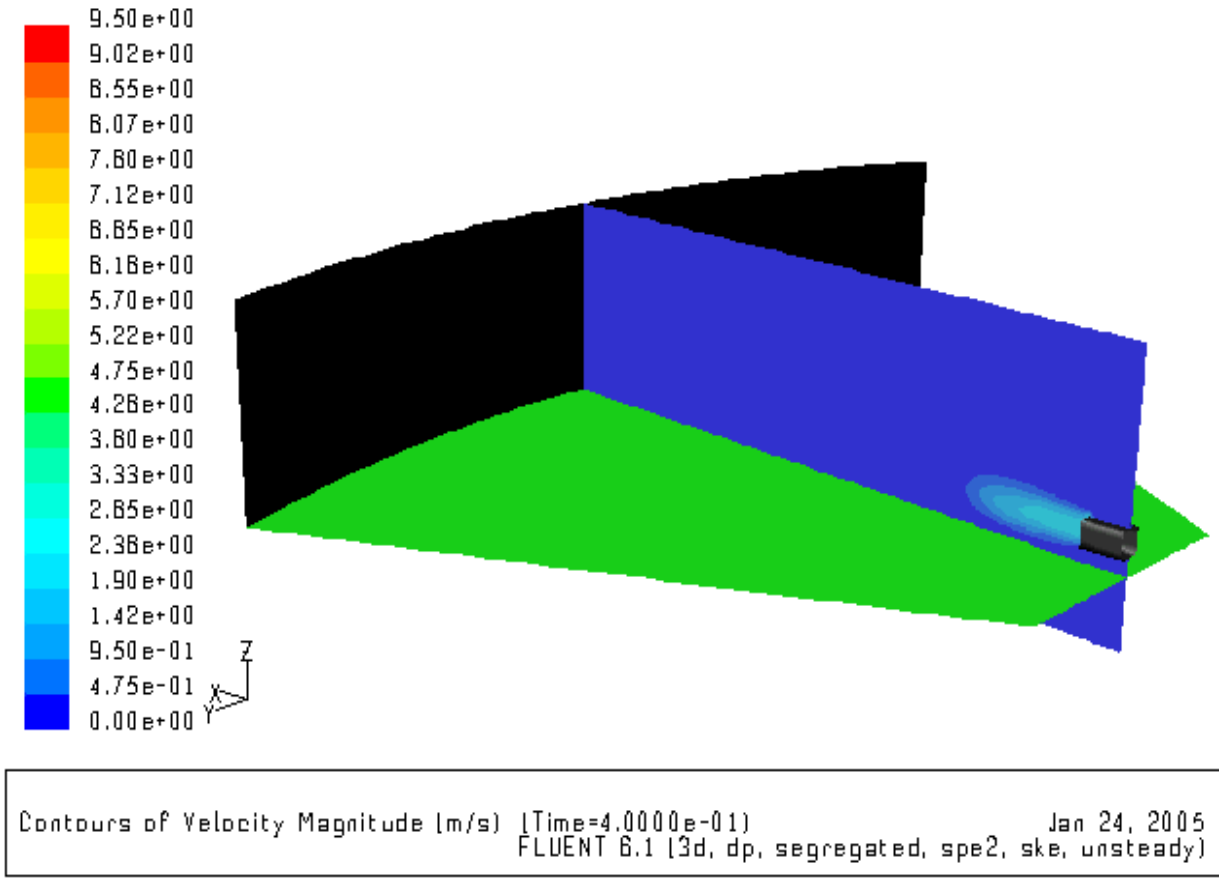
Sludge Erosion (Horizontal Jet - kaolin)



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Windscale Pile 1 Safety

- **The Challenge**

To show that the unlikely event of self-heating due to uranium hydride oxidation in the core of Windscale Pile 1 will not lead to a fire.

To enable a reduction in the cost of surveillance and emergency response cover.

To enable physical intrusion into the core, leading to cost effective decommissioning options.

Windscale Pile 1 Safety

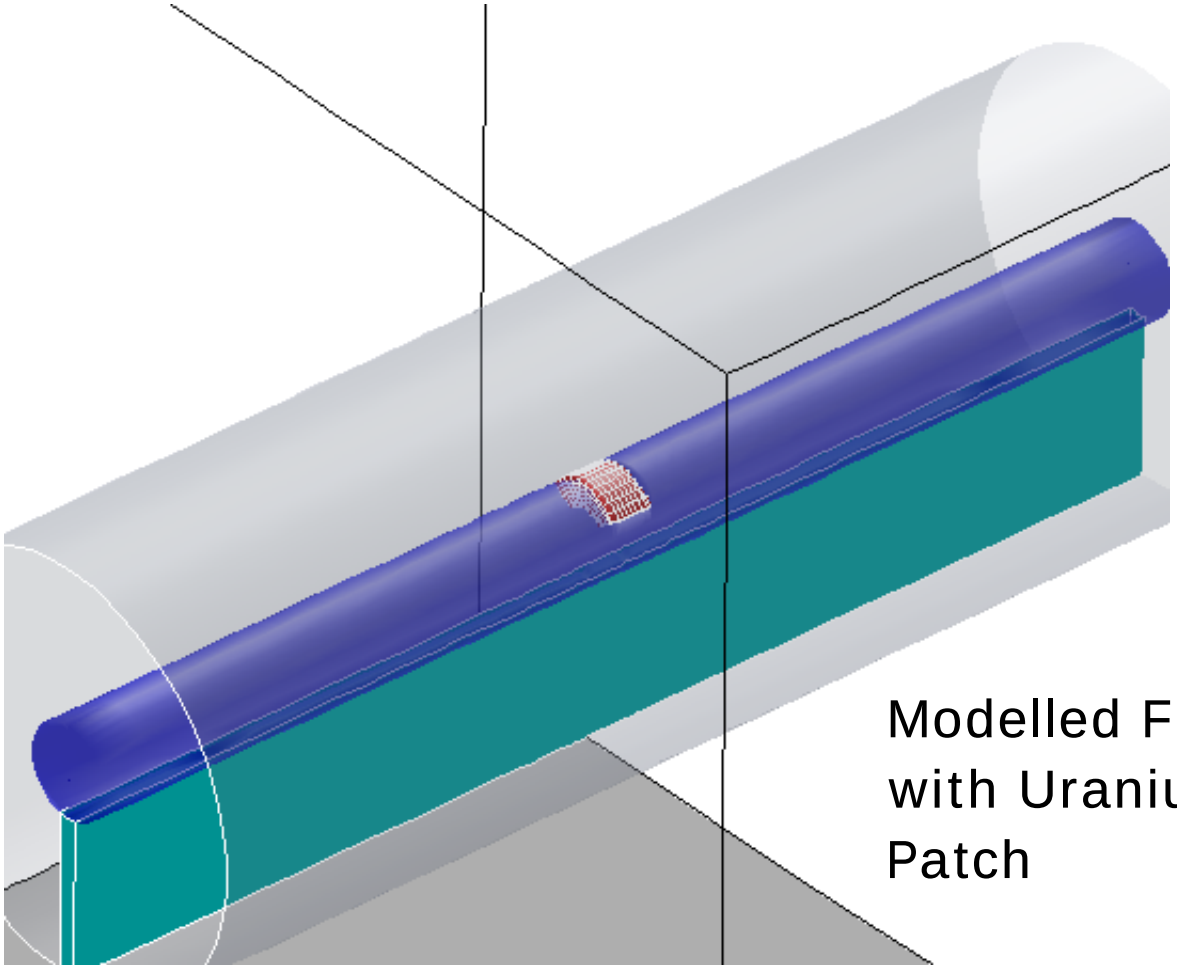
- **The Solution**

Using Computational Fluid Dynamics (CFD) technology and the latest data available for the chemistry of hydride:

The behaviour of the core was predicted under quiescent and accident scenarios;

Rigorous examination of the work was carried out, being reviewed by national and international experts.

Windscale Pile 1 Safety



Modelled Fuel Element with Uranium Hydride Patch

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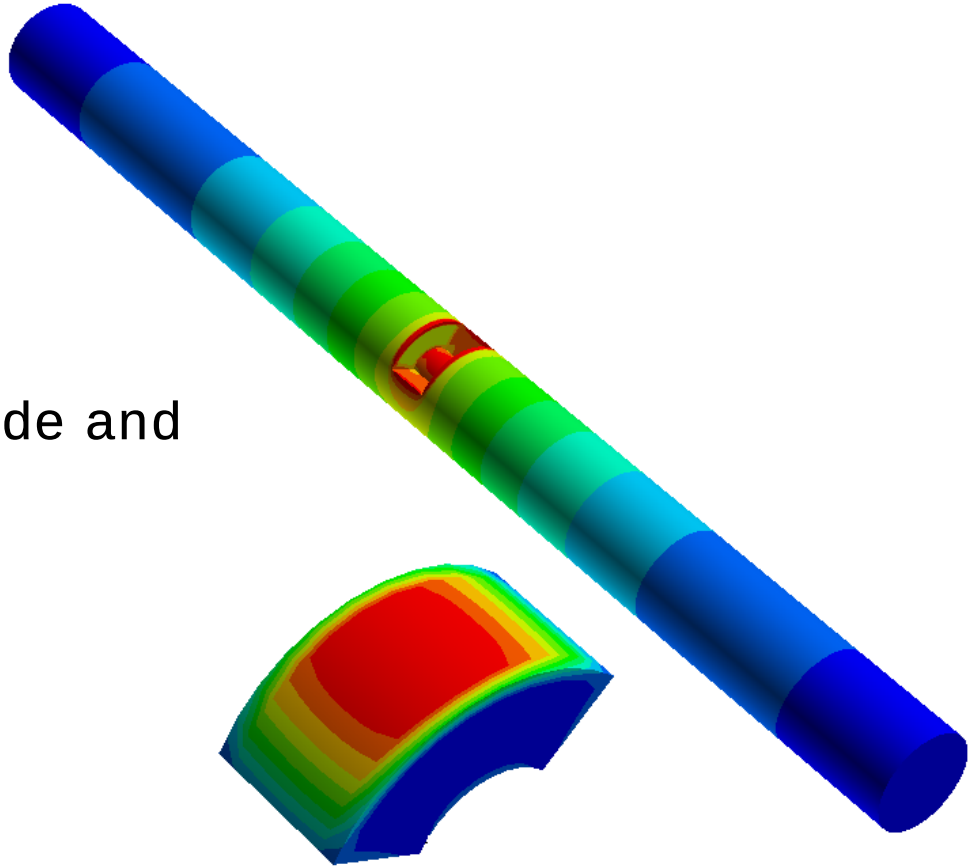
Windscale Pile 1 Safety

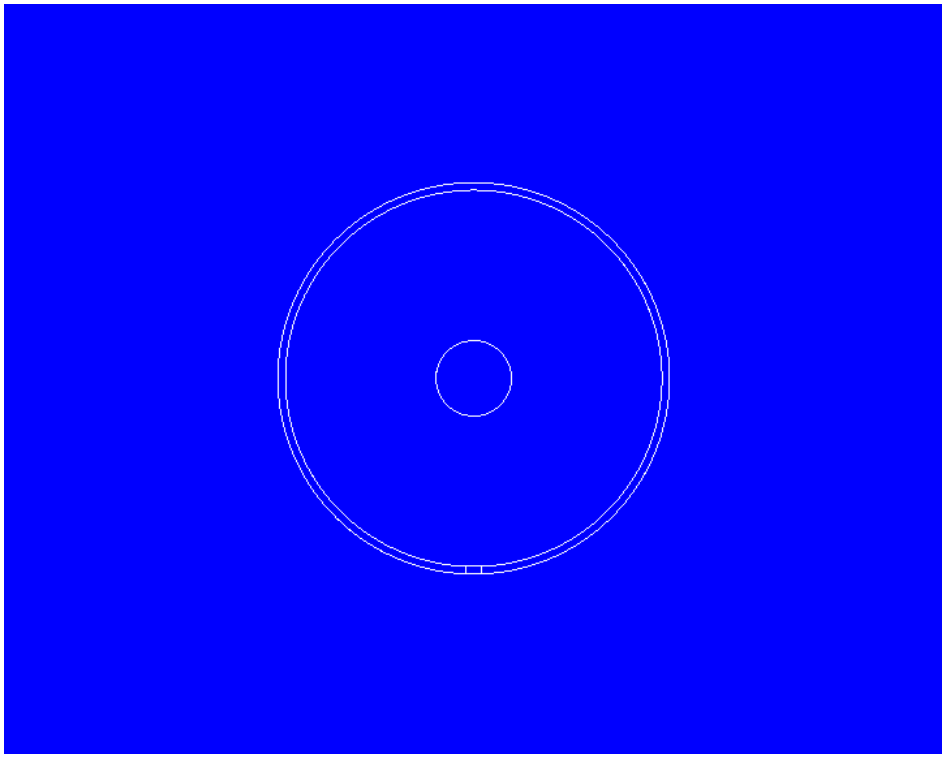
Predicted hydride and fuel element temperatures



Windscale Pile 1 Safety

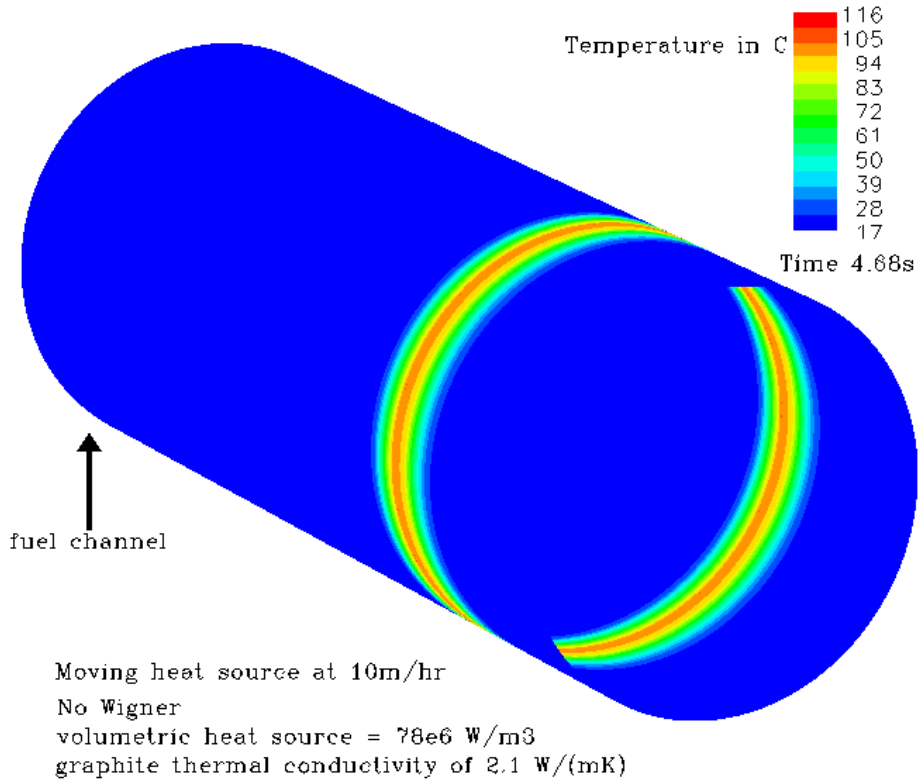
Additional work has now included the presence of stored energy in the graphite core:

Predicted stored energy power density in graphite surrounding a fuel channel



Windscale Pile 1 Safety

Moving heat sources have been represented for decommissioning:



Predicted fuel channel wall temperature distribution

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Windscale Pile 1 Safety

- **The Benefit**

Reassessment of current safety management arrangements leading to a saving of thousands of pounds per year.

The work has shown that large quantities of inert gas are not required for decommissioning, saving millions of pounds and making the process safer and quicker.

Swirly Flow

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Date: 16/01/08

Settling, Pumping Power and Wear

- Circular section encourages particles to collect at the bottom of a pipe. Fluid velocity must be high enough to prevent this
  - N.B. pumping power $\propto$ velocity^3
- High velocity causes pipe wear
  - impact wear
  - Ploughing wear
  - N.B.
    wear per tonne $\propto$ velocity$^{2.5}$
    wear per annum $\propto$ velocity$^{3.5}$

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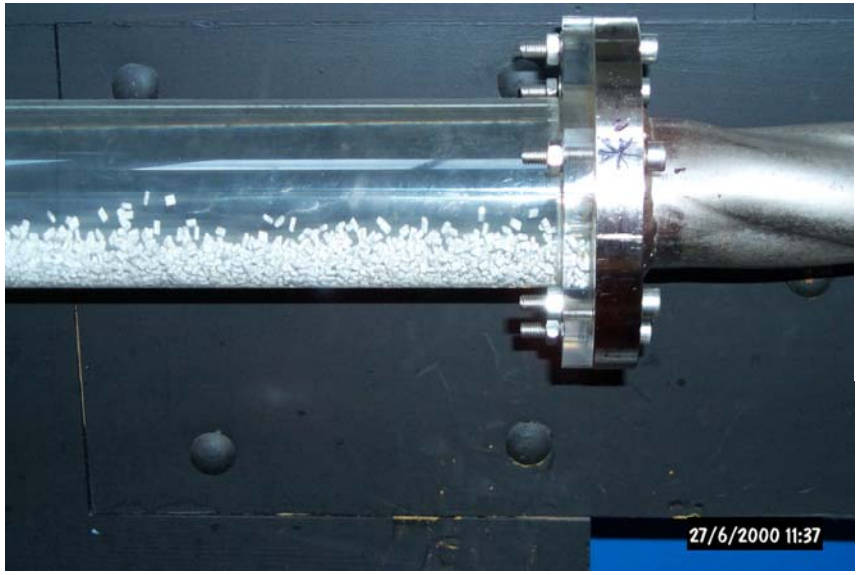
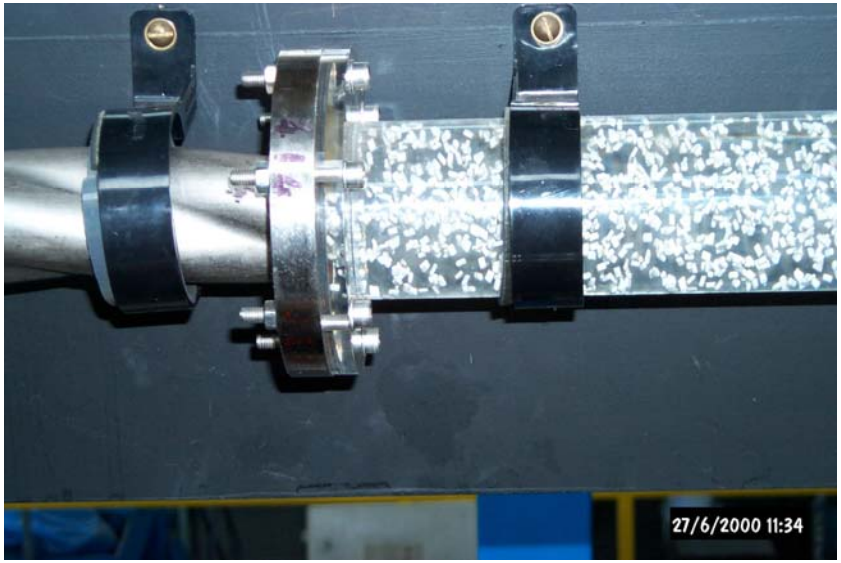
Slurry Transfer

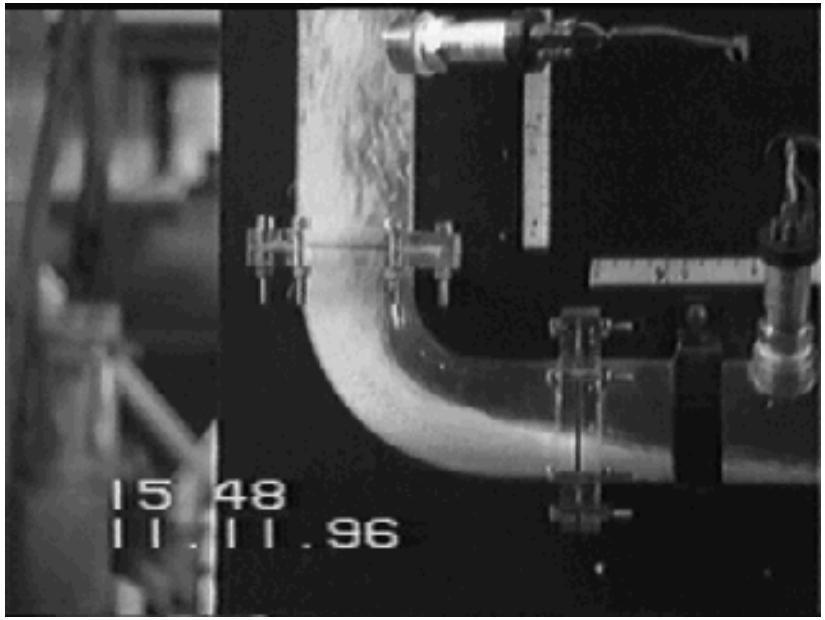




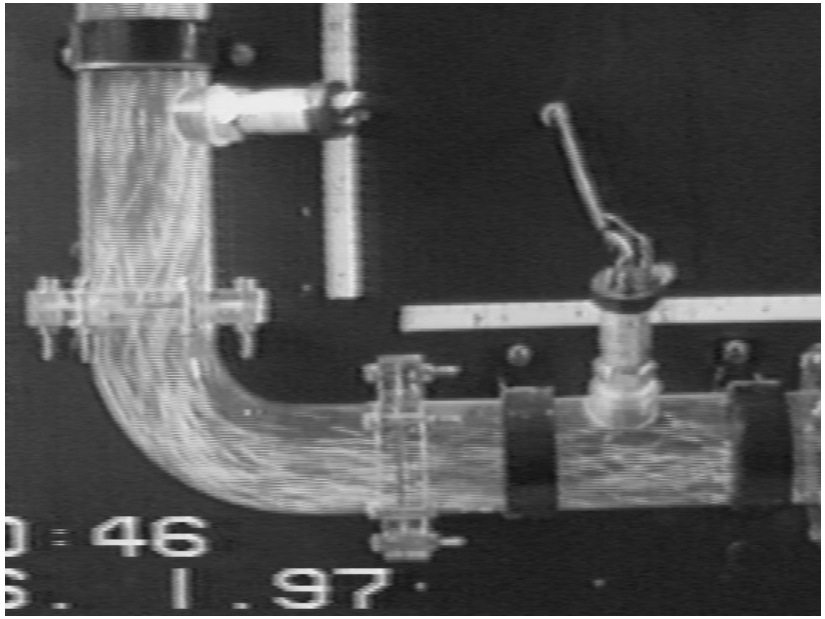
Slurry Transfer

Slurry Transfer

Slurry Transfer



Original



Swirl

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Imaging and Tomography at Sellafield in the UK

Dominic Rhodes & Tim Tinsley
Nexia Solutions

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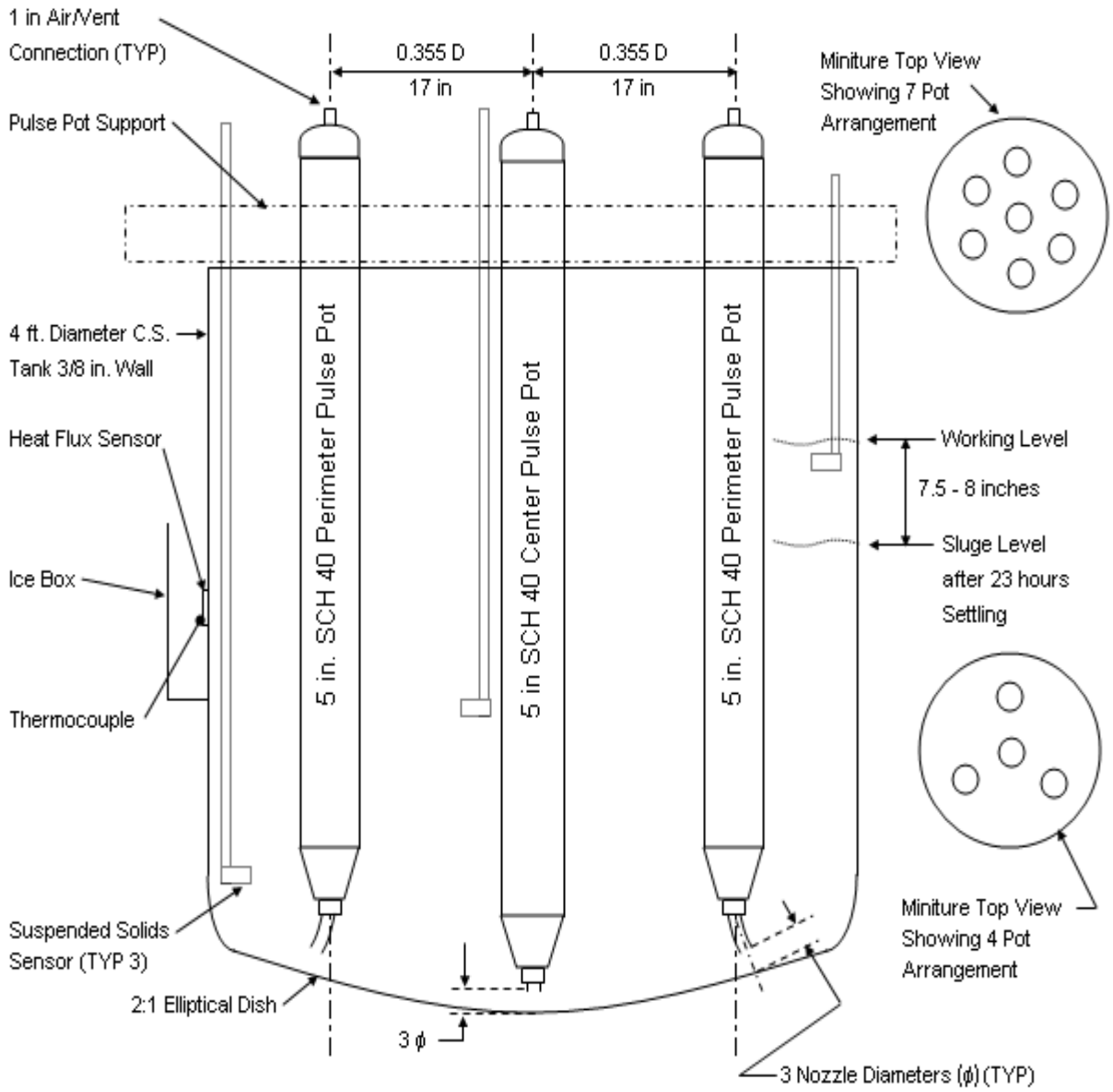
Date: 16/01/08

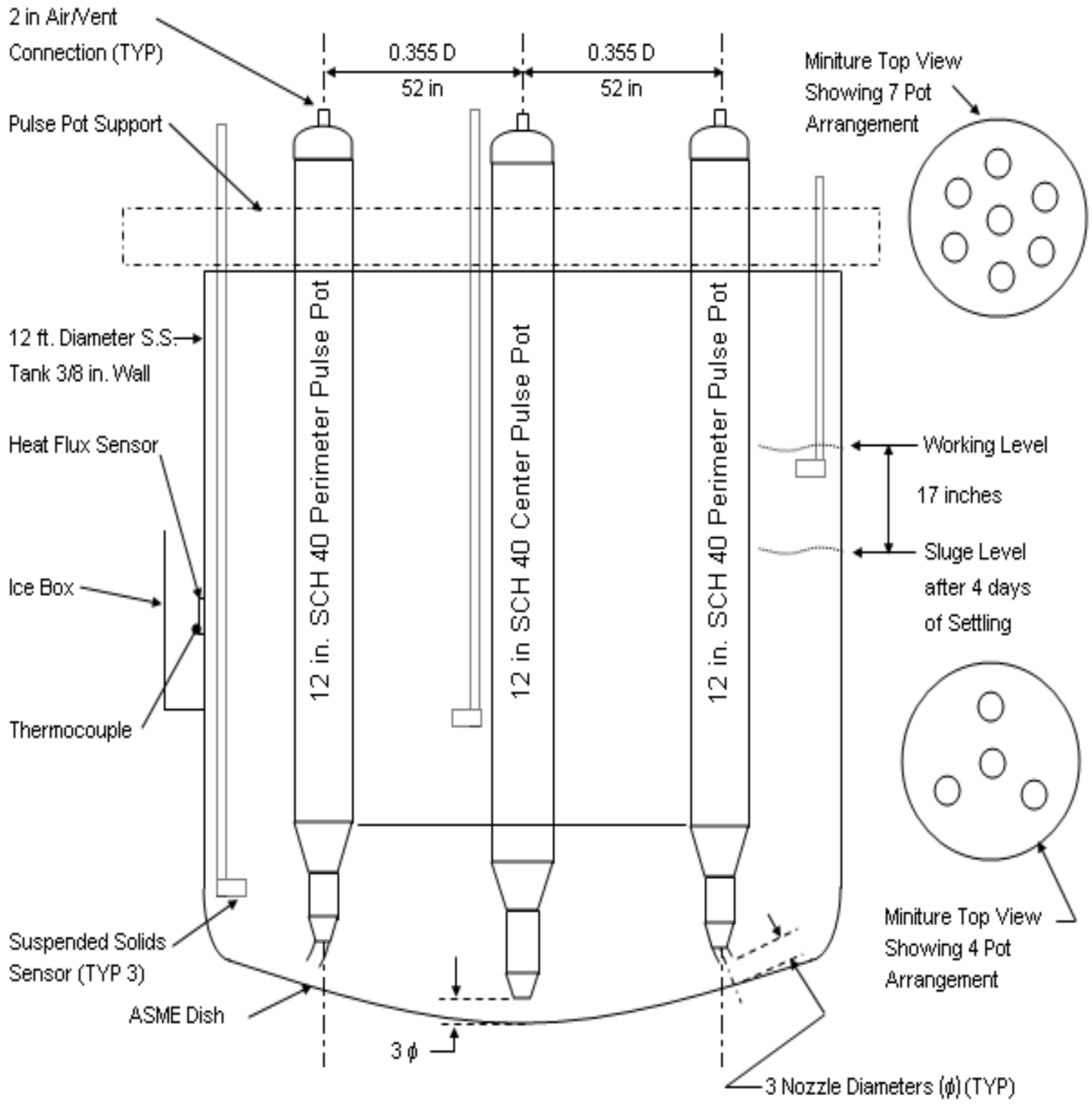
Mixing Within the Salt Waste Processing Facility [SWPF] – Air Pulse Agitators (APA's)

Sam Shicks
Parsons
1080 Silver Bluff Road
Aiken, SC. 29803
(803) 643-2428 Office
(803) 643-2253 Fax
(803) 443-8017 Mobile
E-mail: Sam.Shicks@parsons.com

1/5 Scale Prototype (Tank ID = 47.25 in)



5/8 Scale Prototype (Tank ID = 12 ft)



Test Simulant

Item	1/5 Scale	5/8 Scale
Solids (MST/SRS Sludge Simulant)	8 w.t.%	5 w.t.%
Solution Density	9.83 lbm/gal	10.18 lbm/gal
Solution Viscosity	9 CP	18 CP
Mean Solids Settling Rate	0.35 in/hr	0.17 in/hr
Dilution = Sodium Hydroxide/Sodium Nitrate		
Solution Sodium Concentration	3.3 M	5.6 M

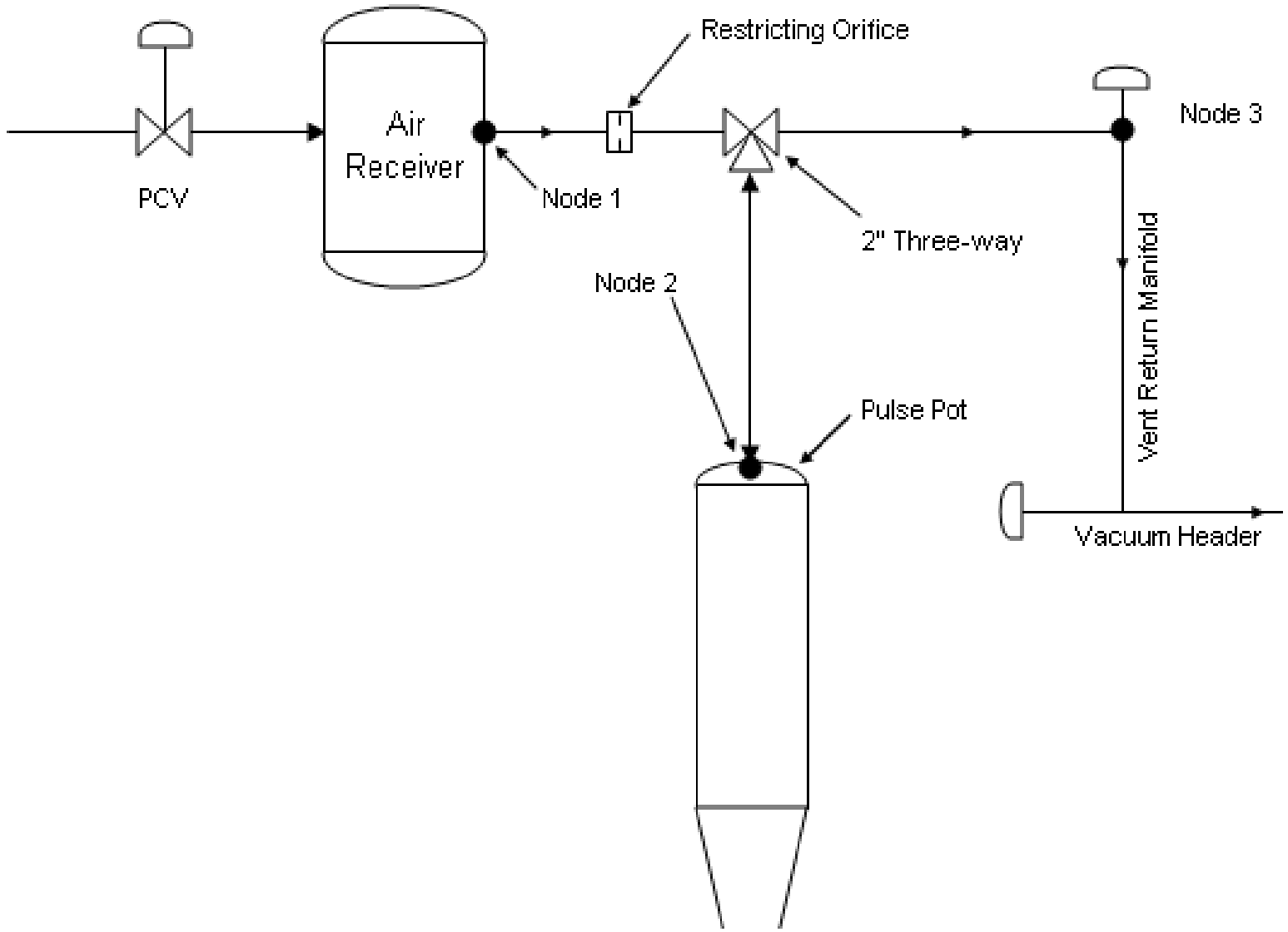
5/8 Scale Perimeter Pot Test Nozzle

5/8 Scale Center Pot Test Nozzle

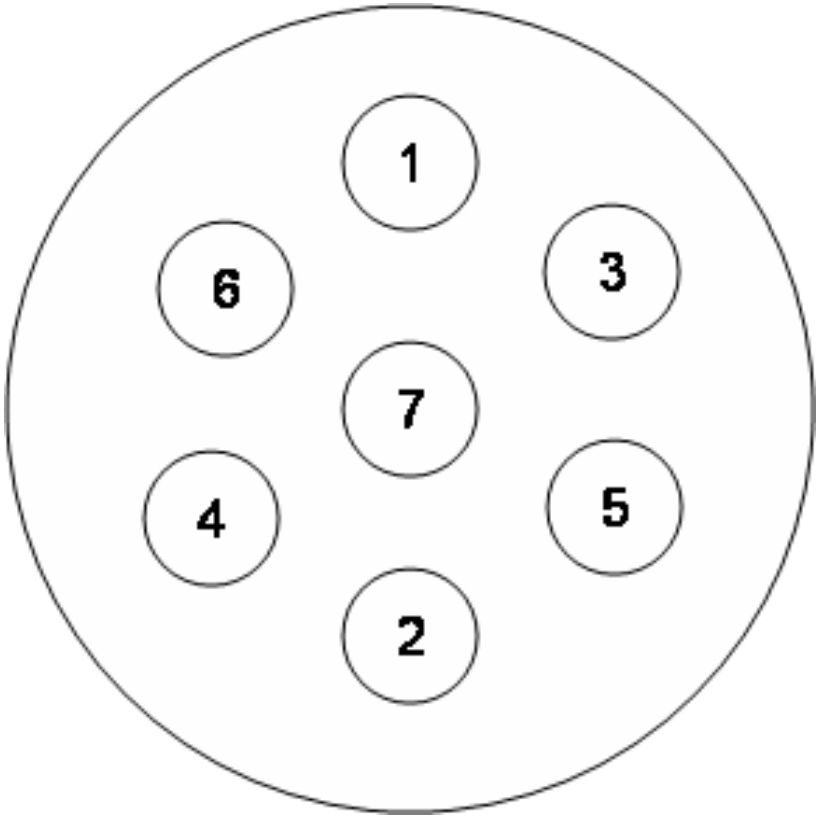
Markland 502-TP Submersible Solids Sensor

Markland 502-TP Submersible Solids Sensor

Baseline Components

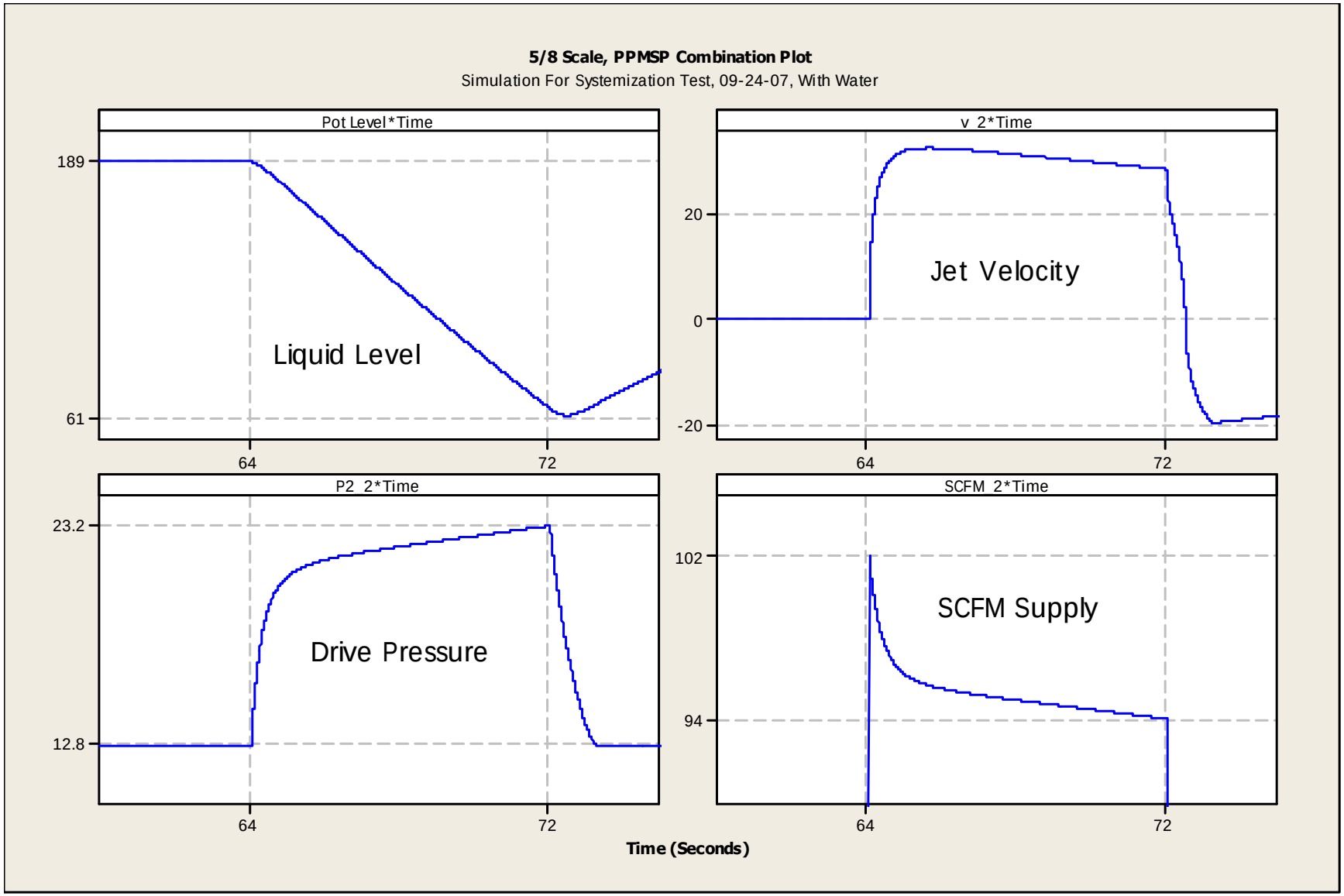


Baseline Operating Mode

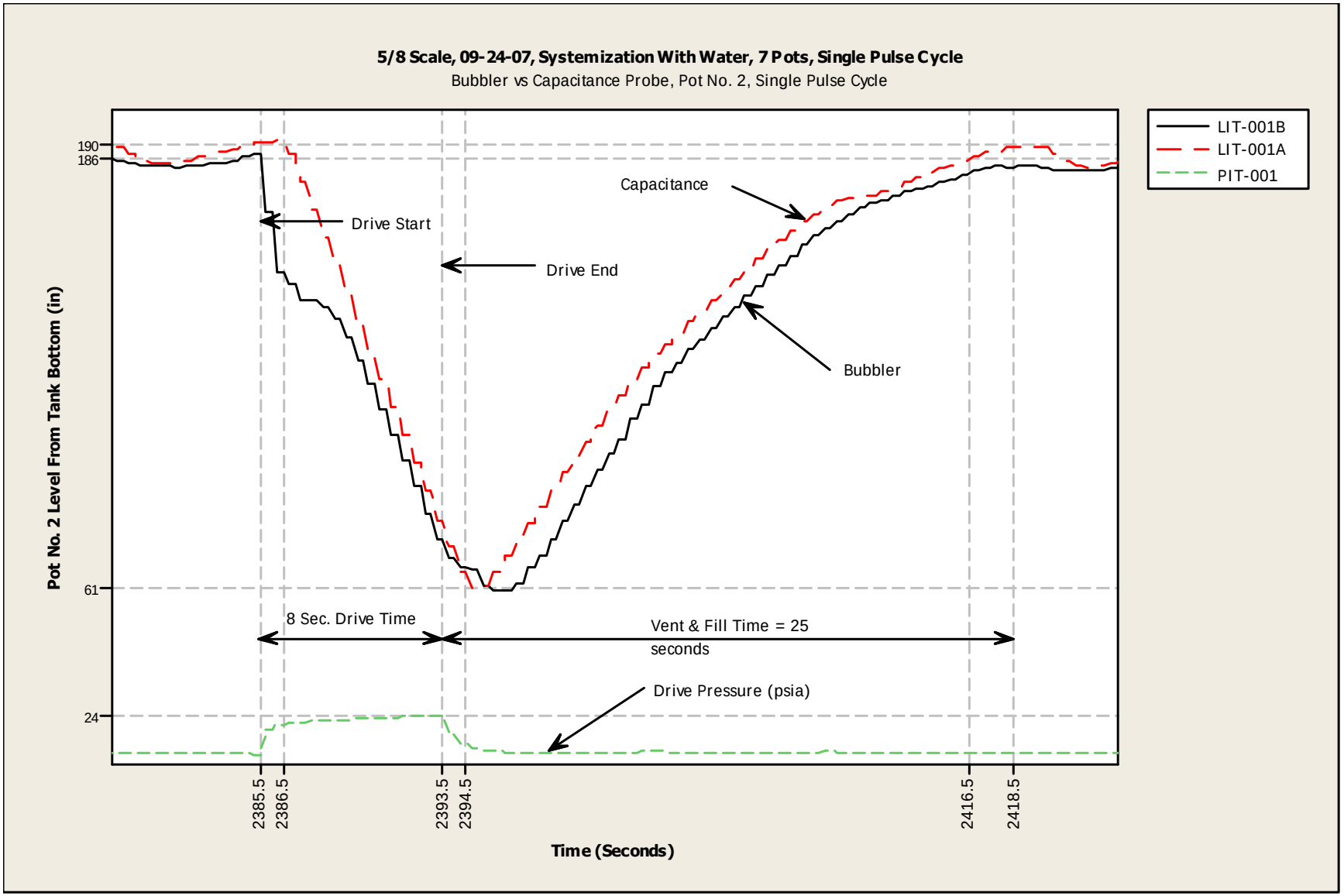


Pots are pulsed one at a time in a cross-fire sequence

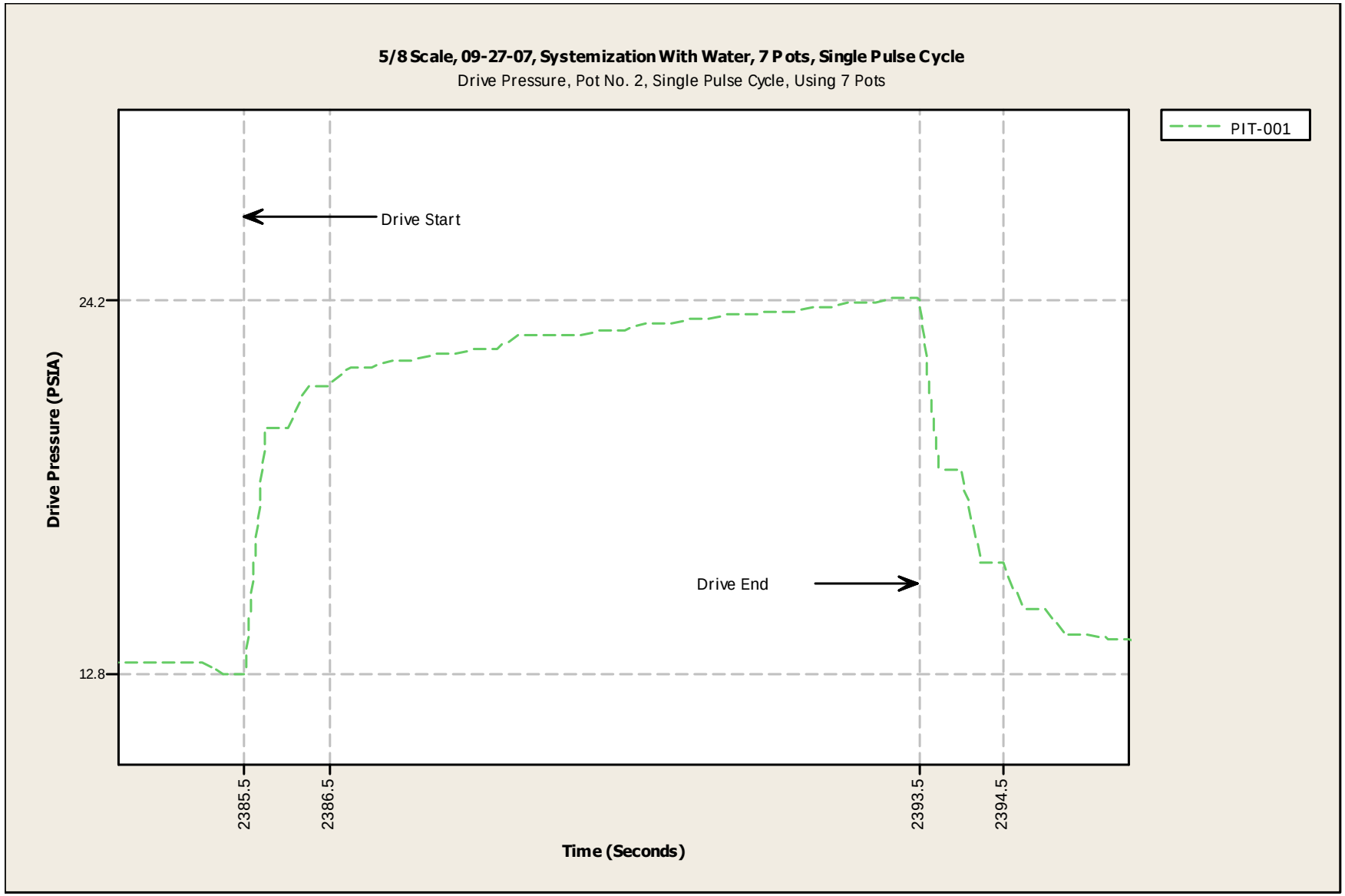
PPMSP Simulation Results



Measured Liquid Level In Pot



Measured Drive Pressure In Pot



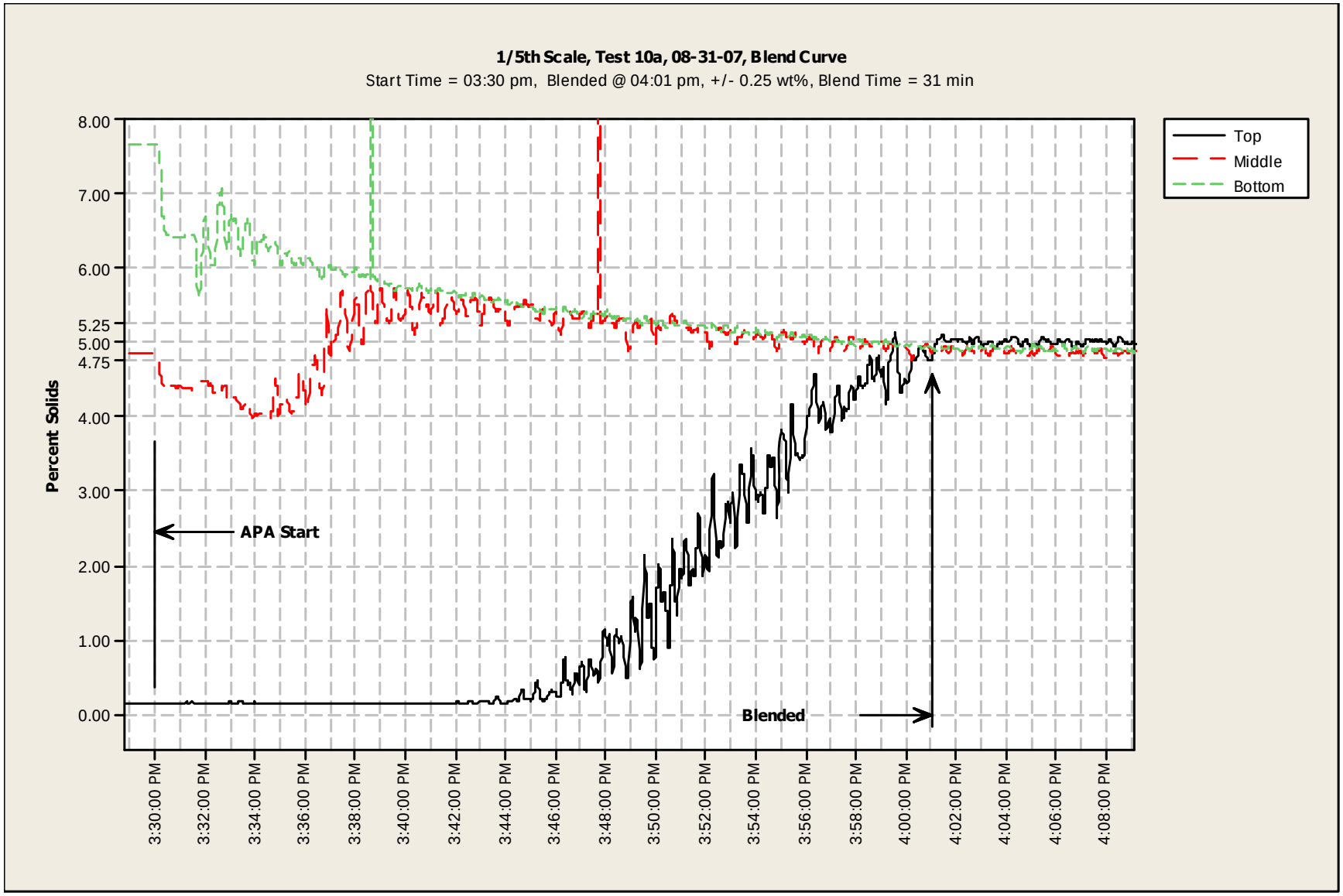
1/5 Scale Blend Tests

- 3 Tests (3/4 in Nozzle)(4-Pots)(High Level)
- 3 Tests (3/4 in Nozzle)(4-Pots)(Mid Level)
- 3 Tests (3/4 in Nozzle)(7-Pots)(High Level)
- 3 Tests (3/4 in Nozzle)(7-Pots)(Low Level)
- 3 Tests (1 in Nozzle)(4-Pots)(High Level)
- 3 Tests (1 in Nozzle)(4-Pots)(Mid Level)
- 3 Tests (1 in Nozzle)(7-Pots)(High Level)
- 3 Tests (1 in Nozzle)(7-Pots)(Low Level)

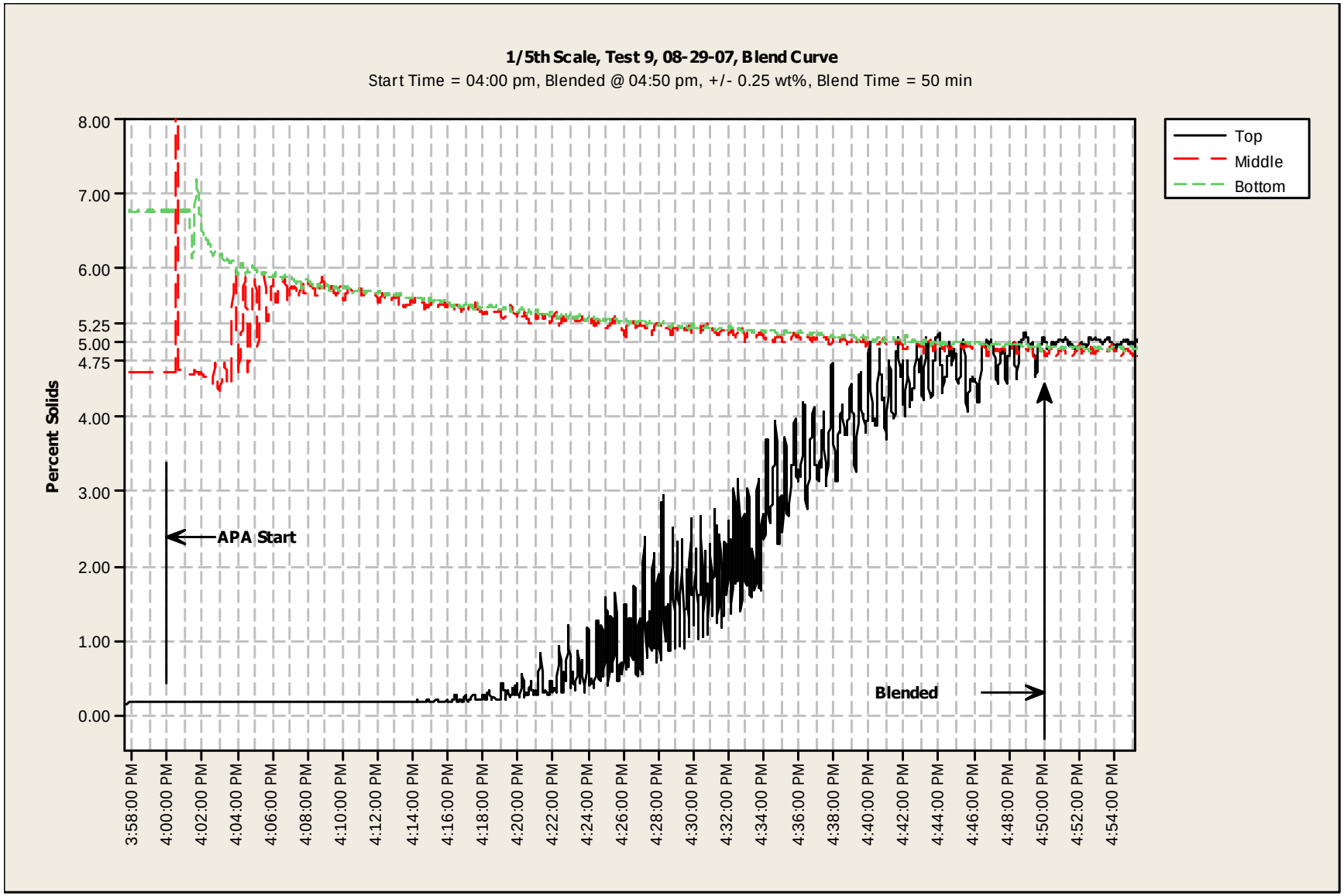
5/8 Scale Blend Tests

- 2 Tests (7-Pots)(High Level)
- 3 Tests (4-Pots)(High Level)

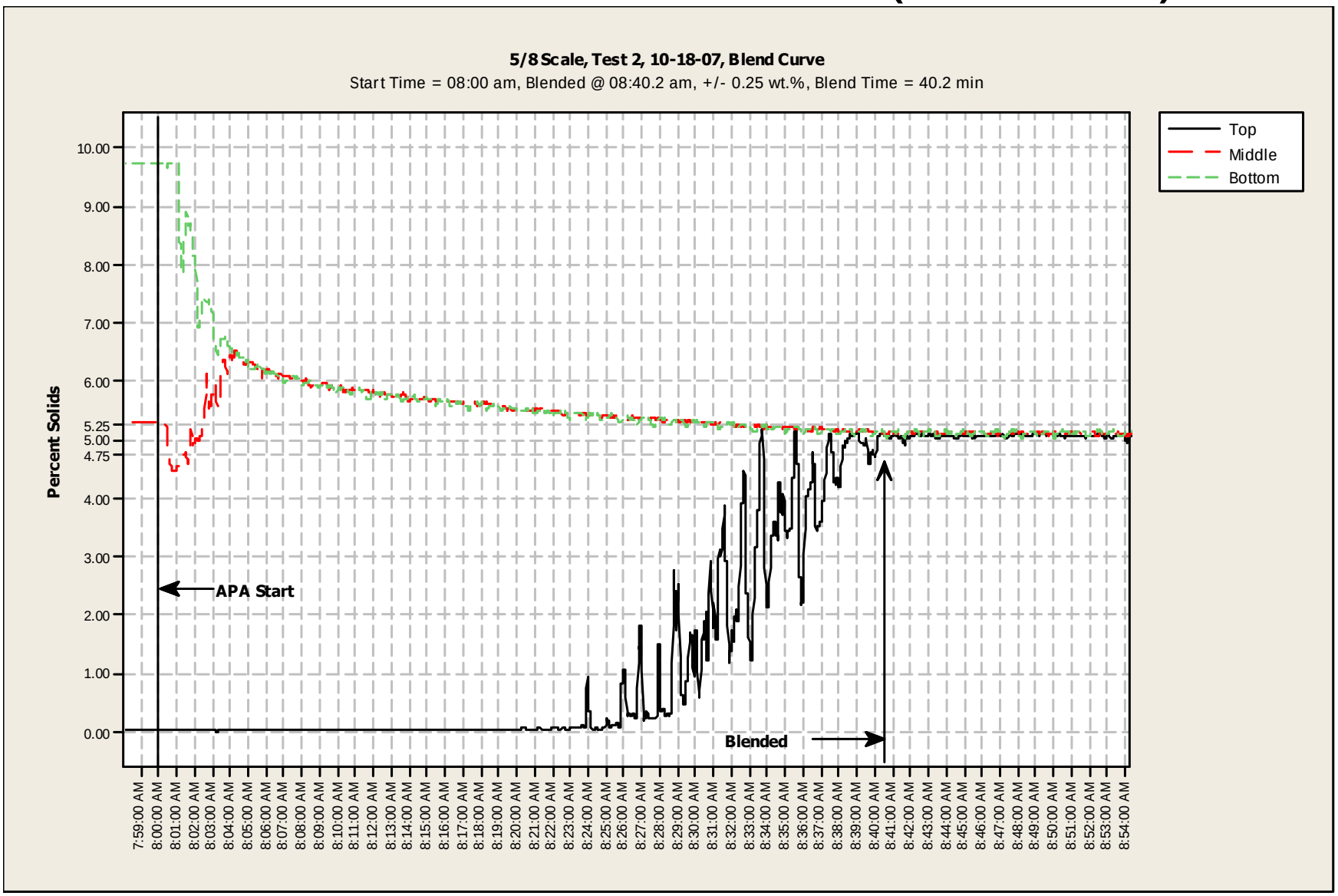
1/5 Scale Blend Curve (TEST 10a)



1/5 Scale Blend Curve (TEST 9)



5/8 Scale Blend Curve (TEST 2)



Technical Basis

First:

Listed

Variables

1.	σ	Average rate of circulation (ft^3/sec)
2.	υ	Average jet velocity (ft/sec)
3.	V	Volume of liquid inside test tank (ft^3)
4.	W_L	Working level of liquid inside test tank (ft)
5.	S_L	Initial sludge level inside test tank (ft)
6.	ϕ	Internal diameter of pulse jet nozzle (ft)
7.	γ	Specific weight of test fluid (lbf/ft^3)
8.	A	Liquid surface area inside the test tank (ft^2)
9.	P	Average jet power (ft-lbf/sec)
10.	D	Internal diameter of test tank (ft)
11.	S	Total suspended solids concentration (lbm/ft^3)
12.	R	Solids setting rate (ft/sec)
13.	μ	Dynamic viscosity of test liquid (lbf-sec/ft^2)
14.	D_H	Tank hydraulic diameter (ft)
15.	ω	Pulse jet frequency (1/sec)

Technical Basis

Then:
Dimensional
Analysis

$$\Pi_t = \frac{t\ \sigma^4\ \gamma^3}{P^3} = \frac{t\ a\ g^3}{v^5}$$

$$\Pi_V = \frac{V\ \sigma^3\ \gamma^3}{P^3} = \frac{V\ g^3}{v^6}$$

$$\Pi_L = \frac{L\ \sigma\ \gamma}{P} = \frac{L\ g}{v^2}$$

$$\Pi_S = \frac{S\ \sigma^7\ \gamma^4}{g_c\ P^5} = \frac{S\ a^2\ g^4}{\rho\ v^8}$$

$$\Pi_R = \frac{R\ P^2}{\sigma^3\ \gamma^2} = \frac{R\ v^3}{a\ g^2}$$

$$\Pi_\mu = \frac{\mu\ \sigma^5\ \gamma^3}{P^4} = \frac{\mu\ a\ g_c\ g^3}{\rho\ v^7}$$

$$\Pi_{Ai} = \frac{A_i\ \sigma^2\ \gamma^2}{P^2} = \frac{A_i\ g^2}{v^4}$$

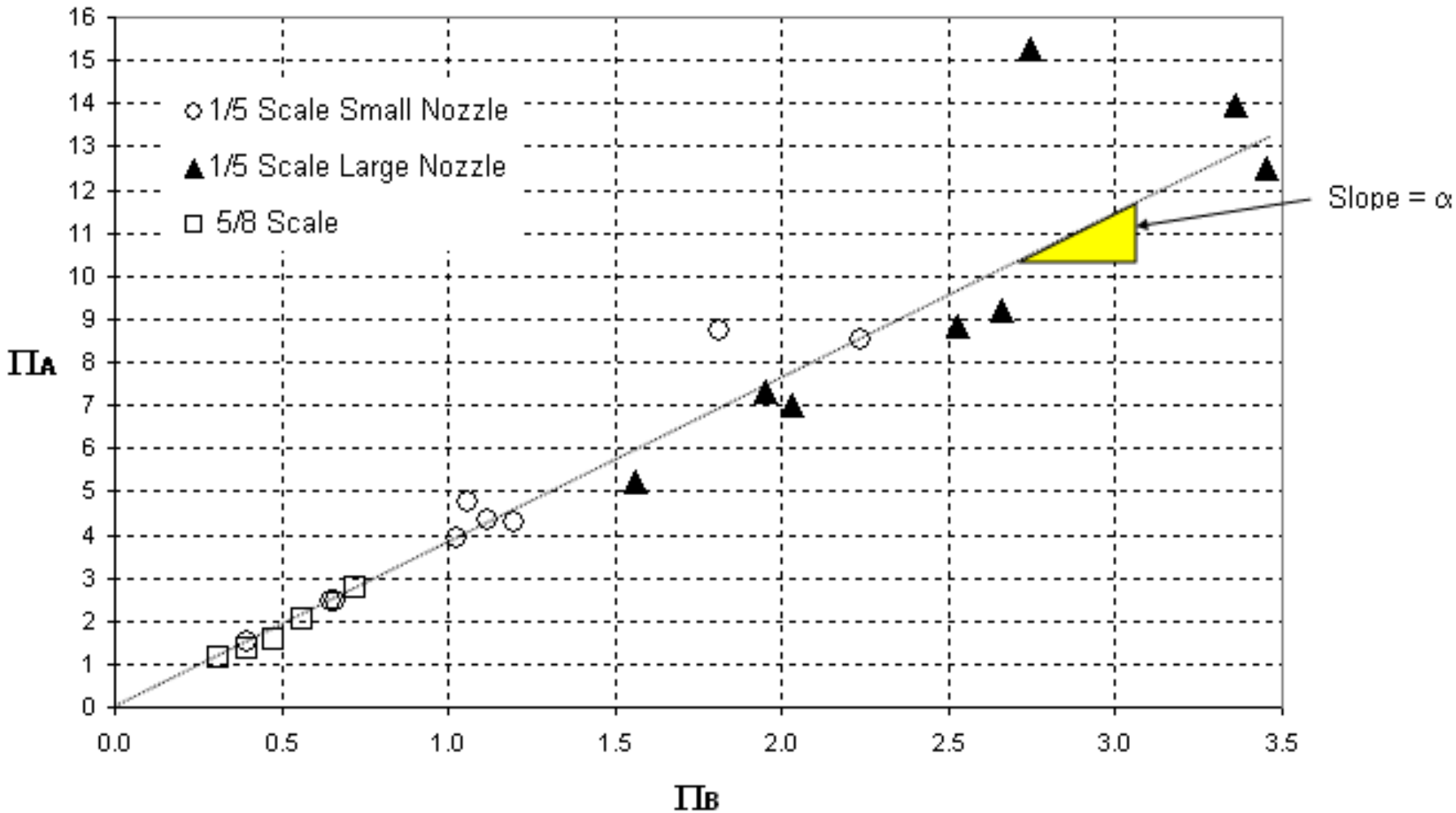
$$\Pi_\omega = \frac{\omega\ P^3}{\sigma^4\ \gamma^3} = \frac{\omega\ v^5}{a\ g^3}$$

?

$$\Pi_3 = \Pi_1^{a} \times \Pi_2^{b}$$

$\Pi_A = \frac{\sigma \quad t}{V}$	Turnovers to blend
$\Pi_{B1} = \frac{W_L}{D}$	The ratio of the working level to the tank internal diameter
$\Pi_{B4} = \frac{W_L}{D_H}$	The ratio of the working level to the hydraulic diameter
$\Pi_{C1} = \frac{v}{\sqrt{S_L\, g}}$	Froude number based on the initial sludge level
$\Pi_{C4} = \frac{v}{\sqrt{D_H\, g}}$	Froude number based on the hydraulic diameter

Blend Scale-Up Model



Blend Scale-Up Model

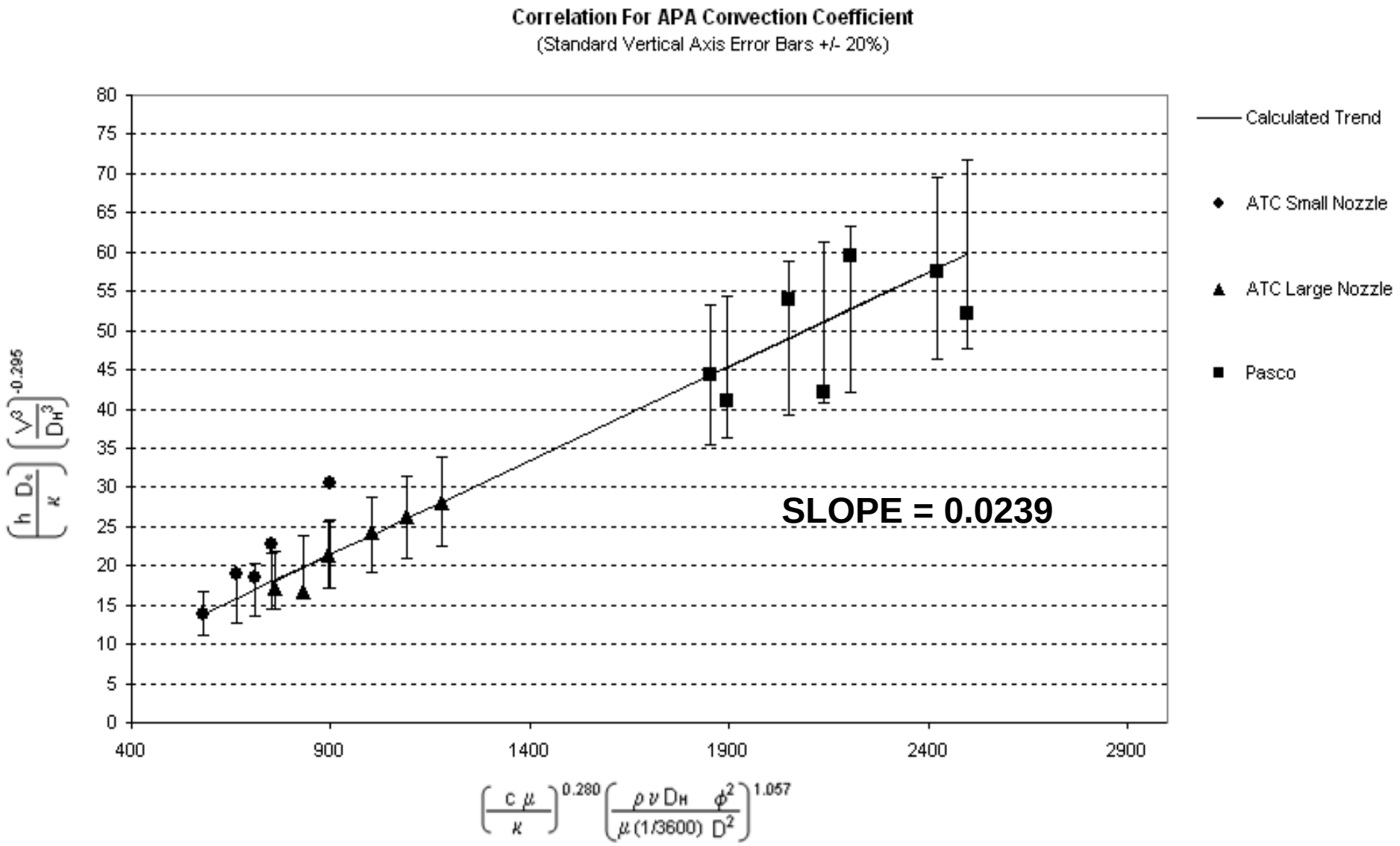
The SWPF APA Blend Model is represented by the linear expression

$$\frac{\sigma \quad t}{V} = 3.82 \left(\frac{W_L}{D}\right)^{3.20} \left(\frac{W_L}{D_H}\right)^{5.88} \left(\frac{\nu}{\sqrt{S_L\, g}}\right)^{4.01} \left(\frac{\nu}{\sqrt{D_H\, g}}\right)^{-7.68}$$

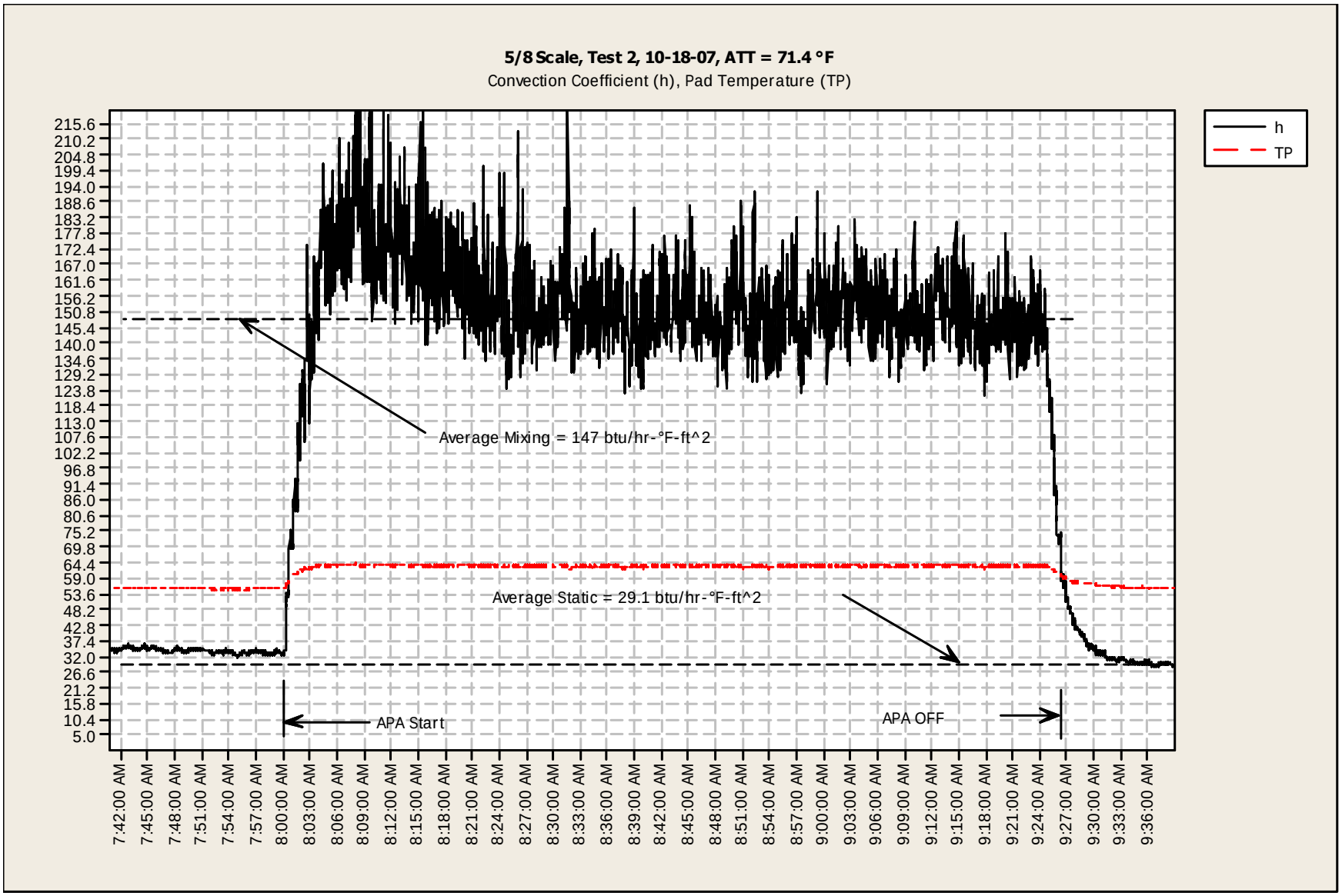
The limitations to the accuracy of this equation are such that

1.0 $5 < (\text{wt.\% Solids}) < 8$
2.0 1/5 Scale < dynamic viscosity < 5/8 Scale
3.0 1/5 Scale < Specific Gravity < 5/8 Scale
4.0 $1.95 < D_H < 8.58$
5.0 $0.684 < S_L/W_L < 0.875$
6.0 $0.933 < W_L/D < 1.43$
7.0 Used for SWPF LRW Simulated Sludge
8.0 $1.0 < (\sigma\, t / V) < 9.5$
9.0 $0.19 < (\phi / D) < 0.254$

Convection Coefficient



Convection Coefficient



Slurry Suspension

David S. Dickey

MixTech, Inc.

What We Know about Mechanical Agitation

- Thoroughly studied for 40 years
- Characteristics
  - off-bottom suspension
  - on-bottom motion
  - uniform suspension
  - cloud height
- Visual observation
- Reliable means of measurement

What We Don’t Know about Pulse Jet Mixers

- We don’t know how a pulse will effect solids suspension
- Pulsed operation will not be better than continuous agitation
- We need to know about the effects of geometry on solids suspension
- We need to know how to scale-up

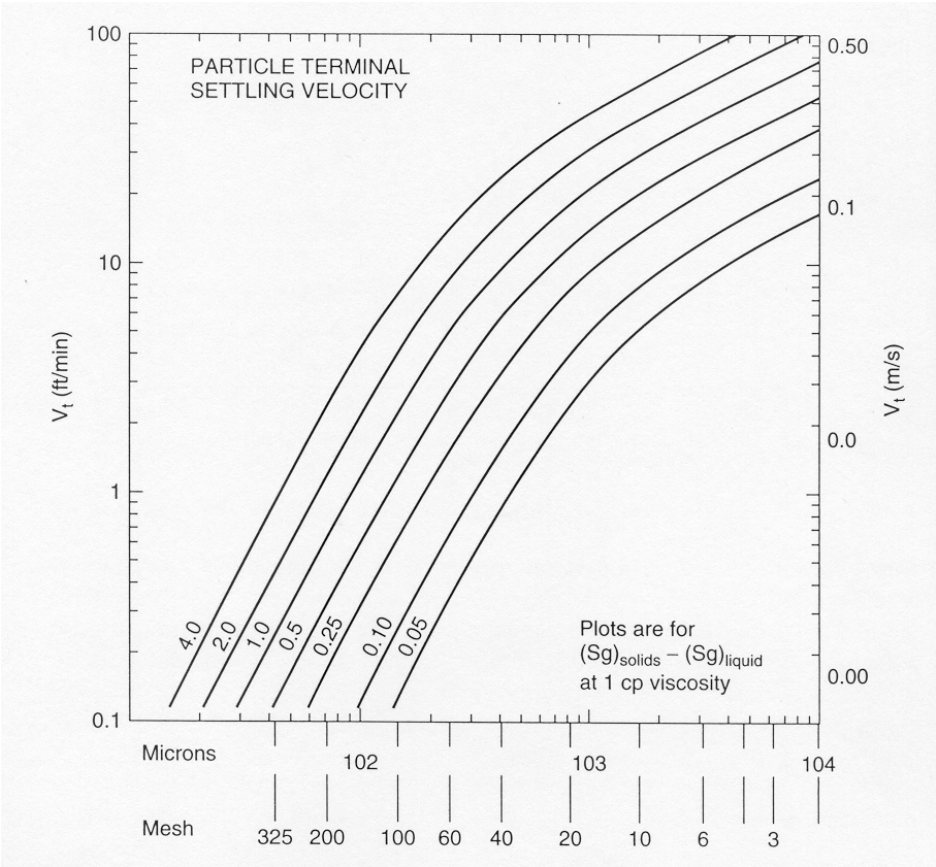
Everything We Know about Solids Suspension is Scale-up

- All of solids suspension is empirical
- Observation of suspension characteristics
- Testing in different scales
- Scale-up of small-scale tests

Difficulty

- Primary factor – settling velocity
  - with density adjustment
- Many other factors involved
  - mixer geometry factors
  - particle property factors
  - fluid property factors

Terminal Settling Velocity



Terminal Settling Velocity

- Settling due to density difference
- Slow settling
  - laminar
  - Stokes flow
- Rapid settling
  - turbulent
  - Newton flow
- Transition between laminar and turbulent

Agitated Suspension

- Not terminal settling velocity
- Agitated flow not vertical at all points
- Initial suspension by horizontal flow across bottom
- Suspension characteristics a stronger function of density than predicted by terminal settling velocity
- Density more important than diameter

Zwietering Correlation

$$N_{js} = S\,\nu^{0.1}\left[\frac{g_c\left(\rho_s - \rho_l\right)}{\rho_l}\right]^{0.45} X^{0.13}\, d_p^{\,0.2}\, D^{-0.85}$$

A.5.295

Density Effect on Terminal Settling Velocity

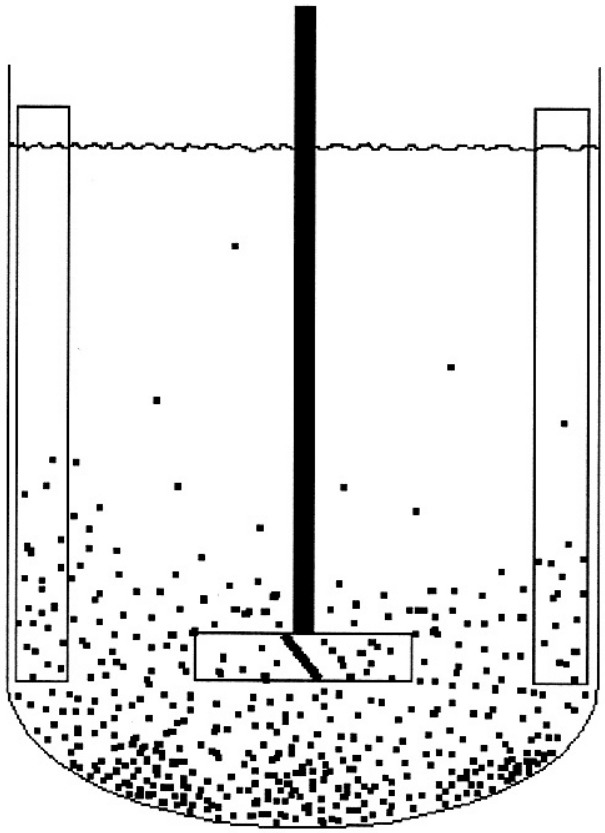
$$N_{js} = S' \left[\left(\frac{\rho_s - \rho_l}{\rho_l} \right) u_t \right]^{-0.28}$$

A.5.296

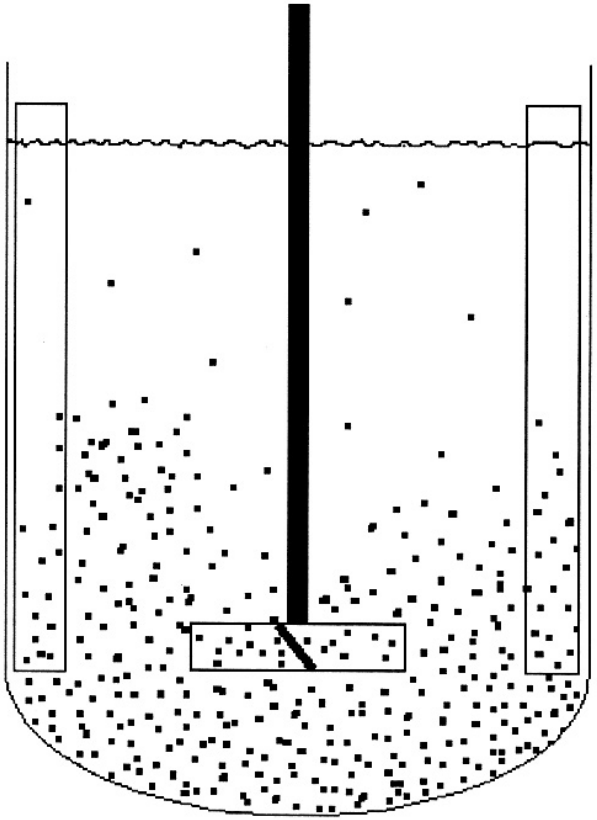
Mixing Intensity

- On-bottom motion
  - all particles moving
  - not necessarily suspended
- Off-bottom suspension
  - all particles suspended
  - cloud height
- Uniform suspension
  - all particles well distributed

On-Bottom Motion

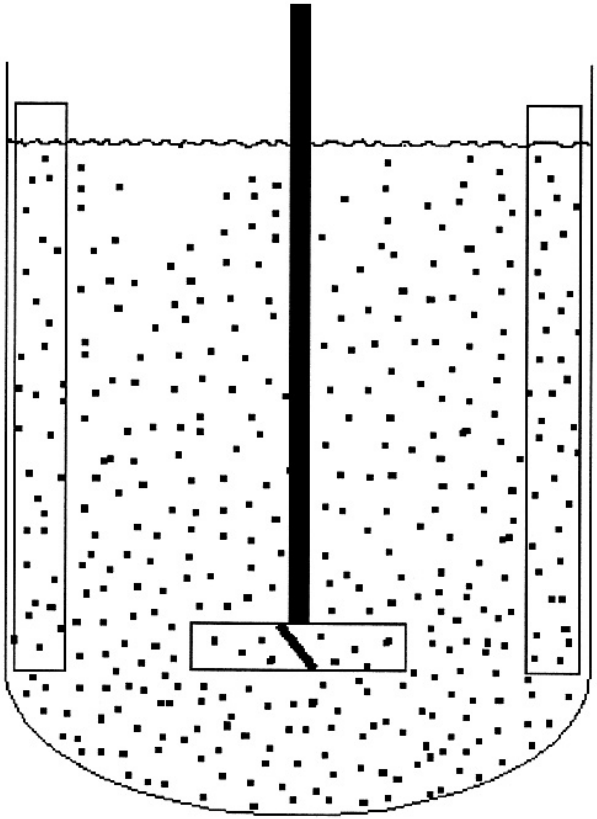


Off-Bottom Suspension



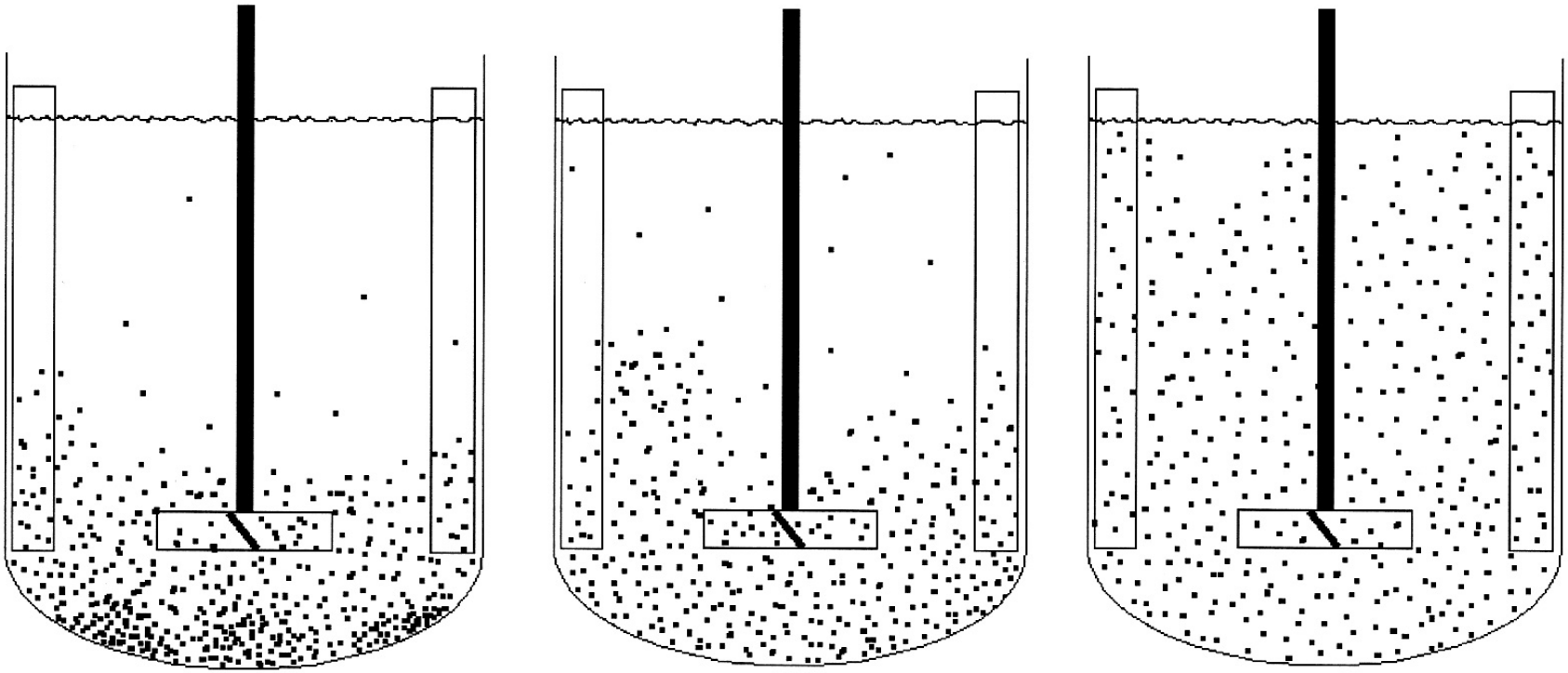
A.5.299

Uniform Suspension



A.5.300

Suspension Levels



On Bottom Motion

Off-Bottom Suspension

Complete Suspension

A.5.301

Mixer Design Factor

- **Just suspended speed – N_{JS}**
- **Classical correlation by Zwietering**
  - **adapted for practical use**
- **Other more complicated and more accurate correlations**
- **Testing and scale-up – best method**

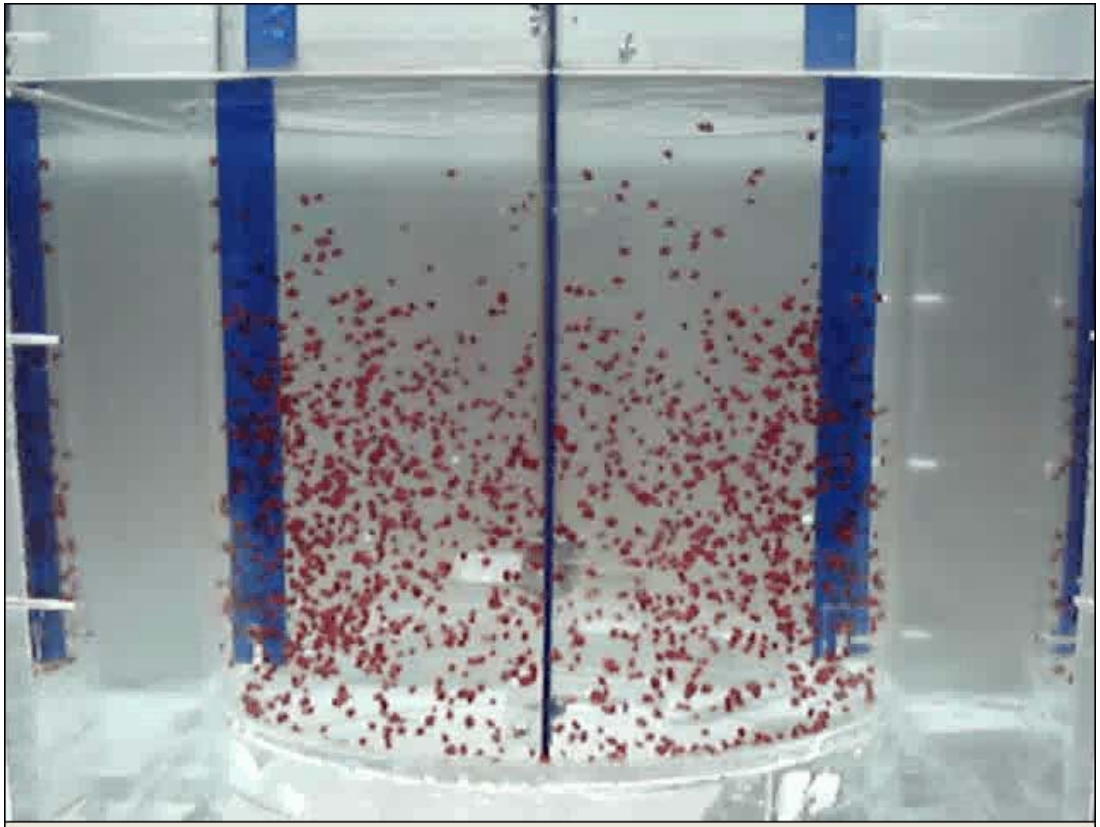
A.5.302

Mixer Geometry Factors

- Impeller type – axial flow is best
- Impeller to tank diameter
- Off-bottom clearance
- Number of impellers
- Liquid level
- Baffle length – off-bottom clearance

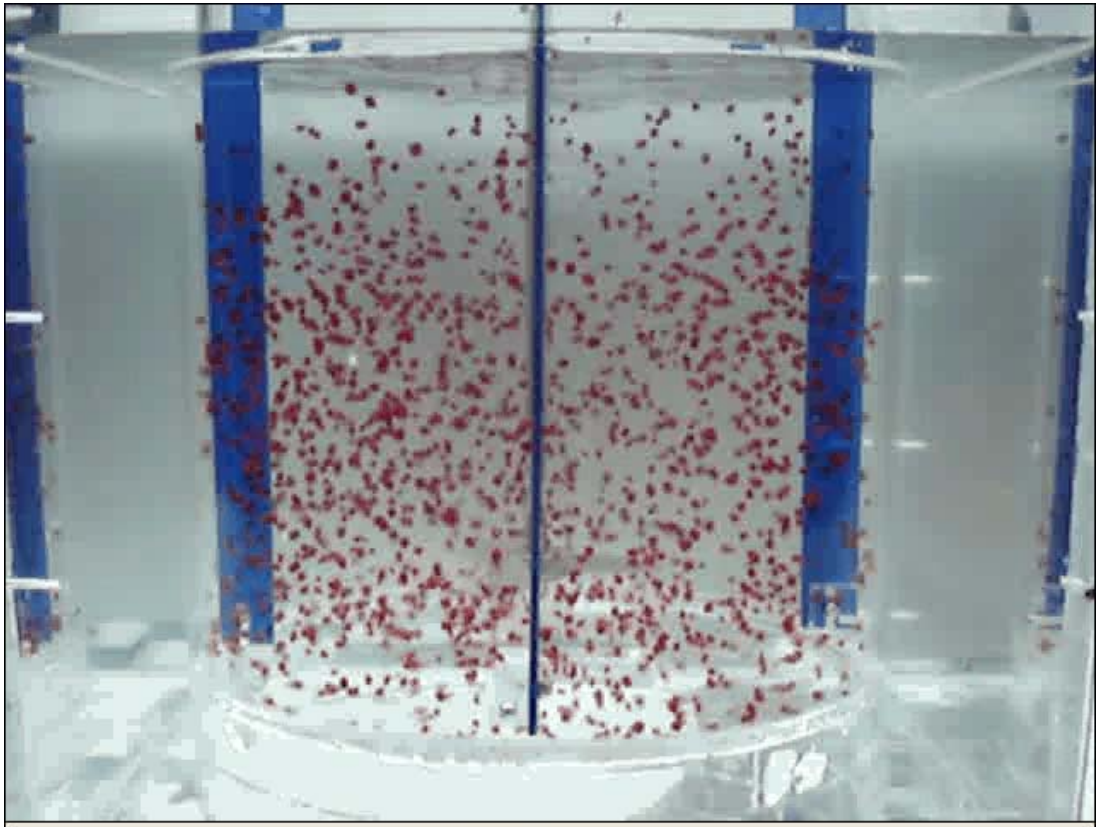
A.5.303

Pitched-Blade Turbine



Pitched 1-4.AVI

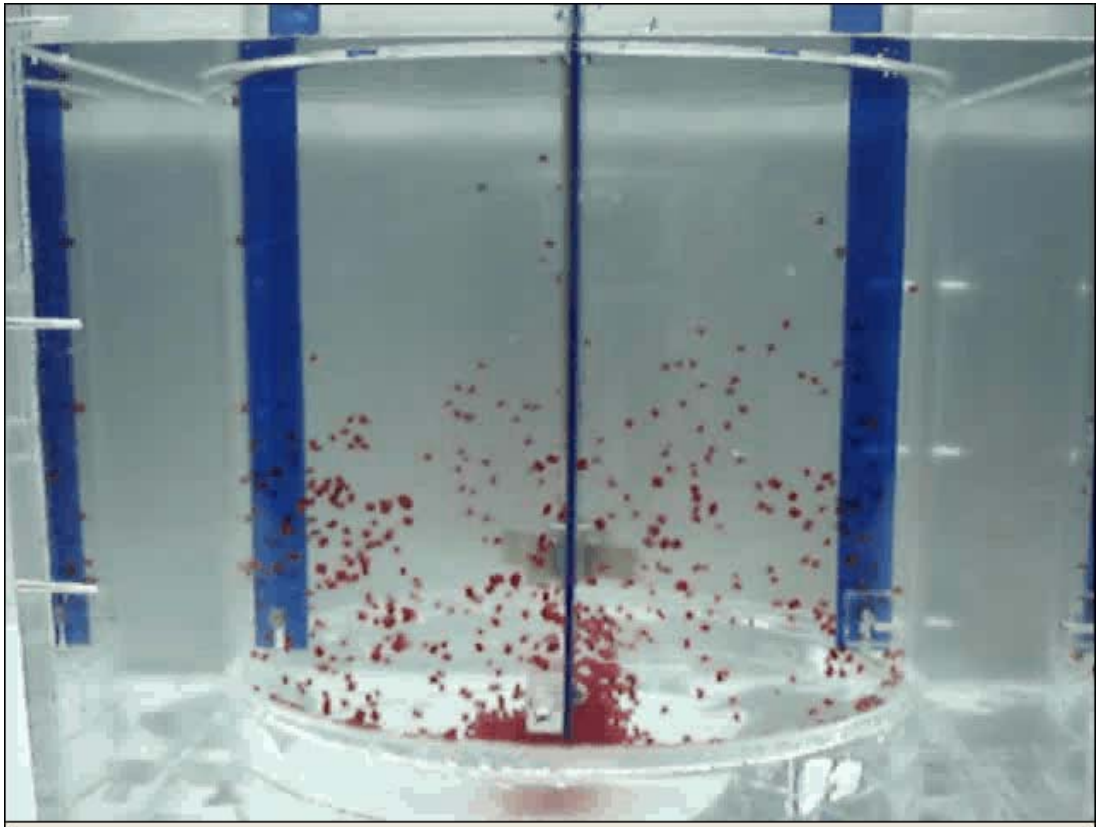
Hydrofoil Impeller



Hydrofoil 1-4.AVI

A.5.305

Straight-Blade Turbine



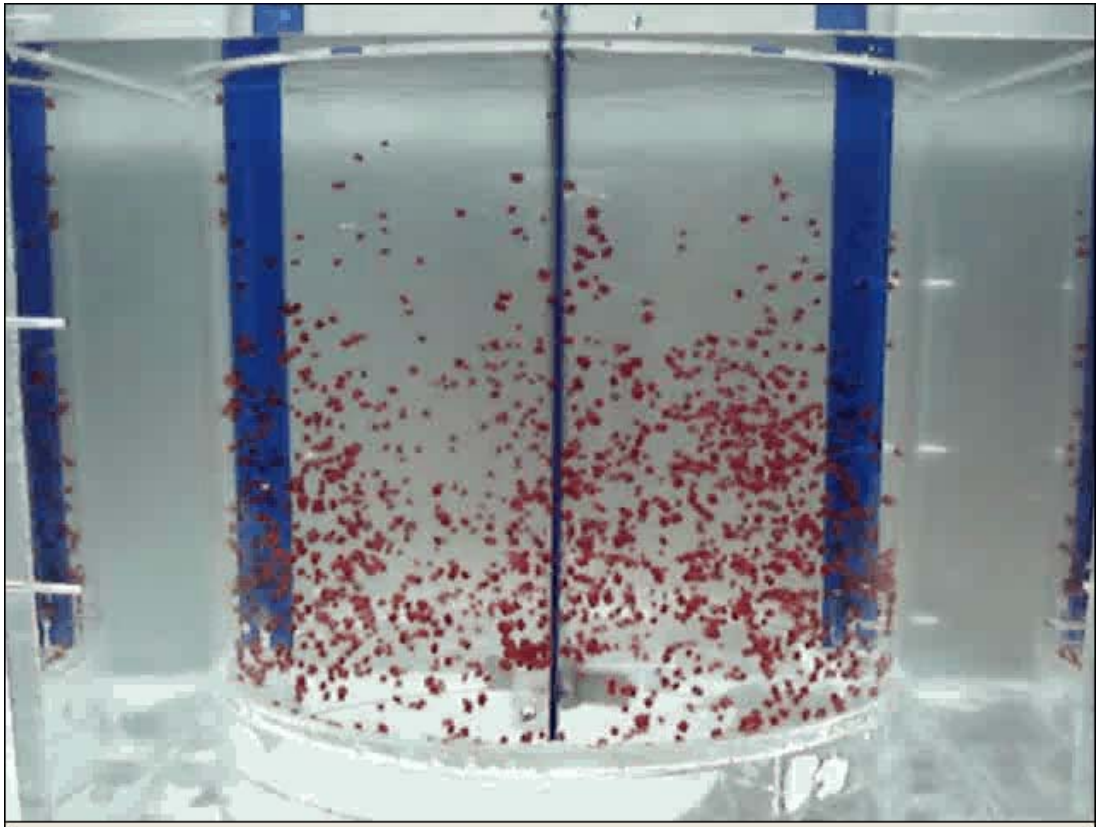
Straight 1-4.AVI

Suspension with Radial Flow

- The straight-blade radial flow impeller did not work with typical off-bottom clearance
- Close clearance does work for solids suspension
- Impeller location can be a major factor in solids suspension

A.5.307

Straight-Blade Turbine – Close Clearance



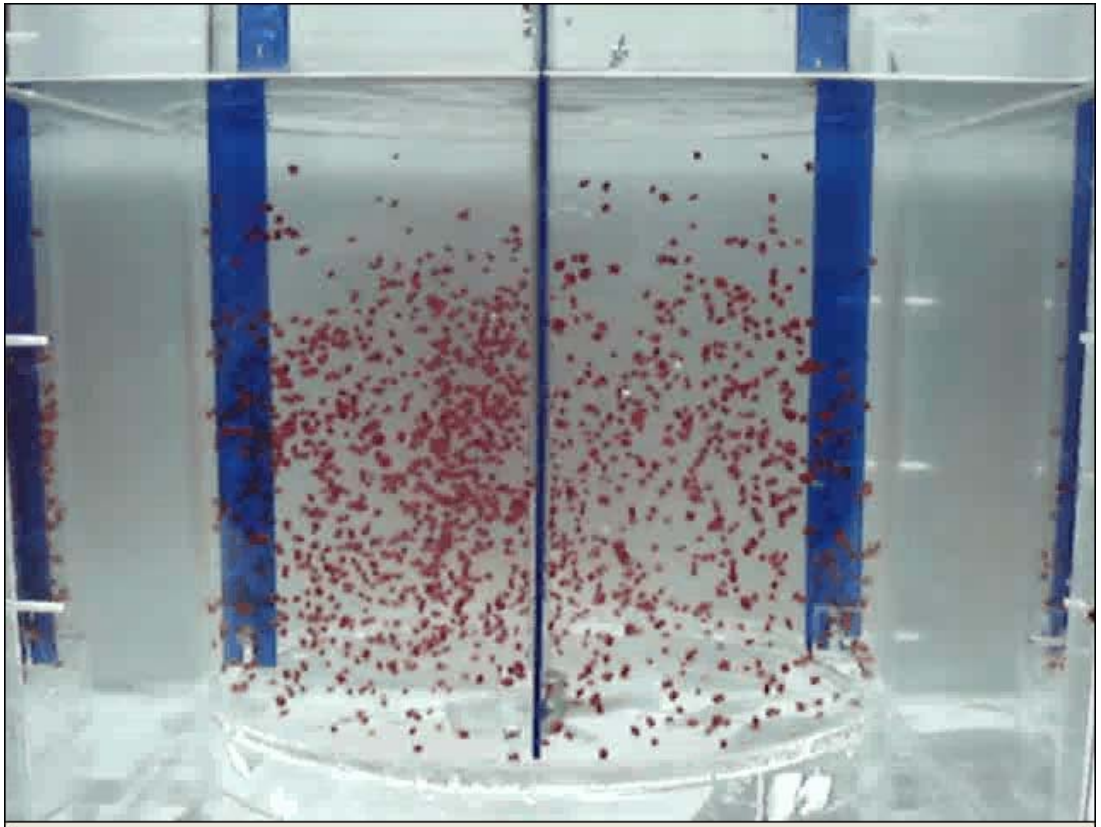
Straight Close.AVI

A.5.308

Suspension with Axial Flow

- Axial flow impellers work at close clearance
  - pitched-blade turbine
  - hydrofoil impeller
- Pitched-blade turbine requires less power than straight-blade turbine for the same results

Pitched-Blade Turbine – Close Clearance



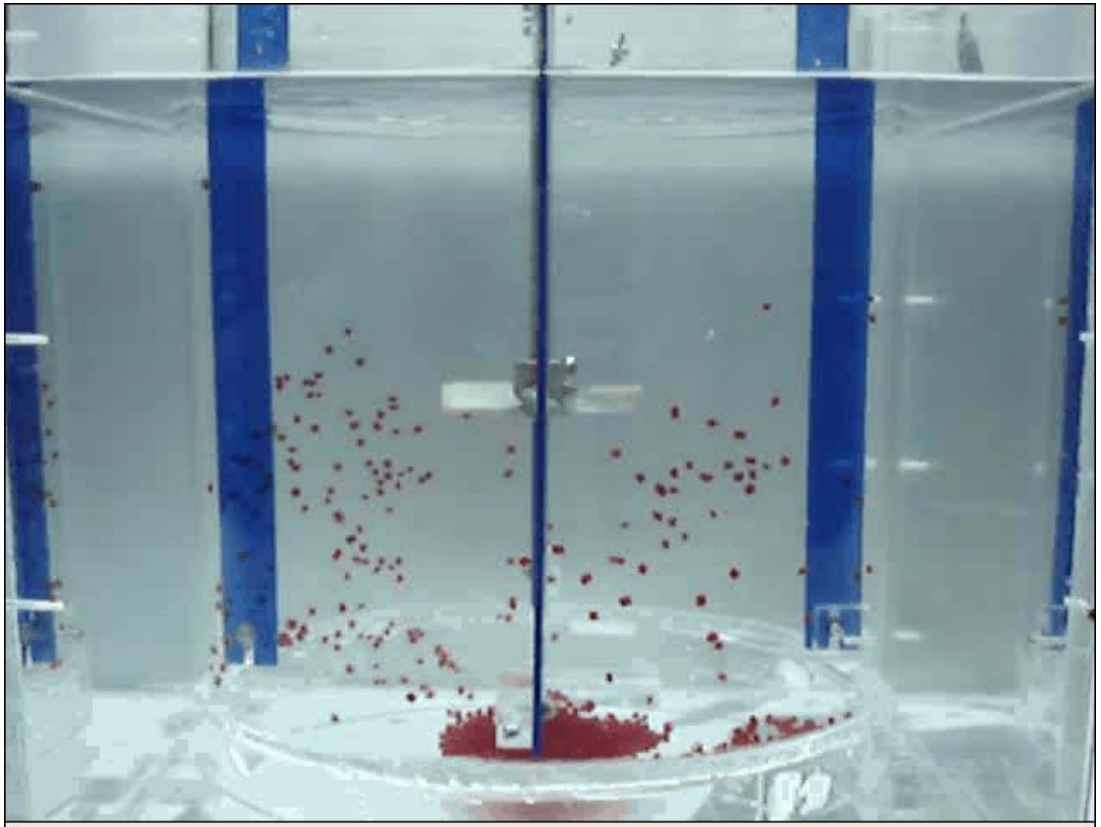
Pitched Close.AVI

A.5.310

High Clearance Causes Problems

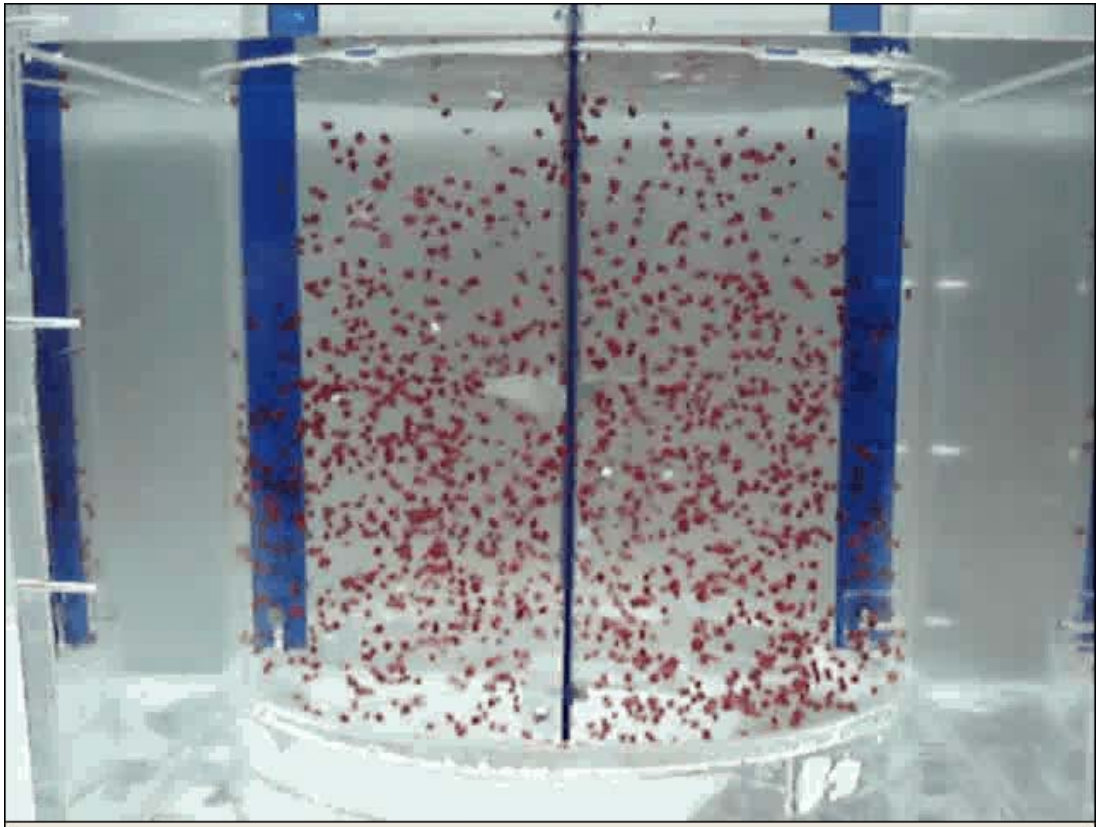
- Pitched-blade turbine fails to suspend solids when too far off-bottom
- Hydrofoil impeller works better even at high clearance position
- Different degrees of axial flow are important for solids suspension

Pitched-Blade Turbine – ½ Liquid Level Off-Bottom



Pitched 1-2.AVI

Hydrofoil Impeller – ½ Liquid Level Off-Bottom



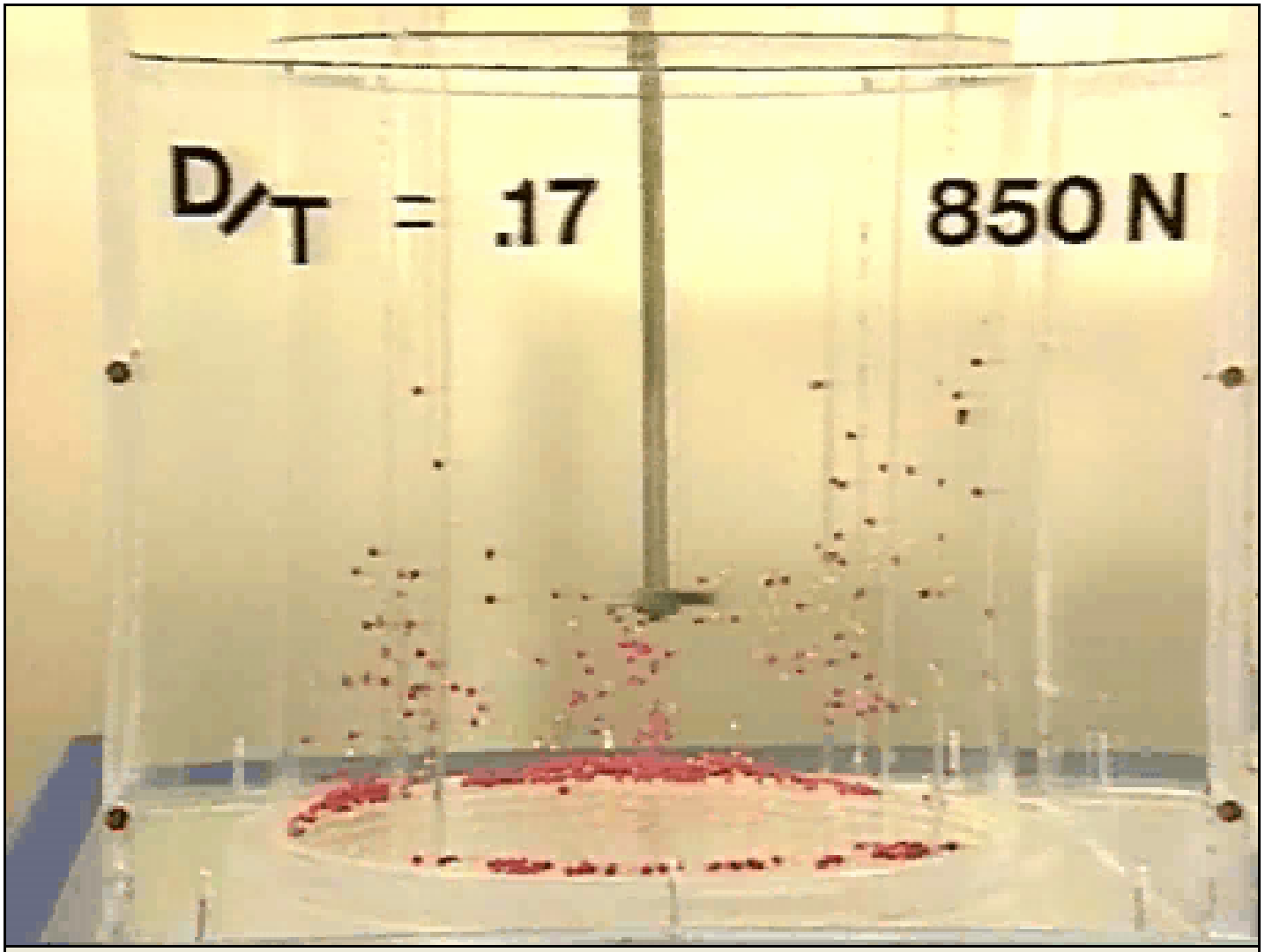
Hydrofoil 1-2.AVI

A.5.313

Impeller-to-Tank Diameter Ratio

- Small impellers require more power than larger ones
- Definite optimum impeller-to-tank diameter ratio – about 0.4
- Large impellers do not sweep the bottom

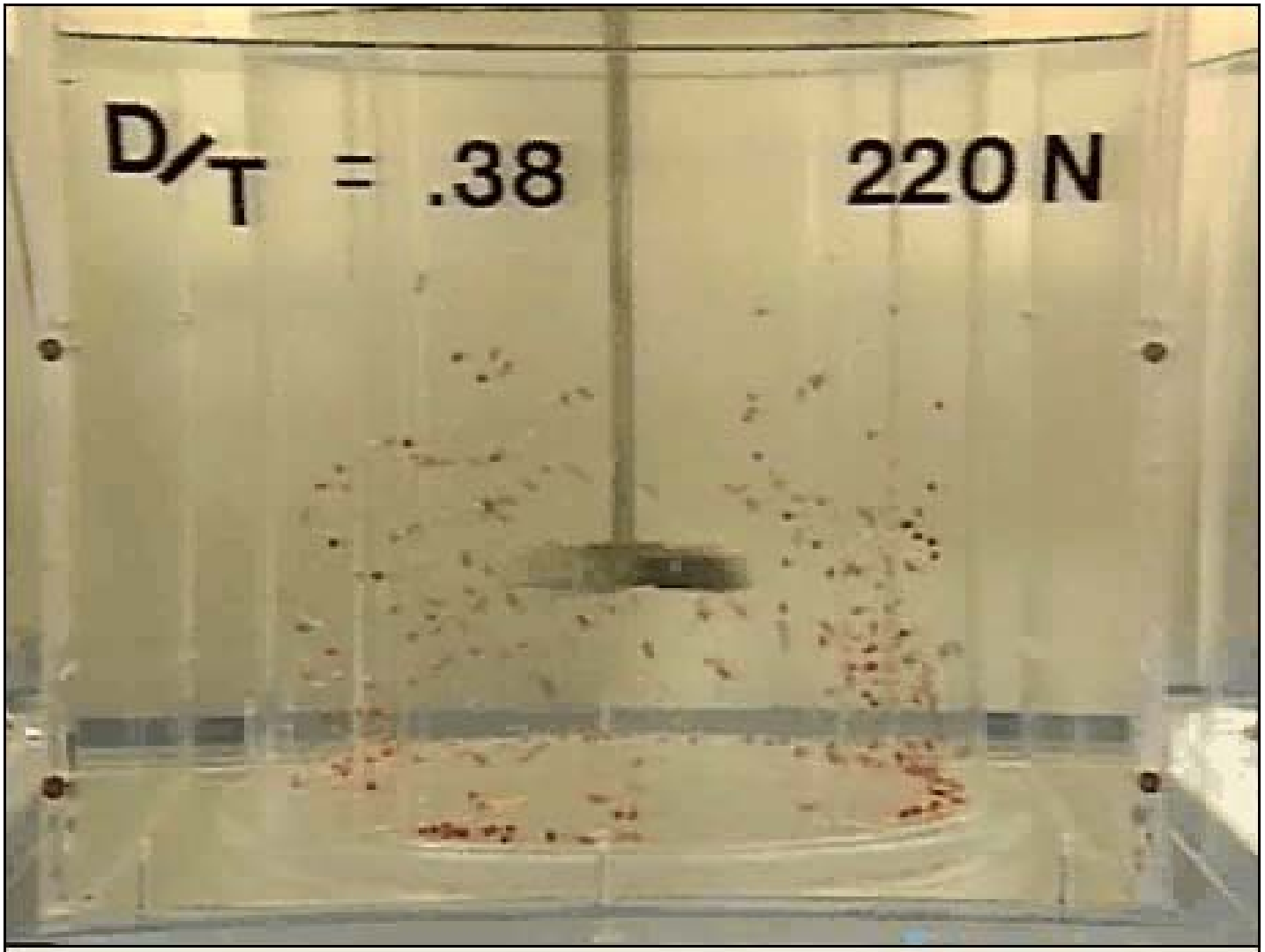
A.5.314



DT17.avi

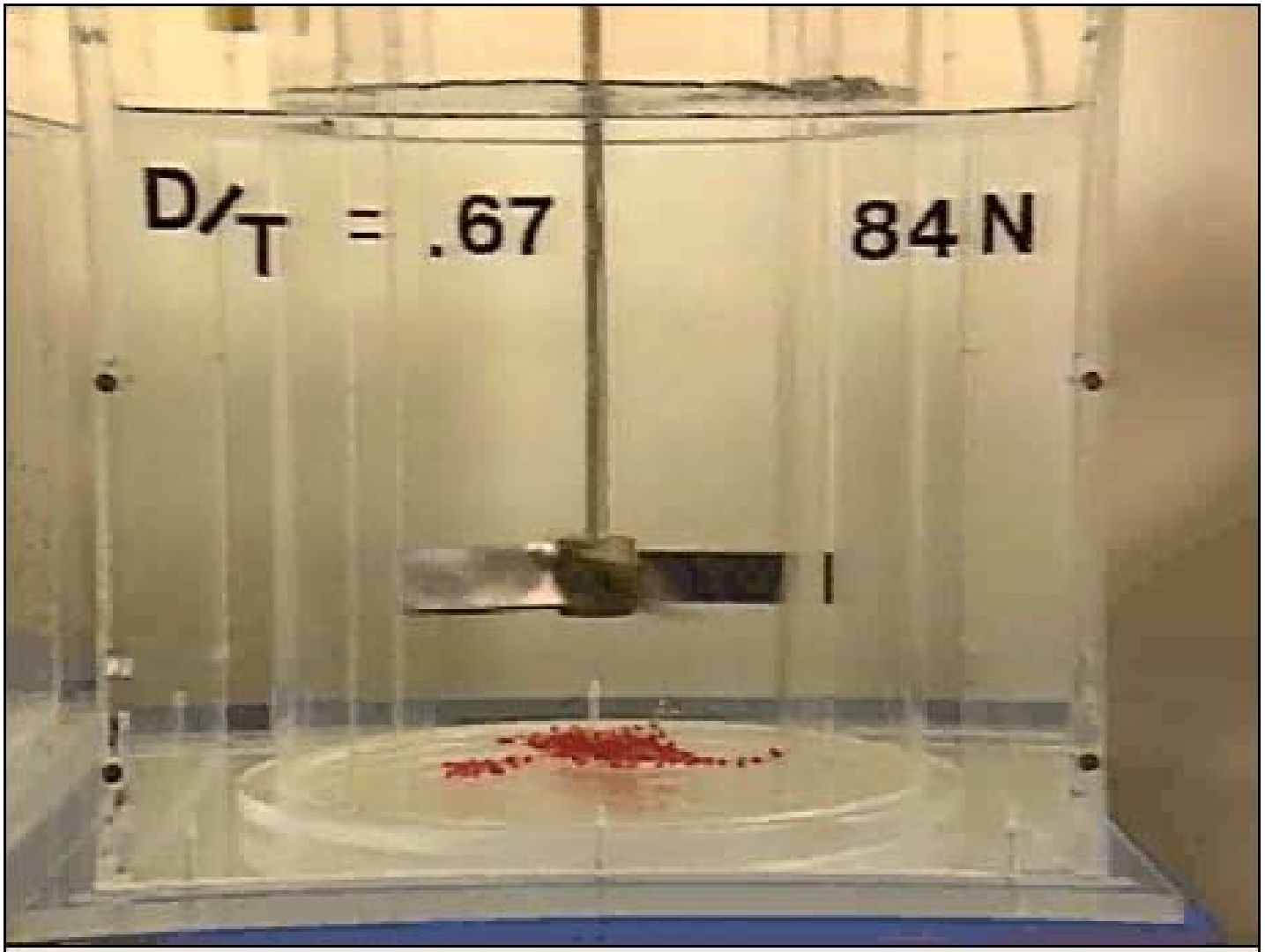
A.5.315

MixTech



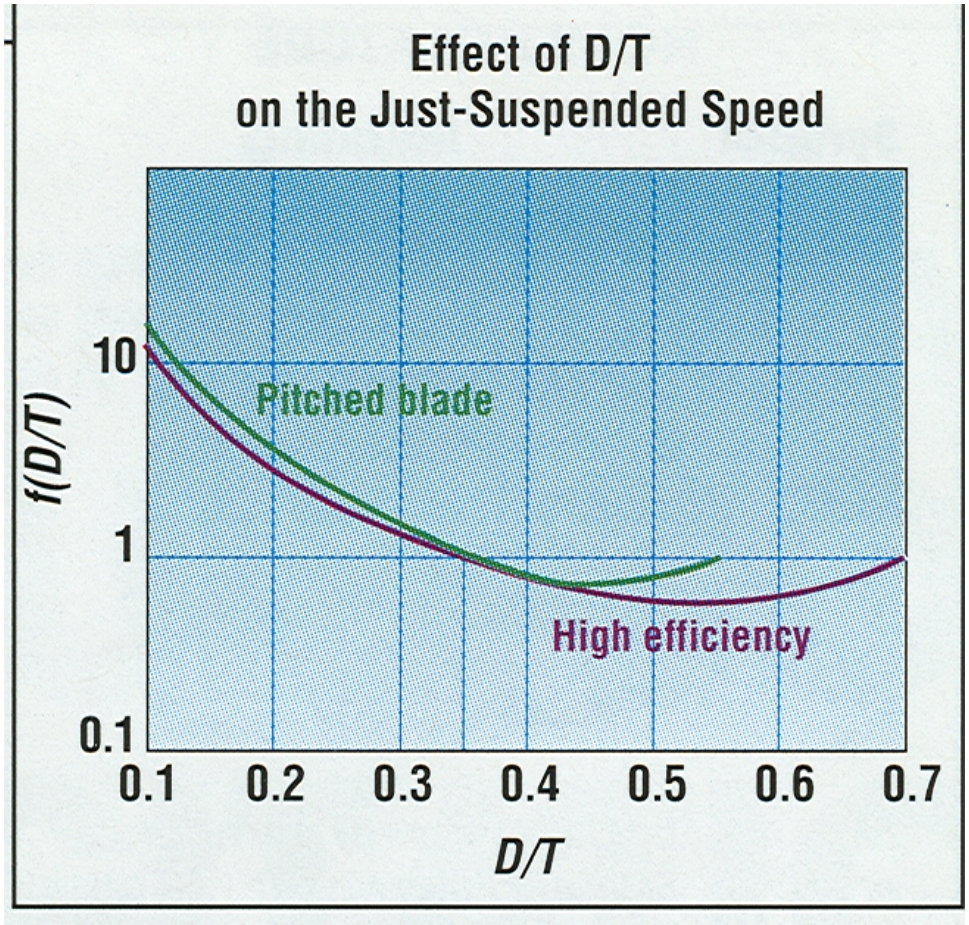
DT38.avi

A.5.316



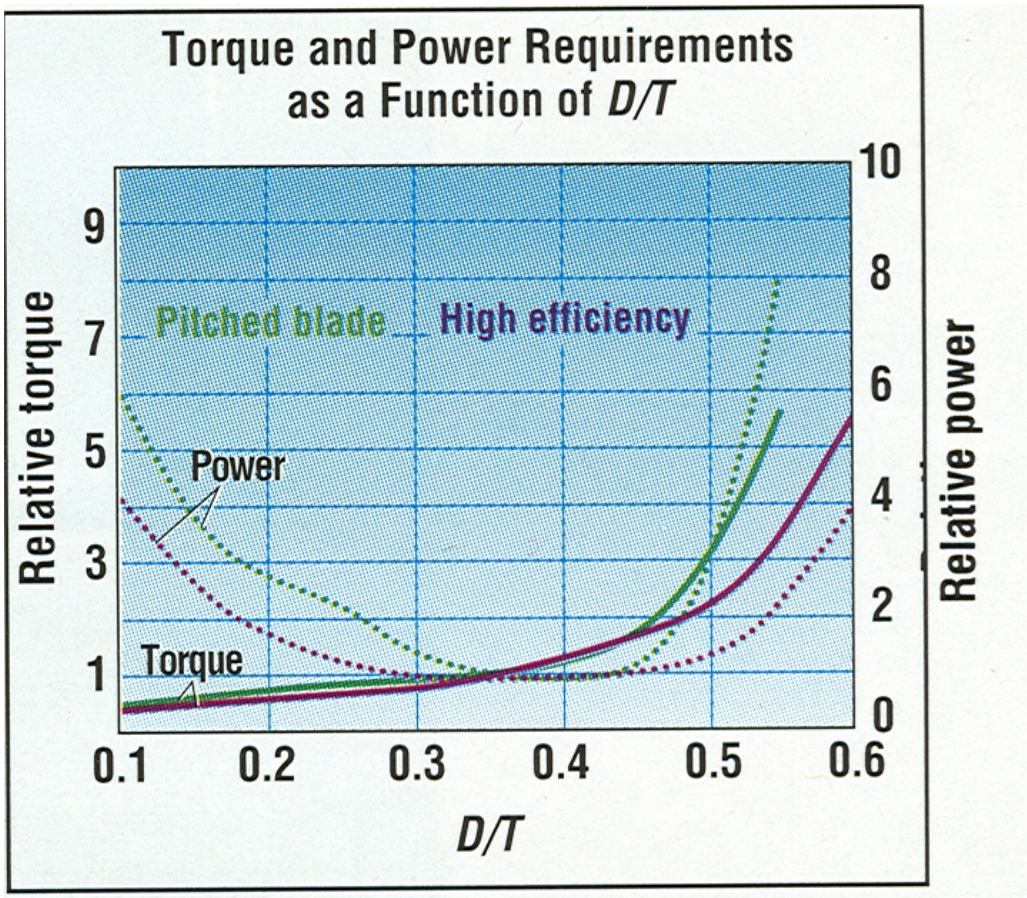
DT67.avi

Just Suspended Speed



A.5.318

Power and Torque



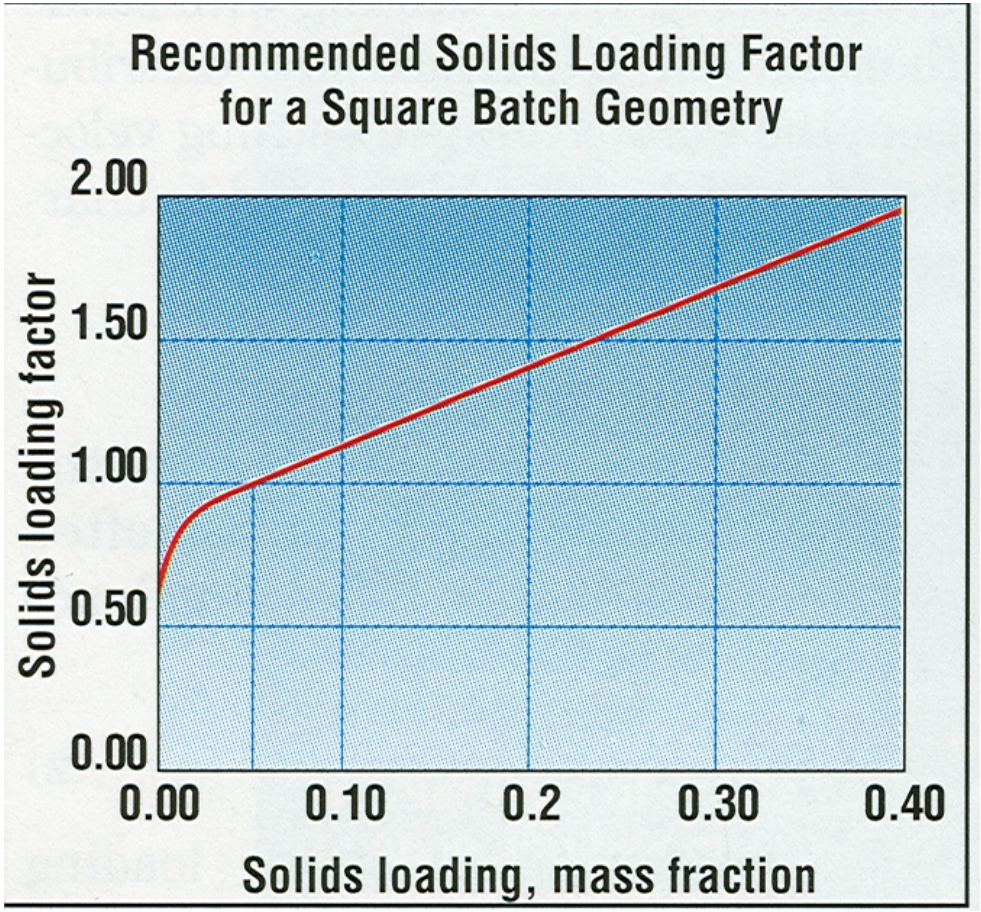
A.5.319

Solids Concentration

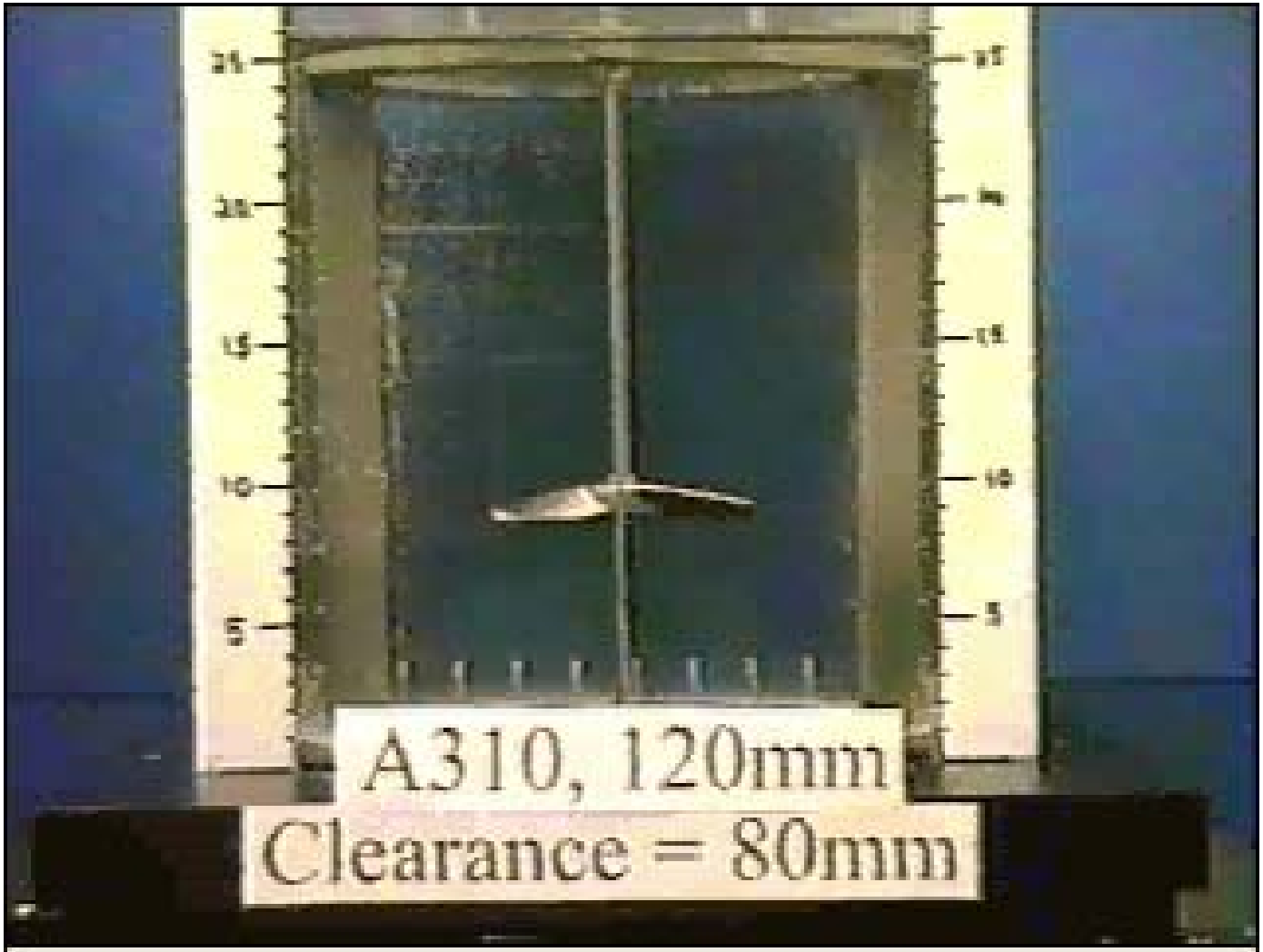
- More solids – more difficult to suspend
- Increased concentration – higher speed for same suspension

A.5.320

Effect of Concentration



A.5.321



Njs.avi

Cloud Height

- Degree of uniformity
- Rapidly settling particles – uniform size distribution
- Distinctive cloud appearance
- More agitation – higher cloud height
- Suspension is never quite uniform

A.5.323

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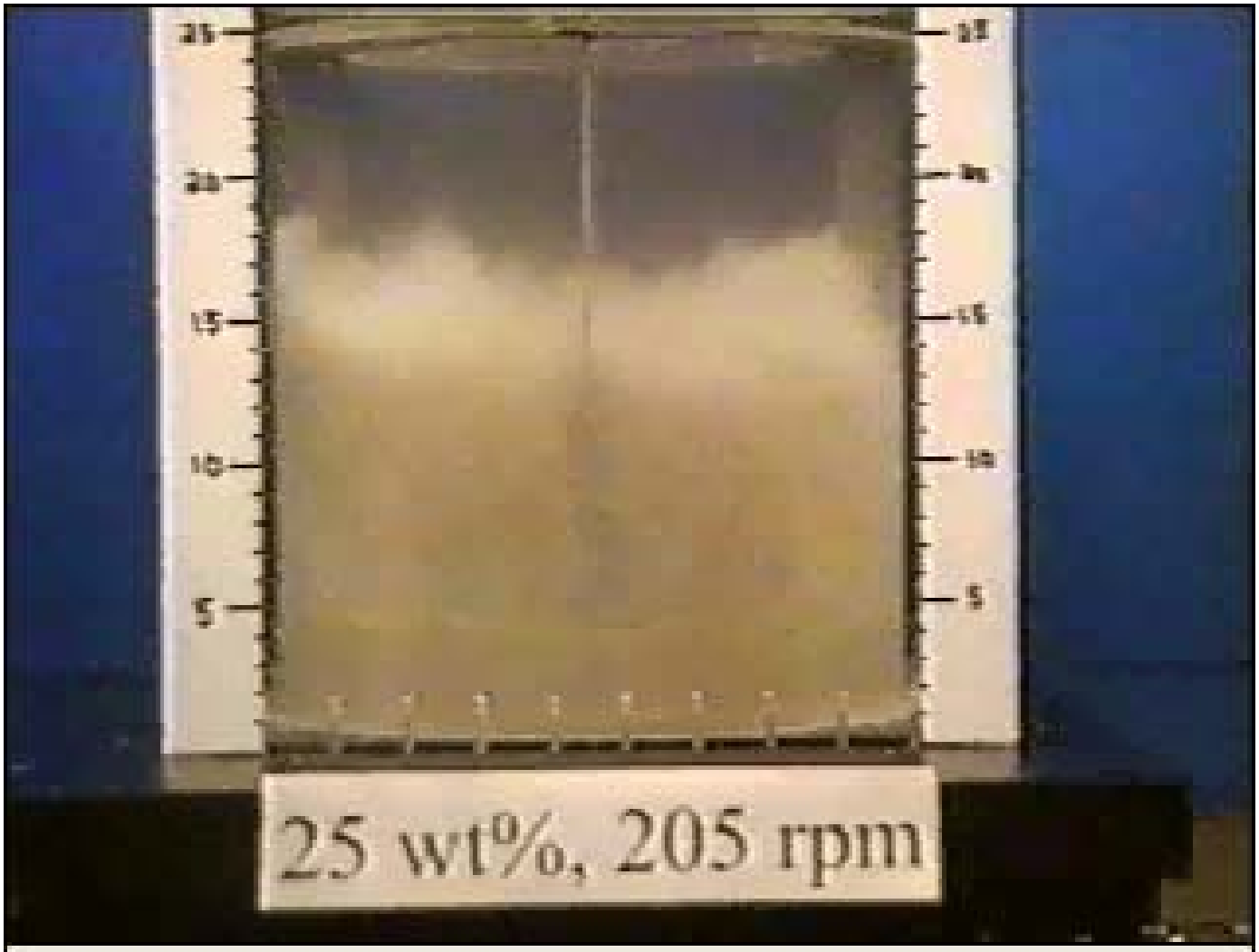


PBTSSN.avi

A.5.324

Similarity and Scale-up

- Geometric similarity – essential
- Kinematic and dynamic similarity – limited benefit
- Scale-up by empirical relationships

A.5.325

Scale-up by Geometric Similarity

- **Speed adjustment only**
- **Large scale speed – less than small scale speed**
- **Exponent maintains one constant characteristic**

$$N_2 = N_1 \left(\frac{D_1}{D_2} \right)^n$$

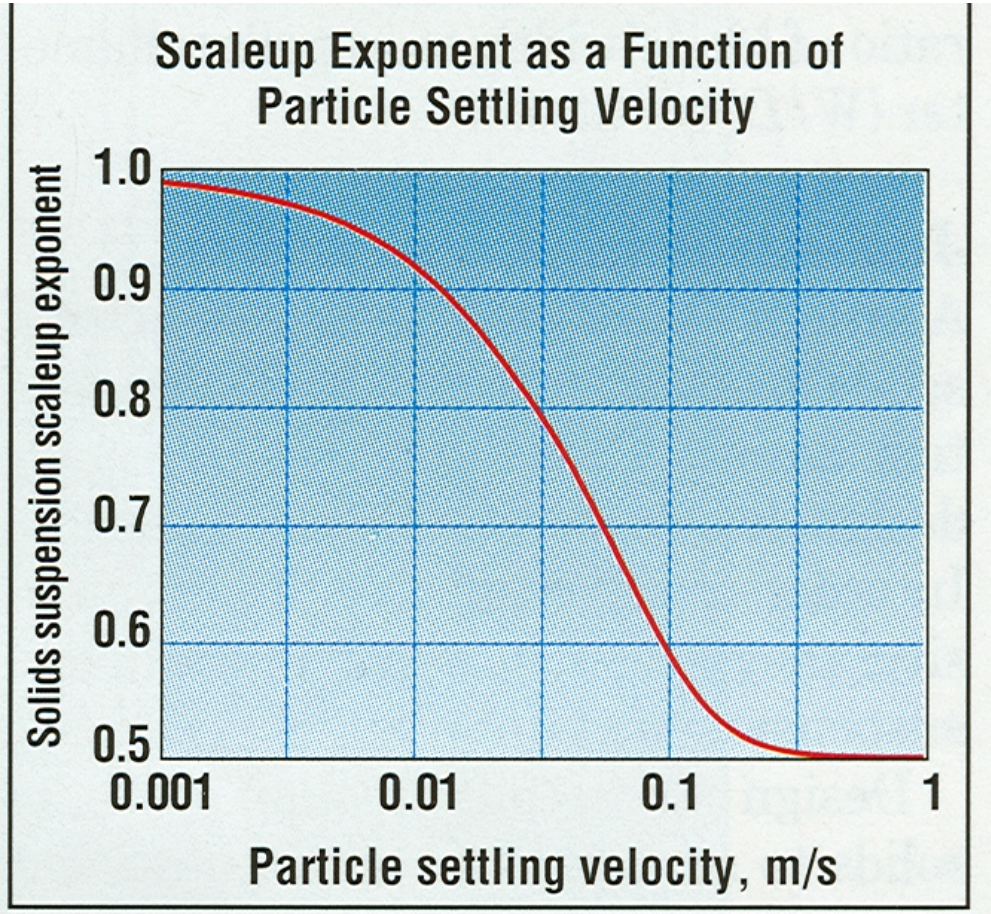
A.5.326

Scale-up Exponent

- Equal tip speed (fluid velocity)
  - n=1
- Equal power per volume
  - n=2/3
- Equal blend time
  - n=0
- Zwietering suspension
  - n=0.85

A.5.327

Scale-up Solids Suspension



A.5.328

Scale-up

- Depends on settling velocity
  - slow settling follows flow
  - rapid settling difficult
- Scale-up may also depend on size of test
- Larger tests less conservative scale-up

A.5.329

Other Factors

- Fluid viscosity
  - 10 to 20 cp more difficult
  - 100 cp easier – no settling
- Particle shape
- Size distribution

A.5.330

Conclusions

- Good understanding of solids suspension with mechanical mixers
- Understanding of steady jet mixers
- Little understanding of the effects of pulse mixing
  - cycle duration
  - refill period
- Uncertain scale-up criteria

Slurry Suspension

David S. Dickey

MixTech, Inc.

Appendix A.6

Workshop Breakout Sessions

1.0 Summary of DOE Site Discussions

1.1 Hanford

The slurry retrieval, pipeline transport and mixing workshop had a series of DOE site presentations. These presentations were designed to function as an information exchange on slurry-related topics at the various sites. A brief overview of each presentation follows, the complete presentation set can be found in Appendix A.3.

Adam Poloski presented an overview of the Hanford tank farm slurry properties. The variations in waste types from slurry generated by different plutonium recovery processes were explained. The characterization of the slurry waste types with respect to particle size distribution, solids density, and rheological properties were discussed. The presentation closed by identifying several gaps in the characterization effort for slurries of various waste types. A follow-on presentation was then discussed on how the tank slurries will be processed through the waste treatment plant. The leaching, filtration, and glass former chemical additions will result slurries with significantly different properties than entering the facility from the Hanford tank farm.

Rick Raymond presented a rheological scenario to explain a waste spill from Hanford tank S-102 during a tank transfer. A transfer pump system is installed in a tank riser from the source S-102 tank with a pump inlet gap of 0.5 to 1 inch between the tank bottom and the pump inlet. At some stage in the transfer, the pump rotation is reversed to clear the system of debris. However, the tank slurry present at the bottom of the pump possessed a rheological property called shear thickening or dilatancy. A dilatant material's viscosity will increase with the shear forces placed on it, meaning it will thicken with shear. The Hanford tank slurry was dilatant enough to form a solid plug at the pump inlet which caused a backpressure through some ancillary water dilution lines connected to the pump. One of the lines burst at a pressure of approximately 400 psi resulting in spill of tank waste.

1.2 Savannah River

Noel Chapman discussed tank closure activities at the F-Tank Farm at the Savannah River Site. Two processes in the tank farm closure project were discussed; the first involved the use of submersible mixing pumps (SMP) to mobilize the settled solids in a series of 75' diameter tanks with cooling coils. The SMP system appears to be effective in removing the bulk solids from the tank. The solids were transferred to a receipt tank and the source tank was subjected to the second process, chemical cleaning. The design of this process is not yet complete but simulant and actual waste tests have been conducted. The sequence in this process is to 1) fill a receipt tank with 50wt% caustic; 2) add approximately 100,000 gallons of 8wt% oxalic acid at 50 C to a tank with a heel height of 45 inches; 3) agitate the source tank; 4) agitate the receipt tank; 5) transfer the acidic source tank contents to the caustic receipt tank. This chemical cleaning regimen is planned for deployment in April 2008.

Michael Poirier discussed an overview of SRNL projects for the retrieval, transport, and solid/liquid separation of slurries at the SRS. On the slurry retrieval topic, SRNL has had projects supporting the design of mixer pump for bulk sludge removal. Flygt mixers and chemical cleaning systems were designed for removing tank heels. Water dissolution techniques have been

developed for saltcake dissolution. For slurry transport, SRNL supported testing programs in the 1980s. These programs include a Georgia Iron Works (GIW) pump manufacturer tests, as well as TNX flow loop testing. These tests involved performing critical velocity measurements for waste simulants as well as an assessment of pressure drop prediction equations to experimental values with simulants. For solid-liquid separation at SRS, SRNL has investigated tubular and rotary crossflow filtration, centrifugation, flocculation, and settle-decant techniques.

Jonathan Bricker presented an overview of slurry rheological and physical properties at the Defense Waste Processing Facility (DWPF). Initially a sludge batch being processed at DWPF goes through a qualification process. The qualification process involves characterization of a radioactive and simulant samples through small scale operations that mimic the DWPF. Characterization involves chemical, physical, and rheological property measurements of the process streams followed by sample glass production and glass testing. Often the rheological properties are the limiting factor and the sludge is diluted to a level to allow for processing. Efforts at SRNL have been undertaken to identify rheological modifiers to lower the rheological properties without dilution of the waste sludge. This will allow for greater plant throughput. Several dispersants have been tested with ethylene glycol and polyacrylate based dispersants being the most effective. Spheroidization of the glass frit appears as an effective technique in reducing the rheological properties of DWPF melter feed.

1.3 Idaho

Rick Demmer presented an overview of slurry processing issues at the INL. Most of the liquid waste at INL was calcined and stored as a solid granular powder in several bin sets. However, some liquid waste still exists in the Idaho Nuclear Technology and Engineering Center (INTEC) Tank Farms. Unlike situations at other sites, this waste is acidic rather than basic. Keeping the waste acidic resulted in less solids precipitation and relatively low volumes of tank sludge. Several tanks in the tank farm have been closed using a commercial tank cleaning technology that utilizes a rotating water jet called a “wash ball”. Corrosion in spent fuel pools are another major source of slurry solids at the INL.

1.4 Oak Ridge

Ben Lewis provided an overview of tank retrieval operations at ORNL. Tank sludge stored in the ORNL Gunite tanks was to be consolidated in the Melton Valley Storage Tanks. The waste was transferred over a distance of one mile in 2” schedule 40 pipe using Moyno progressive cavity or Discflo centrifugal transfer pumps. The transfers were completed in January 2001 and removed 439,000 gallons of waste. A significant number of technologies were developed to support the cleanup of the Gunite tank farm. Several of the key waste retrieval technologies developed on that project were presented and discussed.

Don Gagel presented an overview of the Melton Valley Storage Tank (MVST) sludge solidification overview. An overview of the sludge properties was provided. Cost issues for the sampling/analytical, waste packaging/transportation, and D&D project activities were presented. This was followed by a discussion of the grouted waste form properties. A follow-up presentation on an assessment of sludge mobilization technologies was discussed. Three technologies were investigated including 1) pulse fluidic mobilization (NuVision); 2) mechanical mobilization with remote manipulator and sluicing wand; 3) chemical mobilization (dissolution with concentrated nitric acid). The pros and cons of each method were discussed with the pulse fluidic mobilization

selected as the baseline technology. The pulse fluidic mobilization system was deployed in tank W-23 and the waste was retrieved with satisfactory results.

2.0 Technical Breakout Sessions

Three concurrent technical breakout sessions were conducted during the workshop. The sessions were on waste retrieval, slurry transport, and slurry processing. Each session was charged with obtaining information on best practices, lessons learned, and what worked or did not work in each of these areas. The waste retrieval breakout session is described in 1.2.1; the slurry transport session is described in 1.2.2; and the slurry processing session is described in 1.2.3.

In addition to these general sessions, there were three special sessions that were also held as part of the workshop. First, there was a special session on mobilization to overcome shear strength and was moderated by David Boger. A second session on transport related to open channel flow and critical velocity with viscosity adjustments and was moderated by Robert Cooke. A third session on slurry processing with pulse jet mixers was moderated by Art Etchells. A fourth session on computational fluid dynamics was moderated by Tom Michner. These sessions are all described in 1.2.4.

2.1 Slurry Retrieval

A workshop breakout session was held on Wednesday (1/16/08) and Thursday (1/17/08). The attendees of this breakout session are listed in the table below. The discussions were focused on DOE-EM cleanup projects at each site where slurry retrieval was used in the past or is going to be used in the future.

Table 2.1. Slurry Retrieval Breakout Session Participants

Name	Affiliation	Wed (1/16/08)	Thurs (1/17/08)
Gary Smith	PNNL	X	X
Rick Raymond	CH2M HILL	X	X
Tim Baughman	WSRC	X	X
Michael Augeri	WSRC	X	X
Jim Bellamy	CH2M HILL	X	X
Phil McGinnis	ORNL		X
Andrew Fellinger	SRNL	X	X
Marybeth Buxton	WRSC	X	X
Curt Miska	Fluor Hanford	X	X
Tim Tinsely	Nexia Solutins	X	X
Nick Vrettos	SRNL	X	X
Erich Hansen	SRNL	X	X

Table 2.1. (contd)

Name	Affiliation	Wed (1/16/08)	Thurs (1/17/08)
Steve Taylor	NuVision	X	X
David Boger	Univ. of Melbourne	X	X
Noel Chapman	WSRC	X	X
Sharon Marra	SRNL	X	X
Mike Rinker	PNNL	X	X
Jeff Lindner	Miss. State. Univ.	X	X
Steve Krahn	DOE EM	X	-

Much of the discussion during the first part of Day 1 was defining the question, "What is success?" The group thought it was important to discuss this as we work towards lessons learned in areas that were both successful and unsuccessful. The main criteria for success, in Retrieval, was defined as regulatory agreement to close a tank or tanks. Another criteria of retrieval success is the ability to regain use of the tank after it has been retrieved. An example of this was Tank 48 at Savannah River. While safety is always paramount in all of our activities, it was concluded that we need to state that success is also the ability to operate safely in the tanks farms with no harm to the workers or the environment during those operations. Finally, ensuring that we do not create "new" waste streams or issues downstream during our operations is also success.

The group also discussed important technology/attributes that need to be considered in all retrieval operations. Hanford wrote into the 2000-2001 federal facility agreement that the Site was going to perform "hot demonstrations" of new technologies in order to clean out tanks as well as to determine whether new technologies were capable of meeting retrieval needs at the site. Additionally, it was stated that one technology does not fit all cases. There are different waste properties in different tanks. In many cases, the physical and chemical properties are not that well understood prior to retrieval operations. Another important attribute that was discussed was that there is a need to understand the properties of the materials being retrieved from the tanks.

The retrieval discussions were centered on various functional aspects of retrieval including bulk mixing, heel retrieval, transfer, and characterization & monitoring.

Mixing – At Savannah River, bulk mixing is achieved by the use of slurry pumps including the standard Lawrence pump, the quad volute pump, the advanced design mixer pump, and the submersible mixer pump. In general, all worked for the applications used, but some modifications were necessary. At Hanford, the use of standard mixer pumps is the baseline, and the only recent activities for mixer pumps were the SY-101 mixing several years ago. West valley also used multiple mixer pumps in their tanks in order to remove the heel after bulk mixing was completed. This required large volumes of water to be used for mixing. In the UK, pulse jet mixers have been used for their waste streams for the past twenty years. At Oak Ridge, they also used pulsed jet mixers in their horizontal (W and C tanks) as well as the capacity increase tanks.

Russian style mixers were also deployed in the Gunnite tanks at Oak Ridge as a demonstration of other technologies.

Sluicing – Sluicing has been a method of mixing and retrieval through many deployments at Savannah River and Hanford over the years. It was used extensively at SRS during the 1970's and 1980's. At Hanford, twenty to thirty single shell tanks have been sluiced over the years. Most recently, there were several campaigns to sluice tank C-106 in order to remove the waste and the heel. Oak Ridge used sluicing in some of their tanks. Fernald used sluicing to remove the waste from their silos within the past ten years.

Agitator Based Mixers – These types of mixers have been used at SRS recently during extensive testing as well as removal of the zeolite heel in Tank 19. Oak Ridge also used one of these mixers as well. The results of the use of these mixers varied, but in general it was not as effective as mixer pumps.

Air sparging was used at Hanford via air lift circulators for agitation but not retrieval. The UK used spargers for waste homogenization. Oak Ridge deployed a variant of air sparging with the use of pulsed air mixers. In all cases this technology is not viewed as a strong candidate for waste retrieval.

Heel / Residual Material Removal – There are a lot of methods that have been tested, demonstrated, or used in the past with moderate degrees of success. At Oak Ridge, they utilized both a robotic arm and a remote crawler in tandem to remove the waste from the Gunite tanks at Oak Ridge. This was highly successful, although it would likely be prohibitively costly to use at other sites. Currently at Hanford, they are using a simple articulated arm with a vacuum retrieval system. They are also utilizing a crawler with limited success. The Salt Mantis [ref.?] has been deployed in one tank at Hanford and SRS in considering their use in Tanks 18 and 19. At SRS a wall crawler deployed in the annulus is being developed to remove some of the salt nodules. In the UK, an arm based and crawler based system was deployed in a tank and in a waste basin. A common method for heel retrieval that has been deployed at several sites is the use of high pressure water nozzles and water lances. High pressure systems were utilized at K-Basin at Hanford. The borehole miner was used at Oak Ridge to remove the heels from the Old Hydrofracture Facility horizontal tanks. Another unique method for heel removal includes the use of ultrasonic technology at the UK, although it was not clear whether it has been deployed. Chemical cleaning has been used at SRS successfully in Tank 16, and more chemical cleaning deployments are planned. The Russians have also utilized chemicals for cleaning their tanks as well. They have provided technical information to SRS regarding their chemical cleaning processes. At Hanford, they utilized high molarity caustic for heel removal, and at Idaho, they used acid cleaning in their stainless steel waste tanks. Spray washing has been a popular technique for getting spots of waste off of floors and sidewalls of tanks in the UK. At Oak Ridge, they utilized a gunnite scarifying end effector to remove contaminants from the tanks. At Idaho, they used a sprayball system to get around the coils inside their tanks.

Transfer Out of Tanks – Most waste transfer out of the tanks are accomplished with pumps. There is significant pump experience at each site. Generally, these are commercially available pumps, although some radiation pumps were deployed at SRS. Typically, pumps that have been used include rotodynamic (centrifugal and turbine) pumps, positive displacement pumps, fluidic

pumps, and pneumatic pumps. There was some discussion on the development of mechanical methods, like augers and clam shells, but those were not viewed as acceptable methods of transfer.

Characterization & Monitoring – One of the primary methods of monitoring is the use of camera systems including still cameras and video cameras. All agree that these are essential to retrieval operations; all agreed that all cameras are problematic. While some radiation hardened cameras have been developed, they are expensive, and it may be more cost effective to use commercial systems and replace them on a regular basis. Volumetric measurement, or mapping, is a very important aspect of retrieval. It helps the sites to determine how effective their retrieval operations have been. There are many volumetric mapping techniques including tomography, volume displacement, mass balance, laser range finders, structured light, profilometry, sludge sounding, visual inspection based upon known items in the tanks and radar systems.

At Hanford the most useful means of measuring waste is through volumetric measurements as opposed to mass balance. The use of photos is extremely important in working with the regulators. Hanford is just starting to use gamma cameras, and they are currently using video scaling techniques as well. At SRS, the volume and radionuclide content of the tanks is tied to the performance assessment. They utilize visual techniques with landmarks such as cooling coils and plates on the floor. Oak Ridge and Fernald also utilized landmarks to monitor remaining tank waste volume.

Other characterization tools being used are the Turbidity Meter, which is in process at SRS (supernate vs slurry transfer), a suspended solids profiler at Hanford that did not work. Various densitometers based upon ultrasound or coreolis effects are being used at Hanford. Fernald used microwave technologies and gamma attenuation for radon control. SRS does not have an in-tank characterization and monitoring system.

Radiation Monitoring at Hanford include a prototypical gamma assay system deployed to evaluate mixing, although there were problems with this was deployed during the AZ-101 mixing tests. There are two in-line probes on a transfer line at Hanford for real time cesium estimates. There was a gamma scan on the tank walls for salt at SRS.

Physical Samples (both in-situ & grab samples) At Hanford, there are no *in situ* samples. All samples are removed from the tanks, and they have used many devices including augers, clam shells, the “alligator”, the “aardvark”, dip samplers for supernatant, core samples prior to retrieval, and Hanford has a Raman probe that has not been deployed yet. At SRS, in Tank 5 they utilized a core type sample. In Tanks 18/19 they deployed a clam shell type sampler on their crawler. Dip samples are common including 3, 5, and 25 liter samplers, but these are really for processing and not retrieval. SRS is also working on a Raman probe like Hanford.

2.2 Slurry Retrieval Site Information Input

2.2.1 West Valley Site Input

2.2.1.1 Name of the technology

Retrieval technologies deployed at West Valley include the mast-mounted tool delivery system, long-shaft vertical mixing pumps, and transfer pumps.

2.2.1.2 Description of technology

Mast-mounted tool delivery system: The mast-mounted tool delivery system is a remotely operated mast with tools mounted on carriages that can be raised and lowered along the mast. Tools include an arm-mounted sluicer, arm-mounted wall sampler, lights, and cameras. The system is comprised of a 14.9 m steel beam, deployed through a 65 cm riser that extends to within 30 cm of the tank bottom. The top of the beam extends out of the riser and is mounted to a rotary bearing connected to an electric gear motor. A series of eight hydraulic winches and actuators are mounted to the mast, above the rotary bearing. Each winch can lift a maximum of 2200 kg [5].

Transfer pump: The transfer pump is a 13-stage, 12-m slurry transfer pump. It has a radial inlet suction that extends approximately 7 to 9 cm above the tank bottom. Two concentric strainers prevent large debris from entering the pump. It has a 14.9 kW motor located in a concrete-shielded pump pit directly over the pump column. The pump has the capacity to pump 380 L/min with a 60-m head.

Long-shaft vertical mixer pumps: The 15.3 m long centrifugal pump powered by a 110 kW motor has one impeller that draws material into the pump suction. The pump suction is fitted with a strainer to prevent large debris from entering the pump. The suction is positioned 2.5 to 10 cm above the bottom of the tank. Two tangential, 3.8 cm diameter nozzles discharge the pumped waste about 18 to 25 cm above the bottom of the tank. Each nozzle distributes 2 270 L/min at the 100%-rated pump speed of 1 800 rpm.

2.2.1.3 Deployment Sites

Tank 8D-1 was considered a “spare” tank at the site. It stored 144,000 lb of spent zeolite under an alkaline liquid. Ion exchange columns located in the top of the tank were used to strip cesium and strontium from liquid wastes before evaporation or grouting. When the columns were saturated, the zeolite was dumped into Tank 8D-1. In addition, Tank 8D-1 contained 137,000 gal of excess liquid from pretreatment and zeolite transfer operations. The liquid had a pH of approximately 10.5.

Tank 8D-2 contained a mixture of washed plutonium-uranium extraction sludge solids, zeolite, and supernate. The sludge included the following chemical constituents: iron oxide (35.5 wt %), silicon dioxide (20.8 wt %), sodium oxide (14.4 wt %), thorium oxide (10.4 wt

%), and aluminum oxide (7.1 wt %). The sludge was estimated at 220,500 lb, with a specific gravity of 3.35. The primary radionuclides in the sludge were strontium-90 and thorium and uranium isotopes.

2.2.1.4 How well the technology worked

Most of the waste was removed from the two tanks. In Tank 8D-1, approximately 96% of the caesium-137, strontium-90, and sludge were removed. In Tank 8D-2, greater than 99% of the long-lived radioactivity was removed, only a few small areas of settled cesium-137-laden zeolite remain.

2.2.1.5 Evaluation criteria used to determine success

The volume removed from the tank and the radionuclides removed were the primary criteria for evaluation of success.

2.2.1.6 Lessons learned

A mobilization pump trial failed. The impeller key sheared. Pump designs were then modified so that the easily accessible motor coupling key would fail before the inaccessible coupling key was sheared.

Mobilization pump suctions lowered from 10 cm above the tank bottom to 4 cm provided additional clearance between the jet centerlines and the tank structural grid work. This improved the effective solids mobilization radius, and it allowed for the pumps to be operated at lower tank levels.

Installing transfer pump motors, a pump tachometer, and valve position switches inside the pump pits proved easy and cost-effective.

Positioning equipment outside the pump pit (so it can be easily serviced) eliminated the need for personnel to enter a highly contaminated area, and it kept the equipment cleaner.

2.2.1.7 Key documentation that is available which provides technical information regarding the technology

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PACIFIC NORTHWEST NATIONAL LABORATORY (PNNL), Tanks Technology Guide (TTG), Mast Tool Delivery System (MTDS), Richland, WA (16 July 2001).

U.S. DEPARTMENT OF ENERGY, Ohio Field Office (DOE, OFO), High-Level Waste (HLW) Tank Closure, US Department of Energy, Ohio Field Office (30 July 2001).

2.2.2 Oak Ridge Site Input

2.2.2.1 Technology: Heel Retrieval/Residual Material Removal – Robotics (Arm/Crawler)

Author

B.E. Lewis, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Description of Technology

The primary robotic equipment used in the Gunite and Associated Tanks (GAATs) remediation at Oak Ridge was the Modified Light Duty Utility Arm (MLDUA) and Houdini I and II remotely operated vehicles (ROVs).

MLDUA – The MLDUA provided reach and mobility during waste retrieval operations in the gunite tanks. The MLDUA was an 8-DOF robotic arm with a reach of 15 ft and a payload capacity of ~200 lb (Figure 1). The end of the MLDUA contained a tool plate that could accommodate a Gripper End-Effector (GEE) that was used to grasp and hold the various tools used to modify the interiors of the tanks and remove the liquid and sludge waste from the tanks. The GEE extended the MLDUA reach to 16 ft.

Cameras mounted on the wrist and mast of the MLDUA provided a remote video feed to monitors positioned in the MLDUA's control console. The cameras helped operators grasp tools and end-effectors and to monitor the waste retrieval operations. The robotic arm could be programmed to perform specific operations or could be operated remotely from the control room. The robotic control functions were particularly useful when operating in heavy mists and other low-visibility conditions.

Figure 2.1. MLDUA and Hose Management Arm Inside Gunite Tank W-7

The MLDUA's containment structure was mounted on the equipment platform above the

tank riser. This structure housed the MLDUA when it was moved and helped prevent the spread of contamination. A tank riser interface containment (TRIC) system was located on the work platform between the riser top and MLDUA storage structure for the Vertical Positioning Mast (VPM). Glove ports in the TRIC provided operators with access to the equipment for maintenance and repair activities and for attachment of end-effectors such as the gripper and sampling tools. A decontamination spray ring (DSR) was mounted on the bottom of the TRIC for decontamination of equipment as it was retracted from the tank. A spray wand mounted inside the TRIC was used to further decontaminate the MLDUA.

Figure 2.2. The Houdini I ROV As It Was Deployed Through a 24-in.-Diam Riser During the Cold Testing

Houdini ROV – The Houdini I and Houdini II ROVs were tethered vehicles that could perform a wide variety of operations. The ROV frame had the ability to fold up and fit through 24-in.-diam tank access risers (Figure 2). The tracked vehicles had a parallelogram-shaped frame that could be expanded to a ~4- × 5-ft work platform. Each vehicle weighed ~1000 lb and was equipped with a plow blade; a dexterous, high-payload manipulator; and four remote camera systems.

The versatility and mobility of the Houdini I and Houdini II ROVs allowed operators to remotely perform many types of in-tank operations. Each Houdini vehicle was equipped with an integrated manipulator arm. The 6-DOF arm had a payload capacity of 250 lb at full extension. The arm was used to pick up and organize debris so that it could be retrieved from the tanks and to deploy a variety of tools and end-effectors, which were used to modify the interiors of the tanks, sample the tank waste, and retrieve the waste. The ROVs were also equipped with a plow blade on the front of the frame. The plow blade was used for breaking up sludge heels at the junction of the tank wall and floor and to push sludge towards the confined sluicing end-effector as it was held by the MLDUA to accelerate waste retrieval operations. Cameras mounted on the arm and rear panel of the vehicle near the tracks provided a video feed to monitors mounted at the vehicle's control console. Operators could adjust the camera views to help them grasp tools and to perform intricate operations.

The Houdini ROVs were housed in containment structures that were positioned on the equipment platform above a tank access riser. The containment structure included glove ports, which provided operators with vehicle access during maintenance or repair activities.

The tether for the ROVs was comprised of hydraulic pressure and return hoses; a water line for camera cleaning; and electrical conductors for the on-board cameras, manipulator, track servo valves, limit switches, and pressure switches. The tether was rated at 10,000 lb_f breaking strength, which allowed it to be used as a structural member during insertion and removal from the tanks. The combination of the tether reel and vehicle containment structure was called the tether management and deployment system (TMADS), which also served as the interface with the tank riser. The TMADS provided the vehicle with a sealed compartment in which to store the vehicle; a hydraulically powered, remotely operated tether reel; and included glove ports, a spray wand, and access features for maintenance and decontamination operations.[1]

A skid-mounted Power Distribution and Control Unit (PDCU) was used to convert and condition the site electrical power to the proper voltages for use with the Houdini ROV. The PDCU also contained the computer control hardware and hydraulic power supply for the ROV.

In addition to the robotic equipment, numerous support systems were used to facilitate waste retrieval operations and supplement the capabilities of the robotic equipment. Many of these systems have been previously mentioned; however, one system that was especially important to the successful operation of the MLDUA and ROVs was the Hose Management System (HMS).

HMS – The HMS was designed to minimize the load on the MLDUA and the Houdini ROV by providing a positioning system for the CSEE umbilical, especially the heavy conveyance line. The system also minimized the radiation exposure to the MLDUA by separating the waste discharge line from the MLDUA during waste retrieval operations. The HMS was comprised of a Hose Management Arm (HMA), storage tube, confinement box, and Mast Elevation Table (MET). The HMS provided 4 DOF for deployment and positioning of the waste retrieval tools such as the confined sluicing end-effector (CSEE) and management of cables and hoses.[2] It delivered power and process water to the CSEE and incorporated the conveyance system that was used to transfer waste out of the tank.

The base link of the HMA was a heavy vertical mast that could be rotated and vertically positioned by the MET above the tank riser. The mast could be retracted into the storage tube above the MET using an integral hoist. Two rigid pipe intermediate links extend from a deployment position (folded up against the mast) to a horizontal working position with motorized swivel joints. The distal link to the CSEE is a short umbilical hose and cable bundle. The CSEE power cables and water supply hose are routed along the rigid links and up conduits in the mast to jumper connections to the Balance of Plant at the platform. The conveyance hose connects to the rigid pipe links, which double as structural sections and conveyance conduit. The conveyance conduit continues inside the mast to the jet pump and up to the above-grade platform.

The HMA was retracted from the tank through a DSR and into its containment structure located on the tank platform. Eight glove ports on the HMA containment structure provided access for maintenance operations and to electrical power controls and hose connections. The HMA containment structure housed the jet pump and was also used to isolate the system from workers and the environment. The HMA could be retracted and secured in its containment structure and then moved to the next tank scheduled for remediation.

Deployment Site(s)

Gunite and Associated Tanks (GAATs) at Oak Ridge National Laboratory

Performance

After the bulk waste retrieval operations in the GAATs in the early 1980s, final cleanout of the tanks was conducted during a Remediation Project, which was the first of its kind performed in the United States. Robotics and remotely operated equipment were used to successfully transfer over 439,000 gal of radioactive waste slurry from nine large underground storage tanks. Almost 94,000 gal of remote-handled transuranic sludge and over 81,000 Ci of radioactive contamination were safely removed from the tanks, which were located in a high-traffic area of ORNL near a main thoroughfare.

A phased and integrated approach to waste retrieval operations was used for the GAAT Remediation Project. The project promoted safety by obtaining experience from lower-risk operations in the NTF before moving to higher-risk operations in the STF. This approach allowed project personnel to become familiar with the tanks and waste, as well as the equipment, processes, procedures, and operations required to perform successful waste retrieval. By using an integrated approach to tank waste retrieval and tank waste management, along with specialized equipment, the project was completed years ahead of the original baseline schedule, which resulted in avoiding millions of dollars in associated costs.

The MLDUA provided the dexterity and reach needed to effectively clean the walls of the GAATs and perform bulk waste retrieval operations, the Houdini ROV provided the power and mobility to break up wastes and effectively clean the floors of the GAATs, and the HMS successfully shouldered the loads from the wastes transferred out of the tanks.

Evaluation Criteria

Amount of waste successfully transferred from the GAATs and the overall waste transfer rate.

Waste Characteristics Relative to Technology

Prior to waste retrieval operations, numerous samples were collected from the tanks and analyzed to determine the chemical, radiological, and physical nature of the waste. Sludge samples were taken that revealed a wide range of variability in the waste, ranging from debris, such as chunks of gunite and other material to hard sludge, soft sludge, and crystalline material.[3]

Lessons Learned

Waste retrieval systems and equipment must be selected based on the specific tank operating conditions and constraints at each site. The Houdini and the MLDUA worked well together for efficient waste retrieval operations at the GAAT OU. The combination of the Houdini's mobility and ruggedness in operations in the bottom of the tanks with the MLDUA's reach and dexterity in

operations in the upper portion of the tanks provided an excellent system for use in tank waste retrieval operations. Sluicing operations were most efficient when the plow on Houdini pushed sludge toward the MLDUA. The MLDUA worked best for bulk sludge retrieval and wall cleaning, while the Houdini was better at plowing the residual sludge (<8 in.) to the CSEE while it was held by the MLDUA.[4]

MLDUA – The MLDUA provided the dexterity and reach needed to effectively clean the walls of the GAATs and perform bulk waste retrieval operations.

The MLDUA user interface included system controls and video monitors that displayed the video signals from the integrated MLDUA cameras, as well as the overview cameras in the tank being remediated. The MLDUA information display screens were spread across the MLDUA console, which made it more difficult for operators to monitor the system operation and

performance. Future designs for a user interface should consider the positioning of the display screen carefully and provide needed control information in a unified area that is easily visible to the operator.

Future user interface designs should avoid the use of continuous warning alerts and instead check warning status periodically to see if the warning condition clears. Continuous alerts fill the computer log with multiple alerts that are unnecessary, can hider diagnosis and repair of the error, and desensitize operators to more crucial warnings and alerts.

The MLDUA was ideal for washing and scarifying the tank walls to remove contamination; however, low visibility occurred in the tanks during these operations due to the formation of mist at the higher operating pressures. By preprogramming the MLDUA with the scarifying paths, controlled movement was achieved so that operations could continue in poor visibility.

In future applications, consideration should be given to the installation of at least a 150-lb-capacity winch inside the MLDUA TRIC structure. A winch with increased capacity would allow the use of a wider range of materials and tools.

Locating routine maintenance equipment within the Vertical Positioning Mast (VPM) housing would simplify maintenance of the housing in future applications. Another modification would be to locate certain components outside the contaminated VPM housing, including the VPM housing angle and purge/pressure sensors, the lubrication oil drain, and the fill ports for the VPM tube winches.

In the future, it would be better to mount the MLDUA umbilical tethers in cable carriers that could take the strain of the tether motion and tension, rather than placing the signal-carrying cables under tension.

Houdini ROV – The Houdini ROV provided the power and mobility to break up wastes and effectively clean the floors of the GAATs. Caution was required when driving the Houdini or manipulating the manipulator arm. Administrative controls in the form of slower travel speeds were employed to prevent collisions between the Houdini, the CSEE, and other tools and objects within the tank, including the tank wall. Problems with the hose routing of the hydraulic

transmission lines on the Houdini I ROV, as well as problems with the manipulator arm and umbilical, led to the development and deployment of the Houdini II ROV in the tanks in the STF. Another improvement, which changed the center of gravity of the vehicle to allow it to hang straight during deployments and retractions, reduced tank riser interference and self-inflicted damage to the vehicle. Prior to this improvement, the vehicle was subject to hanging on the edge of the riser sleeve at the dome of the tank during withdrawal.

The ROV should be designed to limit connector and hose stress during folding for deployment and retraction. Many of the connectors on Houdini I were subject to damage or loosening when the vehicle was folded.

Use appropriate fasteners for mobile waste retrieval systems. Vehicle vibrations produced by the lugs on the tracks produced more vibrations than originally anticipated. Locking bolts and other fasteners failed to hold. However, Nordloc™ lock washers proved to be successful in eliminating the loosening effects of the vibrations.

Use of a separate power supply for TMADS should be done to allow the system to remain energized while the ROV is being maintained.

The hoist inside the TMADS should have a separate power feed to allow continued use during maintenance and repair operations on other parts of the system. Power feeds also need to be accessible from the outside of the containment structures.

Sufficient lead time and a cold test facility are needed to ensure that operators are properly trained.

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2.2.2.2 Technology: Bulk Mixing – Sluicing

Author

B.E. Lewis, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Description of Technology

Sluicing technologies deployed at Oak Ridge National Laboratory have varied from conventional single point sluicing to confined sluicing.

Conventional Single Point Sluicing – Conventional single point sluicing was initially used in Gunite and Associated Tanks (GAATs) during the 1980's to break up and mobilize about 90% of the estimated ~400,000 gal of sludge present in the six largest GAATs. The waste was retrieved during 18 months of sluicing operations from August 1982 through January 1984.[1]

A 2.5% bentonite clay suspension in water was used as a sluicing and suspension agent during bulk retrieval operations. The single-point sluicing technique used a remotely controlled, articulated fire-hose-type nozzle positioned near the top of each tank to break apart the sludge layers in the tanks. The jet stream from the nozzle impinged on the sludge and resuspended the sludge particles. The suspended bentonite clay in the sluicing water held the sludge particles in suspension while the slurry was continuously pumped from the tank, through a grinder, and back into the tank. The grinder was used to break up any oversized particles. This operation continued until the solids concentration approached about 15 to 20% by weight (wt %). At this point, sluicing was stopped and the resulting waste slurry was transferred through underground piping to the Melton Valley Storage Tanks (MVSTs) active waste system. This process was repeated until most of the sludge was removed from the tanks.

Figure 2.3. View of the CSEE held by Houdini I and showing the operation of the rotating cutting jets used to mobilize waste material from the GAATs.

The gunite tanks in the STF were visually inspected with a remote video camera following the 1980s bulk sludge removal operations. A quantity of residual radioactive sludge, debris, and abandoned sluicing equipment remained in the tanks. Some of the tanks continued to fill with groundwater because of in-leakage through the domes of the tanks. The visual inspections of the tank interiors showed varying degrees of deterioration approaching the point that the structural integrity of the tanks could not be guaranteed.

Confined Sluicing – Confined sluicing technology was an integral part of the Radioactive Tank Cleaning System (RTCS) used to remove the remaining waste from the GAATs in the late. Various technologies were integrated into the remotely controlled RTCS. The RTCS was comprised of several subsystems, with each subsystem providing unique capabilities. The RTCS used confined sluicing technology to break up and retrieve sludge heels from the tanks. Sludge heel retrieval and wall cleaning were key activities in the tank remediation process. The

Modified Light Duty Utility Arm (MLDUA) and the Houdini I and II Remotely Operated Vehicles (ROVs) played an important role in these
operations. These systems were used to operate various tools and equipment in the gunite tanks, including a confined sluicing end-effector (CSEE). The CSEE contained a rotating manifold with three water jets that were supplied with 200-7000-psig process water. The water jets were rotated at a rate of 0-500 rpm to cut through and break up sludge (Figure 1), dilute soft wastes into pumpable slurry, or wash tank walls. The CSEE rotating cutting jets combined with the vacuum power from the axial-flow jet pump was very effective for removing sludge waste from the tanks.[2]

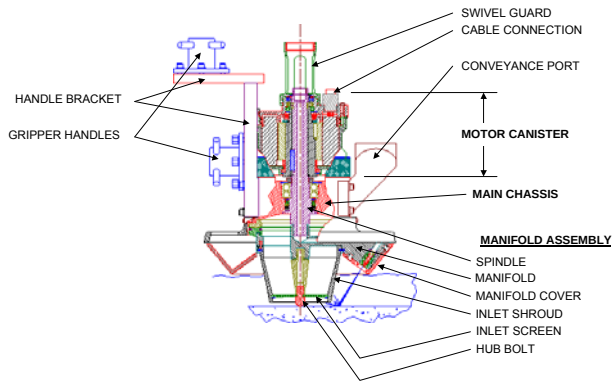

Figure 2.4. Schematic Cross Section of the CSEE Assembly

The CSEE's rotating manifold was a 15-5 stainless steel weldment with the rotor section cut from a single block of plate and welded to the shaft. The manifold arms were normal to the rotation axis and the jets converged at an angle of 35° to the axis and a 5.5° lead angle with respect to the counterclockwise rotation. The water jet nozzles were Leech & Walker type carbide inserts (0.032-in. diam) selected for their high-velocity coherent cutting-jet capability. They were mounted in a custom compression-seal holder that could be installed with just a socket wrench, and were contained in-line flow straighteners placed behind the jets. The flow straighteners were used to enhance the jet coherence and compensate for the acute bend in the water path upstream of the jet.

A frameless dc servomotor was used to drive the manifold rotation. The motor stator was pressed into the aluminum canister and the bearings and seals at the canister bottom and upper end cap supported the rotor. The manifold armature passed through the large central bore of the rotor, to which it was keyed to transfer torque.

A 10,000-psig rotary coupling adapted the manifold to the supply hose and was supported against bending moments by an external cage mounted on the motor case. The manifold and motor case was mounted to the main chassis, which included the protective fiberglass shroud ring, grab handles, and conveyance suction port. An inlet shroud with a 3/8-in. hex screen was fitted to the manifold. Figure 2 is a schematic of the CSEE assembly.

The CSEE motor was powered by a 300-Vdc 10A (continuous), 45-A (peak) power supply operating through a dc servoamplifier. The motor included Hall-effect sensors for feedback. The motor was able to achieve rotational speeds from 60 to 600 rpm. During normal confined sluicing operations 300 rpm was adequate. The motor umbilical was routed along with the waste transfer line through the HMA vertical deployment mast; therefore, no deployment reel was required.

The rotating cutting jets surrounded a vacuum head that connected to the waste conveyance system, integrated with the HMA. The dislodged waste was aspirated into the conveyance line through the central inlet system.

CSEE controls and instrumentation included a power switch and emergency stop, rotational direction and speed controls, speed and torque (inferred from current) indicators, and data connections. The local CSEE controls, amplifier, and power supply were housed in a splash-proof enclosure on the equipment platform and interfaced to remote controls and instrumentation at the control room. The CSEE was demonstrated to tolerate 2000-psi wash-down and to be readily decontaminated by a tank riser decontamination spray ring (DSR) and a handheld spray wash gun inside the deployment system glove box.

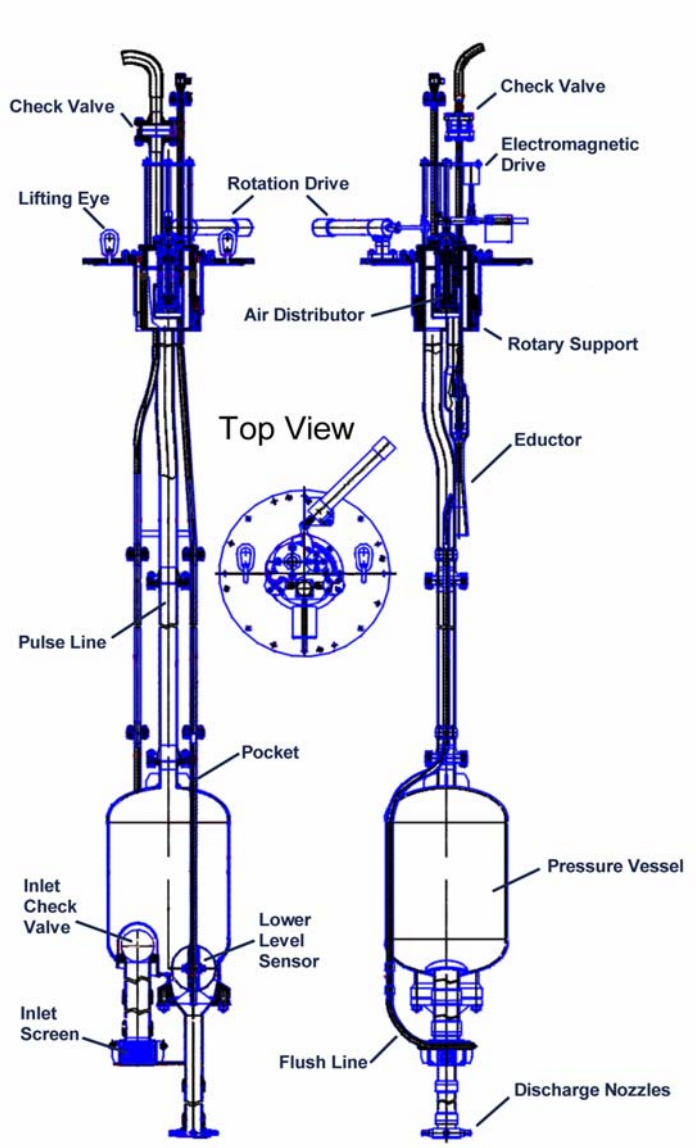

Figure 2.5. Schematic of PMP Assembly

The CSEE, including one grab handle, weighed 46 lb. It generated only moderate dynamic forces during cold testing, so it was compatible with the structural capability of the MLDUA. The CSEE is made of aluminum, stainless steel, and selected polymers. It proved sufficiently resistant to the radiation levels and chemical environment of the tanks in the STF.

Deployment Site(s)

Gunite and Associated Tanks (GAATs) at Oak Ridge National Laboratory

Performance

Conventional Single Point Sluicing – Conventional sluicing operations in 1984 removed an estimated 2,195,400 lb of sludge from the STF tanks, leaving ~10% of the original sludge in the tanks.

Confined Sluicing – Sludge retrieval rates as high as 8 gal/min were observed during cold testing. The CSEE consumed about 10 gal/min of process water, most of which was needed to drive the jet pump. The total sludge and supernate removed from the GAATs using confined sluicing technology was ~391,352 gal. These transfers required the use of a total or 420,533 gal of fresh water, or ~1.075 gal/gal of sludge and supernate.

Evaluation Criteria

The evaluation of sluicing technology performance was based on the quantity of waste removed and the amount of water used to mobilize the waste.

Waste Characteristics Relative to Technology

Resuspended sludges from the single point sluicing operations were reported to be non-Newtonian power law fluids having an apparent viscosity of 16 cP at a shear rate of 510/s.[1]

Prior to confined sluicing, numerous samples were collected from the tanks and analyzed to determine the chemical, radiological, and physical nature of the waste. Sludge samples were taken that revealed a wide range of variability in the waste, ranging from debris, such as chunks of gunite and other material to hard sludge, soft sludge, and crystalline material.[3]

Lessons Learned

Conventional Single Point Sluicing – The single point sluicing worked very well for bulk waste retrieval; although the process usually took longer than originally anticipated.

Confined Sluicing – The CSEE was a major factor in the successful retrieval of the wastes from the GAATs. The system was operated in a harsh environment with minimal problems. The only problems noted in the project summary were as follows:

- A minimal water flow should be maintained through the cutting jets on the CSEE while the exit nozzles are submerged. Maintaining a water flow to the cutting jets will prevent clogging of the nozzles when they are submerged in tank waste.
- To minimize the creation of aerosol and splattering of waste, the nozzles of the CSEE should remain submerged as much as possible while using the rotating cutting jets. Aerosols reduce visibility and provide a pathway for the spread of contamination. Splattering of waste on the

retrieval equipment and around the tank makes waste retrieval and decontamination operations more difficult.

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2.2.2.3 Technology: Bulk Mixing – Pulse Jets – Russian Pulsating Mixer Pump (PMP)

Author

B.E. Lewis, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Description of Technology

A schematic of the Russian PMP that was deployed at the Oak Ridge site is shown in Figure 1. The system consists primarily of an in-tank pressure/vacuum vessel coupled with pressurized air and vacuum sources. In addition to the PMP assembly, a tank riser interface (TRI) and a decontamination spray ring (DSR) were used to couple the PMP with the waste tank. A DSR was used to provide a water rinse of the contaminated equipment as it was removed from the tank. The TRI supported the PMP and permitted height adjustments and alignment with the tank riser. During hot deployment, the DSR was mounted to the central tank riser at TH-4 and was connected to the TRI by a flexible bellows to allow adjustment of the elevation of the PMP.

During operation of the PMP, materials from the waste tank are pulled inside the pressure/vacuum vessel (PV) through an inlet check valve when a vacuum is applied to the vessel. The inlet port is separated from the discharge line and is at a higher elevation relative to the bottom of the tank. The discharge outlet is typically positioned in the sludge layer, closer to the bottom of the tank, while the inlet remains in the supernatant. This orientation allows supernatant to be drawn into the PV and discharged into the sludge layer, which improves mixing performance. After the PV is full, the vacuum is turned off and air pressure is applied to close the inlet check valve and force the contents of the vessel out through four nozzles on the bottom of the discharge line. These operations are repeated using the fluidized waste material in the tank to

break up and mix the solids in the bottom of the tank. Conventional pumping systems are then used to transfer the waste out of the tank. During mixing operations, the PMP can be automatically rotated through a 90-degree arc in alternating clockwise and counterclockwise directions to sweep the entire bottom of the tank.

Compressed air is used to create a vacuum using the in-tank eductor. Control valves are operated in conjunction with an electromechanical axial valve in the air distributor (AD) of the PMP to direct either the compressed air flow or vacuum to the pressure vessel. Tank waste is drawn into the PV through a coarse screen and check valve assembly on the bottom of the inlet to the vessel when vacuum is applied. In the event of a plug in the inlet screen, wash water can be admitted to clean the screen. A level sensor inside the PV is used to control the durations of the pressure and vacuum cycles. A spherical float containing a central magnet surrounds a sealed pipe inside the PV. A sensor is located inside the sealed pipe that is used to detect the high- and low-level positions of the float as the vessel is filled and discharged. The high-level signal is used to signal the control system to pressurize the PV and the low-level signal is used to admit vacuum. The pressure vacuum cycle can also be controlled either locally by using mechanical timers or remotely by using timers built into the computer-based control system.[1]

Deployment Site(s)

The Russian-engineered Pulsating Mixer Pump (PMP) was deployed in gunite tank TH-4 (Figure 2) at Oak Ridge National Laboratory to mobilize the 2–3-ft layer of sludge present in the bottom of the tank. Tank TH-4 was the smallest of the gunite tanks, with a capacity of ~14,000 gal.

Figure 2.6. Tank TH-4 After Waste Mixing Operations Performed by the PMP in 2001

Performance

Waste retrieval operations began in tank TH-4 on January 11, 2001, and were completed on January 15, 2001. The PMP was successfully operated in the tank, and the majority of the waste was transferred to the active waste tank system. At the beginning of the waste retrieval operations, the tank was at full capacity due to the in-leakage of groundwater. Excess liquid was removed from the tank until the initial operating level for the PMP was reached (~5.6 ft from the bottom of the tank). The project safety documentation required that a layer of supernatant be maintained in the tank that was equal to the depth of the sludge present in the tank (initially estimated to be 2.5-ft deep). The PMP was operated over a holiday weekend for several periods ranging from 1 h to more than 10 h. The total accumulated operating time was ~24.5 h. The mixing operations effectively agitated the sludge and mixed it with the remaining supernatant. The waste slurry resulting from the mixing operations in tank TH-4 was transferred through the temporary transfer line to the active waste tank system. Initially, the inlet to the transfer pump was located on a mound of sludge estimated to be ~1 ft deep. This limited the lowest level to which waste could be removed from the tank. The inlet was later repositioned to within ~1 in. of the floor of the tank for the final waste transfers.

After the last waste transfer, only an outer band of sludge remained in the tank. This band of sludge ranged from about 1 ft to slightly over 2 ft wide. The depth of the band was ~1.25 ft. These observations indicated that the PMP had an effective cleaning radius of ~8.5 ft. After the completion of the waste transfer operations, the outer band of watery sludge slumped and spread toward the center of the tank as shown in Figure 2. Supernatant was recycled to provide a mixing medium. During mixing and sludge removal operations, a total of over 24,000 gal of slurry was transferred from tank TH-4. The residual sludge had an estimated volume of ~1098 gal, which indicated that ~82% of the sludge had been removed. The total activity of waste remaining in the tank was reduced from ~3.37 to ~0.59 Ci. It is believed that continued recycling of supernatant and operation of the PMP would have likely resulted in the removal of additional sludge. However, the DOE and Tennessee Department of Environmental Compliance (TDEC) regulators inspected the tank on January 18, 2001, and determined that additional sludge removal was not required.

Evaluation Criteria

The performance of the PMP was based on the quantity of waste removed and ease of operation.

Waste Characteristics Relative to Technology

At the beginning of the waste retrieval operations, tank TH-4 was at full capacity due to the in-leakage of groundwater. The initial sludge content of tank TH-4 was estimated at 6266 gal.[2] The total activity of the waste initially present in the tank was ~3.37 Ci.

Figure 2.7. Photo of the Flygt Mixers Prior to Installation

Lessons Learned

Although the PMP was successfully used to clean out tank TH-4, hot checkout and operation of the system was limited to a 5-day period, due to budget and schedule constraints. Additional operation of the system would have allowed time for troubleshooting and improvement of the understanding of the applicability of the system in radioactive tank waste retrieval operations. Some specific problems that occurred during waste retrieval operations included:

- The air distributor valve for the PMP would not reliably seat, which resulted in a partial loss of vacuum as the pumping chamber refilled. This problem also caused frequent shutdowns by the control system. Since the waste retrieval operations were performed in the middle of winter under relatively cold environmental conditions and the performance of the valve (and, consequently, the PMP) significantly improved as the temperature increased above freezing; moisture accumulation was suspected as the root cause of this problem. Similar problems were observed during cold testing of a slightly modified unit subsequent to hot deployment.[3]
- Erratic motion of the PMP was observed near the end of the 90º arc during waste retrieval operations. It was suspected that the air pressure supplied to the PMP was too low to permit complete movement in one smooth motion.

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2.2.2.4 Technology: Bulk Mixing – Flygt Mixers

Author

B.E. Lewis, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Description of Technology

The Flygt mixers used at Oak Ridge National Laboratory (ORNL) included a mast-mounted 15-hp submersible electric motor with a three-bladed direct-drive axial-flow propeller to agitate the waste in the tank (Figure 1). The submersible motor mixer assemblies had been successfully used in industrial wastewater treatment, paper mills, and the chemical industry applications. Each mixer was attached to a mast assembly that supported all mixer loads from a structural steel platform located above the tank. The length of the mast was adjustable to allow the depth of the mixer to be changed according to the sludge depth beneath each mixer. Once lowered into the tank, the mixer was pivoted 90 degrees from its deployment position and locked into the horizontal-axis operating configuration. In this configuration, the mixer was able to develop high axial flows in the surrounding liquid/sludge materials, mobilizing and suspending the tank sludges into readily pumpable slurry.[1]

Deployment Site(s)

Gunite and Associated Tanks (GAATs) W-5 and W-9 at Oak Ridge National Laboratory

Performance

The Flygt mixer system was generally reliable, requiring little or no maintenance. These mixers were used in conjunction with pulsed air mixer in tank W-9 to provide improved mixing performance in this tank.[2]

Evaluation Criteria

The system performance was based on ability to maintain solids in suspension during tank waste transfers. The presence or absence of piles of sludge in the bottom of a tank after a waste transfer operation was also used as a performance evaluation criterion.

Waste Characteristics Relative to Technology

Lessons Learned

System operators considered the ORNL Flygt mixers to be somewhat temperamental in regard to fault trips. Typically, fault trips were caused by an overcurrent to the motor. The system could be reset and restarted fairly quickly at slightly less current and, consequently, a lower operating speed. The Flygt mixers used in the GAATs were also considered slightly underpowered for this application, with motors rated at only 15 hp. More powerful off-the-shelf models that could be deployed through the existing risers were not available at the time of the GAAT Remediation Project.

Hardened aluminum alloy blade propellers were originally installed on the Flygt mixers, based on the expected operating conditions in the gunite tanks. During installation, one of the propeller blades broke from an impact with the tank riser and the propeller on the other mixer broke from an impact with in-tank debris. The broken propellers were replaced with stainless steel units, and no further problems were experienced.

With only two mixers, the ability to fully mix the contents of the waste tank was somewhat limited, which resulted in a region of low turbulence (and associated settling) in the tank. Potential remedies include using an automatic oscillating mixer mast to sweep the tank periodically. Also, the simultaneous use of three or more mixers in a tank would likely improve the performance of the system.

The Flygt mixer configuration in tank W-5 was such that the mixers could not be positioned any closer than 1 ft above the tank floor. This configuration has a compound effect of focusing the mixing energy directly in front of the propeller above the sludge layer and requiring a greater liquid depth/volume to operate the mixers. The additional liquid depth/volume requires additional energy to achieve a given mixing intensity or velocity relative to what would be required for a lower liquid level/volume. Future designs should provide the flexibility to position the mixers as close to the waste and bottom of the tank as possible.

The in-tank video camera system proved to be essential for positioning the Flygt mixers and monitoring their operation. The camera and lighting system was also found to be essential for inspections of the mixers and the tanks.

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2.2.2.5 Technology: Transfer Out of Tanks – DiscFlo Pump

Author

B.E. Lewis, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Description of Technology

The submersible WTP used in the transfer of waste from the Gunite and Associated Tanks (GAATs) waste consolidation tank (W-9) was a 125-hp electric motor with an integrated Discflo™ low-shear pump head. The Discflo pump combined the relatively simple design of a centrifugal pump with the capabilities of a progressive-cavity pump by using a series of rotating disks in place of a typical impeller. This type of equipment was capable of (1) pumping abrasive solids and entrained air with little or no internal wear to the pump head, (2) handling large solid particles, (3) exhibiting little or no increase in the discharge pressure even when the output line is blocked, and (4) generating discharge pressures in excess of 300 psig. The pump was equipped with a variable-frequency drive to control the pump speed and thus the desired discharge flow.[1]

The WTP was mounted on a mast that could be raised or lowered in the tank. The steel deployment mast was attached to a structural platform located above the tank. The mast enabled the pump elevation to be adjusted over a range of ~20–26 ft below the riser in 1-ft increments. The WTP assembly was deployed through a 30-in.-diam riser on the south side of tank W-9. The pump was successfully used to transfer waste slurry through the ~1-mile-long 2-in.-diam double-contained stainless steel waste transfer line.

Deployment Site(s)

Gunite and Associated Tank (GAAT) W-9 at Oak Ridge National Laboratory

Performance

The Discflo pump functioned almost flawlessly after the initial technical problems were resolved. The pump was operated ~180 h in various recirculation and transfer modes. The total amount of waste transferred was ~483,300 gal, of which ~60,500 gal was wet sludge. Waste transfer operations were intermittent, based on available capacity in the receiving waste system.

Evaluation Criteria

Ability to reliably transfer waste slurries through the cross-site waste transfer line.

Waste Characteristics Relative to Technology

Prior to confined sluicing, numerous samples were collected from the tanks and analyzed to determine the chemical, radiological, and physical nature of the waste. Sludge samples were taken that revealed a wide range of variability in the waste, ranging from debris, such as chunks of gunite and other material to hard sludge, soft sludge, and crystalline material.[2]

Lessons Learned

The Discflo pump was a vital component in the overall success of the waste transfer operations from the GAATs.

A problem with the electric power feed caused an early failure of the variable frequency drive unit and was easily corrected with the installation of an isolation transformer and line reactor.

The pump was designed to operate while submerged in water, and when the liquid level in the tank dropped below the motor housing, the motor overheated and automatically shut down. Using a secondary pump to transfer a stream of supernatant across the housing at ~5 gal/min solved the cooling problem.

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2.2.2.6 Technology: Characterization and Monitoring – Waste Conditioning System

Author

B.E. Lewis, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Description of Technology

The Waste Conditioning System (WCS) used during the Gunite and Associated Tanks (GAATs) remediation at Oak Ridge National Laboratory was composed of in-tank mixing systems, pumps, classifiers (filters), samplers, and in-line instrumentation to measure various characteristics of the waste slurry. The components considered here focus on the classifiers (filters), samplers, and in-line instrumentation, which were located in the Primary Conditioning System (PCS) module and the Slurry Monitoring Test Loop (SMTL) module.

PCS – The PCS consists of a confinement enclosure that was designed to house classifiers, samplers, a pressure transmitter, a process water flush connection, and associated valves and piping. The enclosure included a HEPA-filtered air inlet and a ventilation connection with a back draft damper that maintained a negative pressure by connection to the off-gas system of tank W-9. The PCS also used a sump to collect any leaks that may occur from the process piping and drain them back to the tank. A wash-down capability was installed to remove gross contamination inside the enclosure in the event of a leak from the primary piping. A sludge grinder was originally planned for the PCS, but was omitted based on sludge characterization data that showed the solids retrieved from the GAAT were primarily <100-μm-diam particles, which met the acceptance criteria for transfer through the cross-site transfer line.

The PCS was designed to filter out large solid particles. The two classifiers, manufactured by Orival, Inc., were installed in parallel so either could serve as a backup if one was off-line. During operation, the classifiers could operate automatically to flush large solids back to tank W-9 using either an operator-selected time interval or a pre-determined pressure drop. Isolock™ in-line samplers, manufactured by Bristol Equipment Co., were located both upstream and downstream of the classifiers. The filters were used only briefly. Valving installed in the PCS permitted the classifiers to be bypassed if desired.

Double-contained piping was installed from the waste transfer pump discharge to the confinement enclosure and from the PCS discharge to the SMTL enclosure. Steel pipe was used for secondary containment for above-ground piping in the waste transfer pump loop (with steel primary piping). PVC pipe was used as secondary containment for some under-ground transfer piping applications (with steel primary piping). The classifier back-flush drain was also doubly contained to minimize the potential for a release. System instrumentation and controls permitted remote monitoring and operation of the samplers and valves from the GAAT operations control trailer.

SMTL – The SMTL module contained instrumentation housed in a steel enclosure that provided real-time monitoring of the suspended solids concentration, density (including dissolved solids), viscosity, temperature, flow, and particle-size range in the waste slurry. The criteria for transfer of waste slurries through the waste transfer line required the concentration of suspended solids to be <5 wt % and the maximum particle size to be 100 μm.[1] The SMTL was designed to provide real-time monitoring of the radioactive slurry conditions before and during waste transfers to the active waste system. These monitoring capabilities were designed to operate remotely to minimize radiation dose to workers. The SMTL also included a sample port and HEPA filter air inlet, which were incorporated into a containment box.[2]

The WCS was designed for operation with pressures up to 300 psig, which was consistent with the maximum operating pressure of the waste transfer pump and the ORNL waste transfer line. The PCS and SMTL systems were designed so that any or all of the components could be used or bypassed as operating conditions required.

Deployment Site(s)

Gunite and Associated Tanks (GAATs) at Oak Ridge National Laboratory.

Performance

The PCS was used primarily to collect slurry samples during waste transfers to the active waste system at ORNL. The capability to physically reduce particle size was never installed because it was not needed, based on sludge characterization data that showed the solids retrieved from the GAAT were primarily <100 μm-diam particles, which met the acceptance criteria for transfer through the cross-site transfer line. This limit was applicable during the waste transfers to minimize the risk of plugging the 1-mile-long pipeline cross-site transfer line. The particle size classifiers (filters) were used only during the initial transfer of material to the active waste system and were subsequently bypassed. The filters frequently clogged and reduced the downstream pressure. The pressure drop triggered the automatic backflush cycle. The filters were installed in parallel to allow one to be backflushed while the other continued to operate. The filters clogged almost constantly, making it virtually impossible to maintain the pressure and flow needed for the transfer. The sticky, cohesive nature of the sludge particles appeared to contribute to the blockage of the filters. For these reasons, use of the filters was discontinued. Data from the LasenTec particle size analyzer in the SMTL as well as laboratory analytical data supported the fact that virtually no solids >100 μm were present in the slurry, which led to the conclusion that the classifiers were not required.[3] The SMTL was operated continuously while slurry was being transferred to the ORNL active waste system and was in operation for a total of about 298.5 h.

Project operators and management relied on the SMTL as real-time evidence that the waste slurry met the ORNL LLLW system waste acceptance criteria (WAC) with respect to the 100-μm particle-size limit. The SMTL also provided data on the solids content of the slurry. While the SMTL provided a useful trend in total solids content (i.e., change in value), the actual percentage of solids was not a reliable measurement (i.e., absolute value). Project operators and managers chose instead to rely on analytical laboratory data for solids content. Therefore, laboratory analysis of slurry samples was used to estimate the quantity of solids transferred and the amount of residual material in the tanks following the final batch transfers. In addition to analytical data, operators also relied on radiation readings from a gamma radiation detector that was mounted on the waste transfer line from the SMTL. The detector provided reliable feedback on the radioactive material content of the waste slurry being transferred from the GAATs.

Evaluation Criteria

Ability of the system to provide usable solids content and size distribution data.

Waste Characteristics Relative to Technology

Prior to confined sluicing, numerous samples were collected from the tanks and analyzed to determine the chemical, radiological, and physical nature of the waste. Sludge samples were taken that revealed a wide range of variability in the waste, ranging from debris, such as chunks of gunite and other material to hard sludge, soft sludge, and crystalline material.[4]

Lessons Learned

The PCS performed well with two of the four key functions for which it was designed.

The Isolok™ samplers and the pressure transmitter were vital in monitoring the transfer of waste to the active waste system.

The roughing filters in the PCS were used only once because of (1) difficulties with blinding of the filters by the sticky solids and (2) the evidence provided by the LasenTec instrument and sample analysis data that showed that the solid particles were typically <100 μm.

The SMTL proved to be of critical value to the GAAT project in regard to providing the credible data needed to demonstrate that the waste slurry met the particle size limits of the WAC for the waste transfer line. This capability took on even greater importance when the PCS classifiers were bypassed. However, the inability of the SMTL to provide credible absolute slurry density measurements meant that samples had to be taken during every transfer to verify that nuclear safety limits were met and to maintain a material balance.

Instrumentation in the SMTL provided the operators with information on the instantaneous flow rate. By monitoring the flow rate and estimating the nominal flow rate, the operators could estimate the total volume of waste delivered during a given period of time. The addition of a flow totalizer that is resettable and remotely indicating would be useful in maintaining accurate waste transfer balances.

In-line carrier fluid density measurements are needed to improve the quality of the information from the SMTL. The suspended solids concentration in the slurries was reasonably estimated from the slurry density measurement obtained with the in-line Coriolis meter; however, the suspended solids concentration measurement could be improved by also simultaneously monitoring the density of the carrier fluid.

References

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2.2.2.7 Technology: Characterization and Monitoring – Camera and Lighting

Author

B.E. Lewis, Oak Ridge National Laboratory, Oak Ridge, TN 37831

Description of Technology

A variety of remote-controlled video cameras and lighting systems were used to inspect the tanks and monitor the operation and performance of the waste retrieval systems. Remote cameras and lighting features were an integral part of various components of the waste retrieval system, including the Modified Light Duty Utility Arm, Houdini I and Houdini II Remotely Operated Vehicles, and the Waste Retrieval and Transfer System. Each of these systems included special controls and monitors for its integrated cameras. Figure 1 is a view of the control room monitors used to observe the operation of the Houdini I during cold testing.[1]

Figure 2.8. View of Control Room Monitors Used to Observe the Operation of the Houdini I

For preliminary surveys, a single remote-controlled video camera, with a light integrated into the camera housing was mounted on a 3-DOF (degree-of-freedom) deployment system (pan, tilt, and vertical extension), which was inserted through a tank access riser. The housing for the camera included a single high-intensity light, which illuminated the tank. The camera was mounted on a 3-DOF extended-reach mount that could be inserted through a 4-in.-diam riser but was usually deployed through a 12-in.-diam riser. The camera and integrated light were used to perform tank inspections in each of the gunite tanks. A video cable from the camera ran through the center of the vertical extension and connected with the remote control unit, which in turn was connected to a monitor and video tape recorder. The remote control unit contained a clock to monitor the time; indicators for the degree of pan and tilt; and a text generator to label important information, such as the tank number and date of inspection. The pan and tilt features on the control unit allowed the camera operator to pan and tilt the camera nearly 360° in any direction. The illumination intensity was controlled by the operators. The camera included a zoom feature with both automatic and manual focusing capabilities. This feature turned out to be important when performing tank inspections, because manual adjustments of the focus were sometimes needed to provide a clear picture of interesting tank or waste features, especially when the auto focus

focused on water droplets on the lens. Methodical visual inspections of the interior and monitoring of some waste-sampling operations provided important information on the interior condition of the tanks and gave an indication of the amount of waste the tanks contained.

Before the waste retrieval equipment was deployed into the tanks, four remote-controlled video cameras and lighting systems were positioned in 12-in.-diam access ports installed in the tank domes. A multiplexed pan, tilt, and zoom controller was installed in the GAAT operations control room. Control and video cables from the cameras were connected to this unit. The controller allowed the equipment operators to conveniently and rapidly select and control the various cameras.

Deployment Site(s)

Gunite and Associated Tanks (GAATs) at Oak Ridge National Laboratory

Performance

The cameras used inside the GAAT tanks suffered cumulative damage from overheating and radiation exposure, resulting in frequent repairs and replacements. On average, the camera modules were replaced about once every 6 to 12 months.

Evaluation Criteria

Longevity in tank environment and image quality.

Waste Characteristics Relative to Technology

Prior to confined sluicing, numerous samples were collected from the tanks and analyzed to determine the chemical, radiological, and physical nature of the waste. Sludge samples were taken that revealed a wide range of variability in the waste, ranging from debris, such as chunks of gunite and other material to hard sludge, soft sludge, and crystalline material.[2]

Lessons Learned

At least two camera views are needed for in-tank operations. A single camera view does not provide the operator with an adequate depth perception to reliably operate the in-tank systems.

Cameras used for monitoring interior tank operations should be equipped with adequate zoom capability to provide detailed close-up views and light sensitivity to provide views with adequate depth of field.

Ensure that the camera systems can be easily positioned inside the tanks. Each camera system at the GAATs was mounted on a pole that could be vertically extended by attaching additional 6-ft sections. A separate camera cable was factory installed inside the camera pole for convenience and contamination control.

Ensure that the cameras are waterproof. A waterproof box with a connector was attached at the top of the camera pole so that the main camera cable could be connected from outside the tank. Although this worked well, a plastic bag covering the top of the extension pole and riser was still required to prevent water from entering the connector box and the vinyl boot inside the tank.

Use vinyl boots to protect the camera equipment. A 2- to 3-in. rubber PVC pipe coupler was attached to the vertical extension pole above the camera head using hose clamps. This technique was used to secure a vinyl boot, which was taped at the coupler and at the top edge of the aluminum camera adapter to keep the vertical extension pole from becoming contaminated.

Use cameras that are easy to replace and inexpensive. The in-tank overview camera systems used in the GAATs were not radiation hardened but were high-quality cameras that cost ~$1K each. The total cost of each overview camera system used in the GAATs was ~$30K, which included the waterproof sealed camera module, lights, pan and tilt, extension poles, cables, and controllers.

In-tank lighting systems must be compatible with the environment and the selected camera system. The two factory-standard 35-W lamps integrated into the video camera housing were not sufficient to illuminate the 25- to 50-ft-diam tanks. Camera housings were modified to include a single 250-W lamp with a polished stainless steel reflector shield, instead of the two factory-standard 35-W lamps. Heat from the 250-W lamp, plus the position of the housing relative to the camera, caused the camera to frequently overheat. In future applications, consider positioning the lights to the side of the camera and maintain enough distance so that heat generated from the lights does not overheat the camera. Adequate heat dissipation for the lamp housings are needed to extend the life of the cameras. As a result of the overheating problems with the in-tank camera, a heat shield was required between the 250-W lamp and camera. Cameras can be cooled using a variety of means, including internal purges, internal fans, heat shields, or other means to dissipate the heat from high-wattage lamps. When cameras are not in use, they should be turned off or operated with reduced lighting.

To increase the visibility of equipment during waste retrieval operations, paint in-tank equipment with bright colors that provide high visibility and contrast in the tanks. Visibility is limited during operations that generate a fog/mist. Reflective tape can also be used to make equipment more visible in high-fog conditions. In-tank visibility can also be improved by using indirect lighting during high-mist- or fog-generating operations. Additional light sources, installed perpendicular to the camera view, may also be used to provide indirect lighting and cast shadows to aid in depth perception.

Provide lights and cameras inside the equipment containment structures to monitor equipment deployments or retractions and to provide additional views for equipment operators.

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2.2.3 Idaho Site Input

1. What is the name of the technology?

   Idaho Tank Farm Cleaning/Spraying Systems

2. Brief description of technology (ie no more than 1-2 paragraphs)

Two technologies are involved in the tank cleaning/spraying systems deployed at the INL tank farm. The INL tank farm is located at the Idaho Nuclear Technology Engineering Center (INTEC). The tanks are 300,000 gallon tanks, underground waste storage tanks that are about 40' diameter and constructed from stainless steel. The two technologies are a directional spray nozzle attached a rotating mast and the "wash ball". The controllable, directional nozzle was attached to a vertical mast protruding into the tank that could rotate, the nozzle could move up and down. The two directional nozzles, which carried the lights and cameras for viewing, were used to wash the walls of the tank if solids were not sluiced away with the wash ball. Two masts with directional nozzles were employed in each tank.

The second technology, the wash ball, is a Spin-Jet, Co. articulated jet nozzle. This system is composed of two opposed nozzles that are positioned high in the tank and rotated in two axes such that all surfaces of the tank are sprayed. The nozzles rotate vertically while the base turns horizontally. It carries the solids to the tank floor and agitates solids on the floor at the same time. While two masts and the wash ball were washing the tank and sluicing the solids, a steam jet positioned at the bottom of the tank was removing the slurry and transferring it to a separate tank farm tank.

i. Which site, tank(s), basins, vaults, etc was it deployed?
   This residue removal technology was used at the INL tank farm at INTEC in tanks WM-180 thru WM-186. It has not been used in the basins or vaults.

ii. How well did it work?
   The system worked very well. The INTEC tank farm solutions were acidic, maintained in stainless steel and contained few solids (very low in comparison to SRS and Hanford tank farms). However, a small amount of nitric acid was added to tank WM-185 to dissolve some tenacious solids.

iii. What technical evaluation criteria were used to determine whether it worked (no more than 2-3)?
   These spraying systems were mocked up (in large scale "cold" test beds) and tested to determine their ability to remove solids from the tank walls. It is unknown whether there was formal criteria used with these equipment.

iv. What is the relationship between the technology and characteristics of the waste (ie waste properties, rheology, and other physical behaviors)?
The rheology of the solids in the INL tank farm tanks was substantially different from that in most other tank farm tanks throughout the DOE Complex. A table of tank farm solids characteristics is given at the end of this report. Mainly, the INL solids were free flowing and not consolidated, unlike most DOE tank solids. This allowed the INL solids to be sluiced with water, moved around the tank bottom and removed with a steam jet system.

v. What were the lessons learned (high level – no more than 2-3)?
A larger steam jet (2" versus 11/2") was used later in the cleaning and it worked quite a bit better.
Another lesson was learned when using the nitric acid in tank WM-185. While this was effective at loosening the WM-185 "jelled" solids, it also destroyed the camera cables and caused substantial difficulty because of the nitric acid fumes.

vi. Is there key documentation that is available which provides technical information regarding the technology, especially lessons learned and recommendations?
Some reports exist on the wash ball and other tank cleaning apparatus in use at the INL. Procedures for use may be found in INL-TPR-7098. A plan for tank cleaning is available in DOE/ID-10802, Idaho HMA/RCRA Closure Plan for INTEC Tanks WM-182 and WM-183. Many other videos, reports and other information may be obtained by contacting Steven Butterworth, 208-520-7990; Frank Ward, 208-690-9587; Mike Swenson, 208-533-3376; Mike Patterson, 208-526-5525; Rick Demmer, 208-526-1692.

Table 2.2. INL Waste Slurry (solids heel materials) Properties for Two Tanks

Analyte	WM-180	WM-189	Units
Intersitital Water	80	70	%
Particle Size (med)	10	30	um
Particle Distribution	2-70	2-200	um
Settling rate	Slow (1)	Fast (5)	g/hr
Sludge Viscosity	2.2	1.9	cP
NaNO3	62	29	%
Al(NO3)	17	27	%
K3H6Al5(PO4)8	-	13	%
KNO3	7	-	%
FePO4	-	5	%
Al2O3	5	-	%
Zr(HPO3)2	3	-	%
SiO2	-	5	%
Sr(89+90)	1.00E-04	6.00E-05	Ci/g
Cs-137	1.40E-04	2.60E-04	Ci/g
Pu-238	2.20E-06	8.70E-05	Ci/g

2.2.4 Hanford Site Input

2.2.4.1 Sluicing

a. What is the name of the technology: Modified Sluicing

b. Brief description (1-2 paragraphs):

Tank waste retrieval has been conducted at the Hanford Site over the last few decades using a method referred to as Past Practice Hydraulic Sluicing. Past Practice Hydraulic Sluicing employs large volumes of DST supernatant and water to dislodge, dissolve, mobilize, and retrieve tank waste. New and innovative tank waste retrieval methods that minimize and control the use of liquids are being implemented for the first time. These tank waste retrieval methods replace Past Practice Hydraulic Sluicing and employ modified sluicing. The hydraulic sluicers used for C-Farm tank retrieval are hydraulic actuated sluicers commercially manufactured for removal of waste sludge from petroleum tanks with minor modifications performed to interface with Hanford's waste tanks.

c. Where was it deployed: All 7 tanks retrieved to date have had sluicing conducted in them in conjunction with other methods.

d. How well did it work:

The first retrieval method, termed "past-practice" hydraulic sluicing was initiated in November 1998 and completed in October 1999. This method introduced high-pressure, high-volume DST supernatant in the tank to dislodge, dissolve, and mobilize the waste for removal by the retrieval pumping system. This system was successful at retrieval of about 187,000 gallons of sludge and resolving high-heat safety concerns with tank C-106. About 36,000 gallons of supernatant and hard sludge were left in the tank following this initial retrieval campaign. Tank C-106 was further retrieved to a residual waste volume of 470 ft^3 using modified sluicing and oxalic acid dissolution.

Tank C-103 was retrieved to a residual volume of 351 ft^3 using a modified sluicing technology. Tank S-112 retrieval was completed February 28, 2007, meeting the TPA Limits of less than 360 cu ft using saltcake dissolution, modified sluicing, in-tank vehicle with high pressure water spray and caustic dissolution. Tanks C-108 and C-109 have been retrieved to 90% and 85% respectively using modified sluicing.

- Retrieval of tank C-109 was started in June of 2007 with a starting volume of about 63,000 gallons using modified sluicing. Retrieval of 85% of the waste was achieved by August 2007. The retrieval efficiency again significantly reduced with a significant residual heel. This has made it necessary to install a new piece of equipment. See discussion below of the Fold Track.

e. What technical evaluation criteria were used to determine whether it worked:

Regulatory requirements for SST waste retrieval and tank farm closure are established in the Hanford Federal Facility Agreement and Consent Order (HFFACO), better known as the Tri-Party Agreement, or TPA. The HFFACO was signed by the

DOE, the State of Washington Department of Ecology (Ecology), and U. S. Environmental Protection Agency (EPA) and requires retrieval of as much waste as technically possible, with waste residues not to exceed 360 ft^3 in 530,000 gallon or larger tanks; 30 ft^3 in 55,000 gallon or smaller tanks; or the limit of waste retrieval technology, whichever is less. If residual waste volume requirements cannot be achieved, then HFFACO Appendix H provisions can be invoked to request Ecology and EPA approval of an exception to the waste retrieval criteria for a specific tank.

f. What is the relationship between the technology and characteristics of the waste?

The SSTs contain mostly radioactive saltcake and sludge waste. The waste is primarily sodium nitrate and sodium nitrate salts; and metal phosphate, carbonates, oxides, hydroxides, and sulfate sludge. Sixty-seven of the 149 SSTs are known or suspected to have leaked an estimated 1 million gallons of waste into the surrounding soil.

These tanks received metal waste sludge primarily from PUREX plant operations. Several tanks within the C Farm have had (in the past) waste sludge temperatures in the tank increase to above 212 degrees Fahrenheit due to the quantities of Strontium-90.

g. What were the lessons learned:

- Sluicing works well for 90% to 95% retrieval. For tanks C-108 and C-109 modified sluicing was no longer effective at retrieving the remaining 5,000 to 10,000 gallons of residual sludge. Waste chunks on the bottom of the tank that are too cohesive to break up and/or too heavy to move with the sluice nozzles has resulted in not meeting retrieval goals with sluicing alone. Sluicing has worked well for soft salts, not well for hard monolithic salts, and has been ineffective with heavy sludge for the last 5000 gallons in a 75ft diameter tank.
- Need 3 sluicers to avoid blind spots that cannot be pushed around. Works very slowly on hard heels.
- Sluicing cannot be used for tanks that have or are suspected to have leaked.
- Two critical equipment failures occurred during the retrieval. The slurry pump used for C-103 was an off-the-shelf 10-hp, bottom suction, top discharge, centrifugal pump commercially manufactured by the Gorman Rupp Company. After approximately 24-hours of operation in the waste tank, the C-103 pump failed with a dead short to ground in all three of the motor phases. Although high radiation levels prevented the failed pump from being disassembled/inspected, shop testing of identical spare pumps and disassembly/inspection identified a common failure mechanism characteristic of the pumps (primary and secondary seal failure occurring early in the operating life of the pump which allowed moisture to enter the air filled winding cavity). To improve the pump reliability, CH2M HILL worked with Gorman Rupp to enhance pump manufacturing and testing procedures for pumps supplied to Hanford. Additionally, extensive testing following mounting of the pump on the pump assembly was conducted prior to installation. This pre-installation testing included a 96-hour challenge test, with periodic megger testing, oil removal and inspection, and seal cavity vacuum testing. The enhanced pump manufacture Quality Control measures combined with the extensive on site

testing has greatly improved pump reliability. To date, no additional pump failures have been experienced during subsequent retrievals.

- During C-Farm sluicing two sluicer failures have occurred (one failure during C-103 retrieval and one failure during C-108 retrieval). Both sluicers experienced identical failure mechanisms which resulted in the loss of the sluicer nozzle vertical angle positioning and both were replaced with identical sluicers. High radiation levels associated with the failed sluicers prevented post failure evaluation to determine the exact cause of failure, however, troubleshooting identified both failures were associated with the hydraulic motor gear drive or linkage connection. Prior to installing replacement sluicers, the sluicer manufacturer was consulted and extensive pre-installation testing and inspection was performed. To understand the exact failure mechanism, a spare sluicer was provided to the sluicer manufacturer and subjected to a 300-hour continuous run test. During the testing, the sluicer failed after approximately 200 hours of operation with conditions identical to those previously experienced during sluicing operations. The failed sluicer was disassembled and inspected which identified the hydraulic motor gear drive had failed. Further investigation into the cause of the failure identified the drive linkage (over center cam linkage) was creating unacceptable load conditions during full extension of the sluice nozzle. To resolve the problem, the sluicer nozzle elevation position linkage was redesigned and replaced with a chain and sprocket assembly which eliminated excessive loading at full extension. Following modification, the sluicer successfully completed over 300-hours of continuous operation with no failure or signs of excessive wear.
- One of the sluicers located in C-108 failed in a similar manner to C-103 about 2 months into retrieval, requiring replacement.
- An increase in retrieval efficiency from 45% to 85% has been achieved and reflects the improvements due to lessons learned. The key to implementation of the lessons learned has been to maintain the majority of the system simple, largely unchanged, and target specific failure mechanisms to limit the introduction of new failures. The major equipment failures and replacement time. The first 50% to 75% volume retrieval is achieved quickly, however the final 15% to 25% is time consuming to break-up, move to the pump and mobilize the waste for transfer. Debris, obstacles, monolithic layers and waste chunks are not visible at the start of retrieval; therefore planning for those specific issues is difficult.
- The slurry pump is best located in the center of the tank if risers are available for access. In this manner, the dish- shaped tank bottom can be used most effectively to allow pumping to the lowest point in the tank and minimize residuals.
- Permanently installed stainless steel pipe-in pipe transfer piping was used for transferring waste from C-106 to the DST receiver during past practice sluicing operations. While it was effective for use, materials and installation were very expensive in this application. During oxalic acid dissolution and modified sluicing, an above-ground Hose-in-Hose Transfer Line was utilized which provided high performance at a significantly reduced cost.

- During the bulk retrieval operations in the late 1990s, DST supernatant was recycled during the retrieval operations and used as the sluicing media. This was very effective at mobilizing the tank sludge and reducing the total waste generated during the retrieval operations. About 10 million gallons of supernatant has been recycled during the retrieval of waste from the C Farm Tanks saving important DST space and eliminating the need for additional tank evaporation campaigns.
- The initial methods for determining the residual volumes at a 95 percent confidence interval resulted in very large upper bounds and largely overstated the residual volumes. This methodology was revised, resulting in a smaller residual volume estimate at a 95 percent confidence interval.
- More extensive slurry pump testing is necessary to fully identify potential pump problems. A 96-hour run-in at vendor prior to delivery and pre-installation testing increased confidence in operability of pump.
- Adjustable height slurry pump minimizes plugging and shutdown, and increases sluicing efficiency.
- New sluice controls (electric to hydraulic) allow faster response at sluice nozzle. Removal of ~4000 Ft. of hydraulic hose negated need for glycol heating system.

h. Key documentation available:

- T.L. SAMS,"Retrieval Data Report for Single-Shell Tank 241-C-203," RPP-RPT-26475, Revision A, CH2M HILL Hanford Group, Inc., Richland, Washington (2005).
- CH2M-0502499, "Retrieval Report for Single-Shell Tank 241-C-202," (External letter from E.S. Aromi, CH2M HILL, to R.J. Schepens, ORP, August 25), CH2M HILL Hanford Group, Inc., Richland, Washington (2005).
- RPP-RPT-28451 Evaluation of the C-103 pump failure

2.2.4.2 Oxalic Acid in C-106

a. What is the name of the technology: Oxalic Acid

b. Brief description (1-2 paragraphs):

c. Where was it deployed: C-106 only

d. How well did it work:

Retrieval operations were performed in C-106 in 2003, using a combination of modified sluicing, with oxalic acid dissolution. During this retrieval, a total of 142,000 gallons of 0.9 molar Oxalic Acid were added to C-106 in 6 batches. After each batch, the acid was allowed to fully react with the sludge material, pumped to the receiving DST and sluicing operations were performed to remove the loosened sludge. This process was repeated until no additional waste was being removed. Tank C-106 was retrieved to a residual waste volume of 470 ft^3 using these methods.

e. What technical evaluation criteria were used to determine whether it worked:

Regulatory requirements for SST waste retrieval and tank farm closure are established in the Hanford Federal Facility Agreement and Consent Order (HFFACO),

better known as the Tri-Party Agreement, or TPA. The HFFACO was signed by the DOE, the State of Washington Department of Ecology (Ecology), and U. S. Environmental Protection Agency (EPA) and requires retrieval of as much waste as technically possible, with waste residues not to exceed 360 ft^3 in 530,000 gallon or larger tanks; 30 ft^3 in 55,000 gallon or smaller tanks; or the limit of waste retrieval technology, whichever is less. If residual waste volume requirements cannot be achieved, then HFFACO Appendix H provisions can be invoked to request Ecology and EPA approval of an exception to the waste retrieval criteria for a specific tank.

f. What is the relationship between the technology and characteristics of the waste?

g. What were the lessons learned: Worked moderately well, but was not able to dissolve all residual sludge as was hoped.

h. Key documentation available:

- R.A., DODD, "Progress in Retrieval and Closure of First High-Level Waste Tank at Hanford: Single-Shell Tank C-106," WM'05 Conference, February 27 – March 3, 2005, Tucson, AZ (2005).

2.2.4.3 Ultra-High Pressure Mixers

a. What is the name of the technology: Ultra-High Pressure mixers – Rotary Viper

b. Brief description (1-2 paragraphs):

c. Where was it deployed: S-102 only

d. How well did it work:

e. What technical evaluation criteria were used to determine whether it worked:

f. What is the relationship between the technology and characteristics of the waste?

g. What were the lessons learned:

h. Key documentation available:

2.2.4.4 Fold-Track

a. What is the name of the technology: Fold Track

b. Brief description (1-2 paragraphs):

A mobile retrieval tool which can be installed in the tank through a 12" riser and can mechanically break-up and push the waste to the slurry pump. Water jets installed on the tool also aid in mobilizing the solids. The Fold track was installed in C-109 in April 2008.

c. Where was it deployed: C-106 only

d. How well did it work:

Tank C-106 was retrieved to a residual waste volume of 470 ft^3 using oxalic acid dissolution and modified sluicing. Tank C-103 was retrieved to a residual volume of 351 ft^3 using a modified sluicing technology.

e. What technical evaluation criteria were used to determine whether it worked:

Tri-Party Agreement.

f. What is the relationship between the technology and characteristics of the waste?

g. What were the lessons learned:

Worked moderately well, but was not able to dissolve all residual sludge as was hoped.

h. Key documentation available:

2.2.4.5 Salt Mantis

a. What is the name of the technology: Salt Mantis

b. Brief description (1-2 paragraphs):

c. Where was it deployed: S-112 only

d. How well did it work:

e. What technical evaluation criteria were used to determine whether it worked:

Tri-Party Agreement.

f. What is the relationship between the technology and characteristics of the waste?

g. What were the lessons learned:

Worked moderately well, but was not able to dissolve all residual sludge as was hoped.

h. Key documentation available:

2.2.4.6 High Molarity Caustic

a. What is the name of the technology:

b. Brief description (1-2 paragraphs):

c. Where was it deployed: S-112 only

d. How well did it work:

e. What technical evaluation criteria were used to determine whether it worked:

Tri-Party Agreement

f. What is the relationship between the technology and characteristics of the waste?

g. What were the lessons learned:

h. Key documentation available:

2.2.4.7 Cameras/ Lighting

a. What is the name of the technology: In-Tank Cameras

b. Brief description (1-2 paragraphs):

c. Where was it deployed: C-106 only

d. How well did it work:

Tank C-106 was retrieved to a residual waste volume of 470 ft^3 using oxalic acid dissolution and modified sluicing. Tank C-103 was retrieved to a residual volume of 351 ft^3 using a modified sluicing technology.

e. What technical evaluation criteria were used to determine whether it worked:

Retrieval was stopped when the limits of the technology had been achieved. For example and a residual waste volume in C-103 was measured to be 338 ft^3, with a 95% upper confidence limit (UCL) of 351 ft^3. This tank met the criteria for completion of retrieval per the TPA.

f. What is the relationship between the technology and characteristics of the waste?

g. What were the lessons learned:

- The in-tank cameras have had to be removed and cleaned several times. Mist generated in the tank from the sluice nozzles created a film over the camera lenses limiting the visibility. A lens shield constructed out of an empty drink bottle, kept mist off of the lens and significantly decreased the maintenance required on the camera. In total, about 50 days of sluicing was performed with about 2.3 million gallons of DST supernatant recycled to complete retrieval.
- Moving camera from ventilation exhaust to ventilation inlet. To date this appears to be minimizing the amount of contamination on the cameras, hence, camera life is extended and removal/installation is easier.
- Providing two camera risers with dual ports on each riser will increase operational flexibility and should minimize downtime for camera repairs/replacement. Work platforms at camera risers will also decrease camera swap out time, and minimize retrieval downtime.

h. Key documentation available:

2.2.4.8 Monitoring or Volume Measurement

a. What is the name of the technology: CCMS

b. Brief description (1-2 paragraphs):

c. Where was it deployed:

d. How well did it work:

e. What technical evaluation criteria were used to determine whether it worked:

f. What is the relationship between the technology and characteristics of the waste?

g. What were the lessons learned:

h. Key documentation available:

- J.M. WIMETT, et al., "Calculation for the Post-Retrieval Waste Volume Determination for Single-Shell Tank 241-C-203, RPP-CALC-25672, Revision 0, CH2M HILL Hanford Group, Inc., Richland, Washington (2005).

2.2.5 Savannah River Site Input

2.2.5.1 Advanced Design Mixer Pump

Introduction

The concept of the Advanced Design Mixer Pump (ADMP) originated as a joint program between Hanford and Savannah River Site (SRS). The intent of the ADMP program was to develop technology directed at resolving technical issues associated with traditional long-shaft slurry pump designs. The prototypical pump was built by Lawrence Pumps, Inc. and subsequently tested at the SRS TNX Full Tank Test Facility. A systems engineering evaluation (reference 4) considered the ADMP as an option for suspending the sludge solids in Tank 18 and concluded that it was a viable alternative. The ADMP was subsequently selected because it could meet functional requirements and was readily available. After refurbishment and pre-operational testing, the ADMP was installed in Tank 18. Although unexpected waste conditions and pump phenomena hindered waste removal performance, the ADMP successfully suspended the majority of sludge solids into a slurry allowing transport from the tank via a separate transfer pump. This technical report documents the operational history of the ADMP in Tank 18. For further

background and details, reference 1 provides a brief history and general description of Tank 18, describes the overall approach for removing the bulk of the sludge in Tank 18, and a description of the ADMP configuration.

ADMP History

The Advanced Design Mixer Pump (ADMP) is a prototypical pump developed by Lawrence Pumps, Inc. (LPI) as a joint program between Hanford and SRS. The specification for the ADMP included both Hanford and SRS application criteria. After testing at LPI, the pump was delivered to SRS in December of 1997 and placed in the TNX Full Tank Test Facility. The ADMP was operated at the TNX Full Tank Test Facility for approximately 4200 hours. Of the total operating hours, approximately 130 operating hours took place in a kaolin / water mixture. The majority of operating hours was at a speed of 950 rpm's or less. The ADMP experienced one bearing failure, with other bearings showing signs of failure, near the end of the 4200 hours and shortly after the start of testing in the kaolin / water mixture (references 2 and 3).

A System Engineering Evaluation team identified the ADMP as a viable option to suspend the sludge in Tank 18 (reference 4). The ADMP was disassembled and refurbished (seals, bearings, modified inlet screen, new motor, and refurbished impeller) prior to further operational testing at the TNX Full Tank Test Facility. The final test was executed under procedure HLWD-F-322 and included a 72-hour endurance run-in test in approximately 5 feet of water. Test data concluded that the ADMP was capable of performing its function and meeting Tank 18 waste removal mission requirements. The pump was removed from the test tank, placed on a shipping cradle, and delivered to F Tank Farm. Prior to installation the pump column air supply system was tested while the ADMP was on the shipping cradle. Problems were encountered with leaks at some of the ADMP column flanges. This was attributed to thermal expansion and contraction

of the pump column due to changes in ambient temperature and solar heating of the column due to direct exposure to sunlight.

The ADMP was installed in Tank 18 on October 22, 2002. During installation it was found that the pump would not fully insert into the tank due to unknown obstructions at the bottom of the tank. It was known that there was approximately twenty two inches of sludge in the area of the ADMP inlet screen, but it was believed to be sufficiently loose such that it would disperse under the weight of the ADMP. It was later determined to be compacted sludge and steel tapes. The original design would result in the center-line of the discharge nozzles being approximately 23 inches off of the tank bottom. A mounting modification was implemented allowing the ADMP to be located at a higher elevation to avoid the interference. The final position resulted in the centerline of the discharge nozzles being approximately 39.3 inches off of the tank bottom. The ADMP discharge nozzle orientation with the tank resulted in a sweeping pattern between NE and SW.

The ADMP was initially operated for 3 hours on November 27, 2002 to mix and distribute corrosion inhibitor (approximately 22,000 gallons of sodium nitrite) previously added to the tank. The pump was then run for 8 continuous days starting December 2, 2002. This run was originally intended to be the first cleaning cycle, but was stopped and deferred until February due to changes in F Tank Farm process Advanced Design Mixer Pump History planning priorities. Two slurry samples collected at the end of the 8 day run contained only trace amounts of sludge solids. Questions regarding the sample bottles used (small mouth) and sample collection method made the reason for the low %-solids unclear, however it raised concern regarding the ADMP mixing effectiveness during the eight day run. The tank liquid level of 90 inches prevented a visual estimate of the ADMP effectiveness on the sludge solids. Sludge soundings within three feet of the ADMP centerline contacted the tank bottom indicating some sludge had been moved.
The ADMP was restarted on February 2, 2003 to begin the first cleaning cycle. For this discussion, a cleaning cycle consists of ADMP slurry operation followed by the transfer of slurry out of the tank. The Rotek assembly was operated throughout this cycle at approximately .5 rpm.

The ADMP was operated continuously for 10 days prior to the start of the transfer and continued during the transfer until the tank liquid level reached approximately 43 inches in Tank 18. Estimates predicted that the majority of waste would likely be removed during the first cleaning cycle (reference 1), therefore a substantial reduction in visible solids was expected to be observed when the transfer was complete. The target final liquid level in the tank was 7 inches; however the transfer was terminated at approximately 22 inches. Camera inspection during the transfer showed that the transfer pump was located in a “sludge well” such that the transfer pump suction became starved as the liquid level reached the peak of the sludge surrounding the pump. Sludge mounds were observed on the north and south sides of the tank, peaking at elevations of 23 and 25 inches respectively. The visible size of the two mounds indicated that the ADMP had not suspended as much sludge as expected. Conservative estimates show that as little as 3000 gallons of sludge may have been removed, versus an expected 30,000 gallons. A detailed estimate of the remaining sludge was not possible given the 22 inch liquid level.

An effort was initiated to lower the ADMP to its original design elevation (~23 inches) to improve pump discharge impingement on the sludge mounds because the elevation of the discharge nozzles was above the peak elevation of the two sludge mounds by approximately 16

inches. After allowing time for sludge solids to settle, an underwater camera was inserted into the tank and used to inspect below the ADMP inlet screen. A large tangled mass of steel tapes directly below the inlet screen was observed with an outside diameter roughly the same as the inlet screen. A "grappling" tool was designed, developed and built to snag the mass of steel tapes and remove them from below the ADMP. The ADMP was lifted to allow access below the pump inlet for moving the pile of steel tapes. Roughly 75% of the visible steel tapes were removed from beneath the ADMP. The mass of tapes was suspended from a nearby access riser with several strands extending into the waste. This activity was completed on March 7, 2003. The ADMP was then successfully lowered to the original design elevation. The orientation of the ADMP was adjusted to provide a NW to SE nozzle sweeping pattern to increase the contact time with the visible sludge mounds (reference work order 418008). The speed of the Rotek was lowered to .2 rpm to reduce discharge jet shearing losses and maximize the effective cleaning radius. In addition to these changes, a strategy to operate the ADMP indexed to a stationary position was developed to enhance breakup of the sludge mounds (reference 6). The ADMP was not originally equipped with the means to operate in varying fixed (indexed) positions, so equipment to hold the ADMP in the desired position had to be developed. A Bolton strike plate was positioned on the Rotek flexible receiver such that it would contact the south over-travel hard-stop, holding the ADMP in the desired position. The ADMP had to be shut down and the strike plate repositioned to change the discharge nozzle direction while operating in the "indexed" mode (reference work order 422421 and print P-PM-F-0241). A thermocouple was also installed through one of the center riser inspection ports to periodically monitor slurry temperature to confirm the temperature rise predicted by the Tank 18 thermal analysis. After operation resumed, an Unusual Occurrence (SR-WSRC-Ftank- 2003-0005) was declared when it was observed that the HLLCP had become entangled with the remaining hanging steel tapes. The main wad of tapes eventually fell back into the waste during subsequent ADMP operation, however no change in operation was observed. Tank liquid level during the remaining cleaning cycles was sixty to sixty five inches.

The second cleaning cycle was started on March 9, 2003. ADMP operation was initiated in a fixed (indexed) position, i.e. without Rotek operation. The ADMP was operated for approximately two days in each of 7 indexed positions as defined in reference 6. At this point, the ADMP had operated sufficiently to begin trending of the pump operating data. Analysis of this data showed the ADMP was losing load with operating time, i.e. motor amps reduce over time. A typical start up load would be ~250 – 290 amps at 1185 rpm, but would drop to ~210 amps within 1 hour of start up. Data from the 10 day cleaning cycle 1 operating run showed that the load settled to approximately 200 – 190 amps after approximately 56 hours of operation. "No-load" amps were measured to be approximately 166 amps at 1185 rpm during testing. See chart 1 attached. There were no other indications of problems with the ADMP operation. The drop in load was considered undesirable because it indicates the ADMP may not be doing as much work as expected, resulting in less cleaning effectiveness. The 72 hour endurance test run at TNX showed a steady load of ~266 amps. The Tank 18 Team developed a trouble-shooting matrix to identify potential causes, indicators, confirmation methods, and corrective actions. See tables 1 and 2 attached. The pump operating speed was lowered to reduce the potential for pump damage from cavitation or vortexing while the loss of pump load was being investigated. Initially the pump operating speed was lowered to 1050 rpm and later lowered to 900 rpm. There was no significant change in pump performance at the lower speeds, as measured by the pump current,

other than lower start up loads. The operating speed was subsequently increased back to 1050 rpm. An operating strategy of shutting the ADMP down when the amps dropped below a minimum load value specified by Engineering (typically 230 to 210 amps) was implemented. The ADMP would remain down for 1 to 4 hours prior to restarting. A minimum of one hour was specified by the motor vendor to allow cooling prior to restart. Experience showed that shutting down for longer than one hour would occasionally result in longer run-times before the minimum amp loading was reached.

Cleaning cycle 2 was completed in this fashion. The remaining cleaning cycles were also performed this way, however the ADMP speed was increased back to 1185 rpm. Although the ADMP cleaning effectiveness was still not as good as predicted, substantial improvement was noted based on remaining solids volume estimates between the first and second cleaning cycle. Another observation was noted as the tank level was pumped down. The operating strategy called for the ADMP to continue operation until the tank liquid level was within approximately one inch of the top of the discharge nozzles to keep solids in suspension as long as possible while transferring out of the tank. However, the amp readings would begin to excessively fluctuate (approximately +/- 70 to 80 amps) before reaching the top of the discharge nozzles. Field observations also noted an increase in noise and vibration at the ADMP mounting platform. The ADMP was shutdown as these symptoms occurred. For the last cleaning cycle (number6) the ADMP speed was lowered until the symptoms diminished to acceptable levels. This strategy extended ADMP operation by approximately 2 extra hours and allowed approximately

4 additional inches (14,000 gallons) of waste to be transferred with continuous mixing. A pump down test to within one inch of the discharge nozzles was performed during TNX testing in water. No erratic fluctuations in motor amperage were noted during this test.

The trouble-shooting matrix identified two general potential causes that could result in diminishing pump load: 1. Low fluid density, and 2. ADMP malfunction. Simple inspections to confirm proper pump operation (i.e. pump rotation, VFD output, seal gas flow rate, vibration, etc.) were performed. No malfunctions were identified. The focus shifted to potential causes of low or changing fluid density. Three potential causes of changing fluid density were considered: Air entrainment in the liquid builds up in the pump casing; pump cavitation; and lower than estimated liquid density. Low liquid density was essentially ruled out because this should cause a consistently low amp load.

Additional SRS pumping expertise was consulted in addition to the pump manufacturer. In general, the consensus was that the pump behavior was more indicative of gradual air build-up in the pump casing, or a gradual loss of slurry density, rather than obstructing the inlet or pump cavitation. Obstructing the pump inlet generally results in a sudden loss of pump load as cavitation begins, in addition to other observable characteristics such as noise and excessive vibration. The observations and data suggest that the phenomenon is more gradual in development and is repeatable at varying pump speeds and tank levels. A scoping calculation was performed to analyze the potential for vortices in the liquid reaching the pump inlet, but the calculation showed that this was unlikely for the operating tank levels. The column seal gas supply was considered as a potential source of air that could somehow get into the pump casing, but the gas flow rate was found to be the same as the flow rate during TNX testing, and therefore was also considered unlikely. Some foaming on the liquid surface was noticed during the first cleaning cycle pump down, dissipating rapidly when the ADMP was shutdown. Considerable

surface foaming was noted during the pump down phase of the second cleaning cycle, but did not dissipate after the ADMP was shutdown.

While slurry foaming is still considered a possibility for the root cause of the loss in amp loading, investigation of this phenomenon is long term and could not be implemented to meet project schedule requirements. No near term options for addressing foaming or air entrainment of the slurry were identified other than the expected dilution of the waste with each planned water addition, so the team focus shifted to addressing other potential causes such as debris or trash restricting the pump inlet.

Two activities were initiated to address the possibility of the pump inlet screen becoming clogged with waste debris. The first activity was to flush the pump inlet screen and the area around and below the inlet screen with a high velocity water lance. The intent was to move sludge solids and debris from the vicinity of the pump inlet and into the effective zone of the pump discharge nozzles. A water lance was designed and built for connection to a high pressure (800 – 1000 psig) subcontractor supplied water pump. This activity was performed between the end of cleaning cycle 3 and the start of cleaning cycle 4 when the pump inlet was partially visible using a camera inside the tank (tank liquid level was approximately 7 inches).

The hydro-lance consisted of 1-1/2 inch schedule 160 carbon steel pipe nozzled down to two horizontal discharge, opposing ¼ inch schedule 80 pipes. The hydro-lance was designed, built and tested to ASME B31.3 requirements (reference work order 434423). Because of very limited access at the tank top, the hydro-lance discharge could only be rotated around its vertical axis and lifted up and down, but could not reach around the perimeter of the pump inlet. To maximize access to the pump inlet screen, the turntable (Rotek) was operated as the hydro-lance cleaned the top and sides of the screen. The hydro-lance was also lowered to the tank bottom and rotated such that the discharge would sweep from side to side to move any debris and solids from below the pump inlet screen. Five 1500-gallon batches were discharged into the tank. The hydro-lance discharged at approximately 60 – 65 gallons per minute at 800 psig supply pressure. A second hydro-lance operation was performed during cleaning cycle number 4. ADMP performance after hydro-lancing was essentially unchanged. ADMP operation during cleaning cycle number 4 was also impacted by failure of a pressure switch that triggers the low column pressure interlock causing numerous unplanned shutdowns of the pump. Inspection of the switch showed that oil used to separate the sensed media from the switch had leaked from its chamber causing the switch to trip at a higher set point. No more unplanned ADMP shutdowns from the low column pressure interlock occurred after the switch was replaced.

The second activity was to increase the open area of the inlet screen. Camera inspections during the steel tape pile removal and hydro-lancing showed that a significant amount of debris and solids were accumulating on the screen. Increasing the open area required physically modifying the inlet screen after it had been in use and exposed to radioactive waste. The ADMP inlet screen was originally manufactured with approximately ¾ inch rectangular openings. The manufacturer was asked to design and supply a screen with smaller openings (approximately 5/16 diameter) to prevent steel tapes from entering the pump inlet and becoming tangled with the impeller. Final TNX testing (in water) was performed with the modified screen. To enlarge the open surface area of the screen, a method to cut away part of the screen and replace it with screen containing larger openings was developed. To provide containment and minimize exposure to the

workers, the pump was lifted until the inlet screen was inside the shielded tank chamber located on top of the center riser plug (reference P-PM-F-0240). Work on the screen was performed through the 12 inch by 12 inch chamber access panel. The vertical sections of the inlet screen were removed using air-arc welding technology. The removed sections were replaced with patches removed from the original inlet screen. This modification was estimated to increase the open screen area by approximately 20%. The inlet screen was modified June 6, 2003. The top of the inlet screen was observed to have embedded material even after flushing. Only a marginal improvement in pump performance was noted after the modification. Based on the operating performance after the hydro-lancing and the screen modification, screen pluggage was no longer considered to be a primary cause of the reduced pump loading.
No further changes were made to the operation or configuration of the ADMP through the completion of waste removal. After the final pump down using the dewatering pump, a significant amount of debris (steel tapes, hose, grappling tool, etc.) was observed to be near the vicinity of the ADMP inlet screen.

The primary consequences of the initial discharge elevation and reducing amp load phenomena can be seen in the total water consumption required to remove the bulk of the sludge solids and the overall duration of the waste removal campaign. Reference 1 predicted a total of approximately 475k gallons of water would be required in four cleaning cycles. The total campaign actually used more than 782k gallons of water spread over six cleaning cycles. The total ADMP operating time was very similar to that predicted in reference 1, forty days. However, of the approximately 40 operating days, roughly half of that time was at or above 210 amps, and only six days above 230 amps. See figure 2.

Summary and Conclusion

The ADMP successfully suspended sludge solids for removal from Tank 18 to meet current tank closure criteria. An evaluation of the ADMP operations, testing, and vendor supplied information identified probable causes for the unpredicted pump loading (reference 10).

References

M. Hubbard, “Tank 18 Waste Removal Operating Plan”, U-ESR-F-00014, Rev. 2, November 2002.

John R. Gordon, “Review of ADMP Operating History”, SRT-RSE-2001-00313, December 18, 2001.

C.L. Sharp and D.B. Stefanko, “TNX/HLW Long Shaft Pumps 1995-2000”, WSRC-TR-2001-00313,June 2001.

“HLW Tank 18 Waste Removal Systems Engineering Evaluation”, WSRC-RP-2001-00024, May 22, 2001.

“Tank 18 ADMP Pre-Installation Test at TNX (U)”, HLWD-F-322, August 27, 2002.

"Tank 18 Waste Removal Operating Plan, Revision 2 Addendum", CST-FTK-2003-00018 Rev. 0, March 6, 2003.

J.L. Thomas, "Characterization of Tank 18 Residual Waste", U-TR-F-00005, October 15, 2003.

"Advanced Design Mixer Pump Reliability Testing", IWT-OP-114.

"Industrial Wastewater Closure Module for the High Level waste Tank 18 System", WSRC-TR-2003-00273.

J.J. Purohit, "ADMP Performance Evaluation", M-TR-F-00011, Rev. 0, April 12, 2004.

2.2.5.2 SRS Folding Crawler

1. Name of technology: SRS Folding Crawler
2. Description of technology:

   In FY2000, the Savannah River National Laboratory (SRNL) completed developmental testing of a mobile-platform system designed to aid the removal of the tank heel wastes. Beyond standard chemical and radiation material compatibility requirements; the system was designed to deploy through a twenty-three inch riser opening, carry a one-hundred pound payload, and traverse a ½" horizontal discontinuity through sludge and supernate. The crawler system was designed around commercially available stainless steel, deep-lug tread Inuktun tracks mated to a collapsible chassis that unfolded in the tank. Heel mobilization was performed through a water spray system. Deployment of the system employed a two-step process to assemble the system on the tank floor. Initially the collapsed frame was lowered through the access riser, landed on the tank floor and unfolded. The water spray system was lowered separately and landed on mating dowel pins on the unfolded crawler chassis.

   The system was tested in a series of performance tests at the SRNL Tank Wall Mock-up facility and the TNX Full Tank Test Facility. Performance was evaluated based on the criteria that the crawler and water system be capable of clearing a 23" access riser opening and was capable of being mated together remotely. Performance was also measured on the ability of the crawler to manage the tether, two control cables and one-hundred feet of 2-½"inch diameter fire hose while maneuvering around the tank floor.

3. Which tank was it deployed:

   The SRS Folding Crawler was intended for deployment in SRS Tank 19, but was never deployed.

4. How well did it work:

   The crawler and water system worked well in mock-ups and performance testing.

5. What technical evaluation criteria were used to determine whether it worked:

   Evaluation criteria were assessed based on the ability of the crawler and water system to fit within a 23" riser separately, and to be remotely assembled. The crawler was assessed on its ability to pull the 100' tether and fire hose. The water system was evaluated against the ability to mobilize simulant as well as stability of the vehicle during water system operation.

6. What is the relationship between the technology and characteristics of the waste:

   Waste material was accumulating in areas not directly accessible from overhead spraying nozzles (behind concentrations of coils and other in-tank obstructions).

7. What were the lessons learned:

   Formal lessons learned were not documented for this particular work.

8. Key documentation:

   WSRC-MS-2000-00497, Development and Testing of a Mobile Platform for Tank Remediation, T.A Nance, R. F. Fogle, M. Collins, D. Krementz (electronically available at http://www.osti.gov/bridge/)

2.2.5.3 SRS Digital and Wide Angle Photography

1. Name of technology:

   SRS Digital and Wide Angle Photography

2. Description of technology:

   In FY2005, the Savannah River National Laboratory (SRNL) in conjunction with SRS Tank Farms Engineering recommended a comprehensive program to replace aging film technologies with digital technologies to perform annulus inspections at the SRS Tank Farms. The proposed program was based on the growing dependence on inspection technology countered by limited resource availability which in turn demanded efficiencies in operations. SRNL provided a conceptual design centered on commercially available digital photography technologies. The program also proposed pursuit of digital image evaluation. All SRS Wide Angle photographs (WAP) and Direct Photography (DP's) planned annual inspections were made with a digital camera in 2007. The WAP rig has been used for over 800 inspections and has performed very well with no significant issues. Quality is no less than equal to prior film based technologies (see Figure 1). The inspections performed in 2007 with the DP rig were a challenge and made completing the annual schedule difficult. The lighting system and some minor modifications to the deployment system for the DP rig were proposed in 2008. The changes made have eliminated the problems encountered in 2007 and all inspections (~40 entries, >1200 photographs) performed to date have been very good with no related problems. SRNL is planning delivery of a second spare system before the end of FY08.

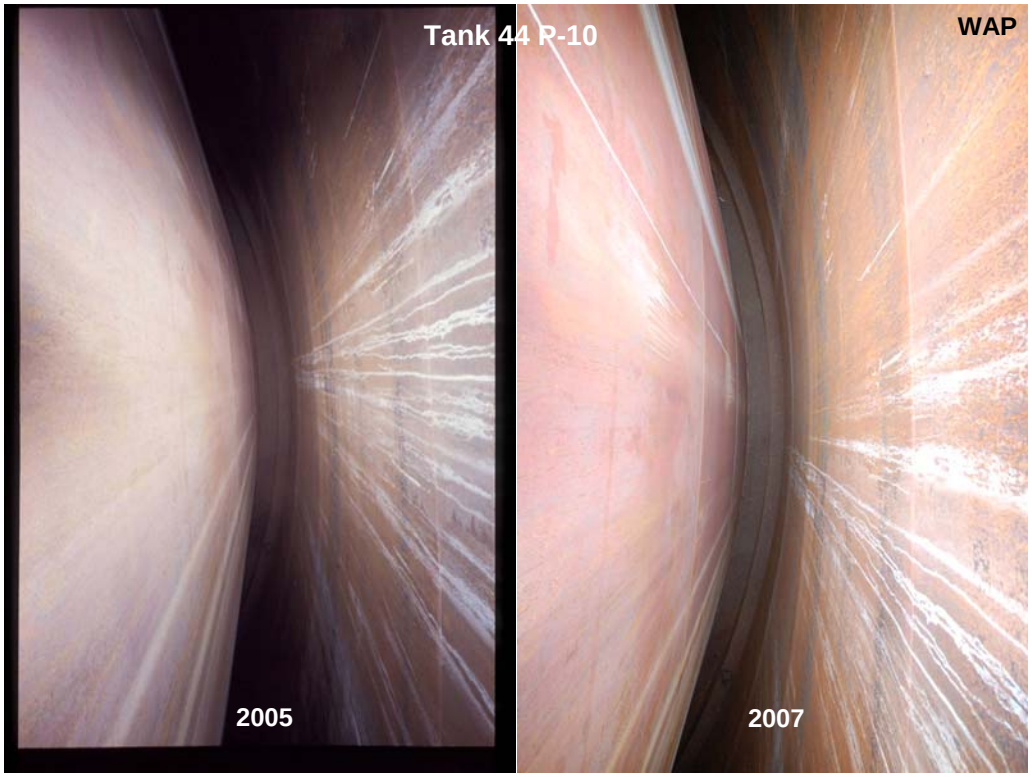

Figure 2.9. Comparison of tank annulus picture (looking down) using conventional film photography (left) and digital Wide Angle Photography (right)

3. Which tank was it deployed:

   The SRS Digital Photography (DP) and Wide Angle Photography (WAP) rigs have been deployed in multiple tank annuli at SRS.

4. How well did it work:

   The systems are continuing to work very well following some changes early in FY08 to the lighting system and deployment system. An image evaluation system has not yet been pursued.

5. What technical evaluation criteria were used to determine whether it worked :

   Evaluation of the system's performance was based solely on ease of operation and quality of inspection pictures.

6. What is the relationship between the technology and characteristics of the waste:

   Very little relationship to the characteristics of the waste, if any (multiple tank annulus inspections with very minimal interaction with waste).

7. What were the lessons learned:

   Formal lessons learned were not documented for this particular work.

8. Key documentation:

a. SRNL-IES-2005-00064, SRNL Memorandum titled "Transition of LWDP Inspection Program from Film-Based to Digital Technology" from R.W. Poland (SRNL), G.D. Thaxton, D.C. Blair to D.B. Little, December 2, 2005

b. SRNL-EES-2008-00010, SRNL Memorandum titled "Recommended Technology Upgrades to the LWDP Inspection Program" from M.C. Hall, R.K. O'Donnell, D. Krementz, J.B. Battle to E.J. Freed, and C.E. Blair, April 7, 2008

2.2.5.4 Annulus Inspection / Cleaning Wall Crawler

The Tank 6 Annulus Inspection / Cleaning Wall Crawler has magnetic wheels for driving along the tank wall within the annulus. It is used to travel through the tank annulus and inspect leak sites and the internals of the ventilation duct. The entire crawler is designed to be able to access the annulus through a five inch riser. The crawler is driven just above the ventilation duct, where a boom arm can be lifted to place a pan/tilt camera the desired distance from the inner wall. At that point the entire boom arm assembly can be lowered on a track to insert the camera into the ventilation duct. The crawler is also designed to remove the salt nodules from the leak sites. Cleaning is performed by blasting the nodules with water through one of two spray nozzles. Mechanical cleaning is achieved with a brush located at the lower end of the crawler. Fabrication of the wall crawler has been completed and the technology has been successfully demonstrated to the customer.

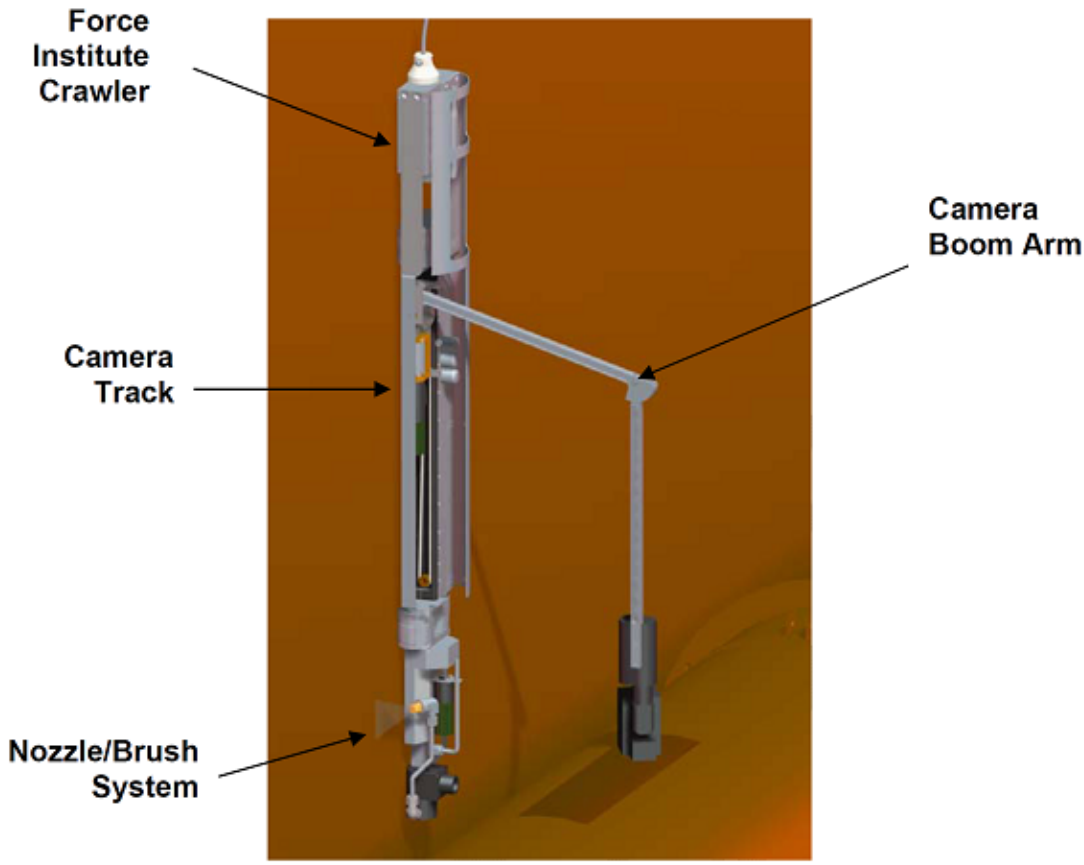

Figure 2.10. Magnetic Wall Crawler for Tank Inspection

2.2.5.5 Waste Tank Turbidity Meter

The waste tank turbidity meter uses a fixed distance (gap) between a photoconductive cell and a light bulb. The light bulb is connected to a 6 volt lantern battery and the photoconductive cell is connected to an ohm meter. The resistance of the photoconductive cell increases as the light transmission from the light bulb is attenuated by the presence of the tank waste between the gaps. The supernate liquid is fairly transparent and offers little attenuation compared to the more opaque insoluble solids. The turbidity meter is slowly lowered into the waste tank until the liquid-solid interface level is detected by a large sudden increase in resistance.

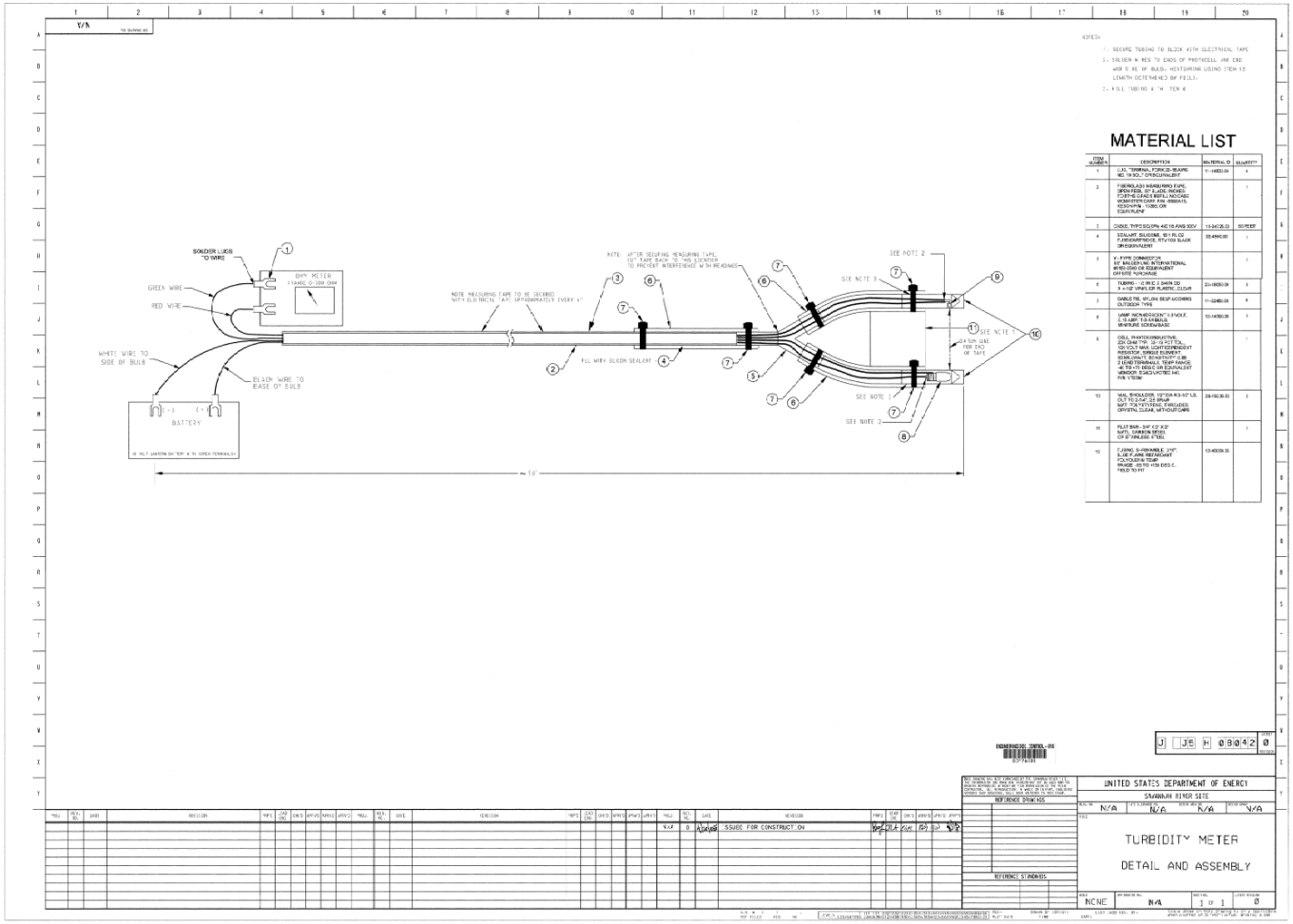

Figure 2.11. Waste Turbidity Meter Diagram

2.2.6 Fernald Site Input

2.2.6.1 Fernald Silos Slurry Pumping

Fernald had two million gallon silos almost the same dimensions as the Hanford tanks. The Diameter was nominally 80 ft in internal diameter, and the working height was approximately 30 ft. The material in the silos was ore where the U had been leached out during the war effort and immediately after WW II. This material was finely ground ore from the leaching process, and was quite abrasive due to the silica and the Al in the wastes. The retrieval was done during 2004, the testing for this system was done in 2003 and 2004. A full scale diameter tank was built and surrogate was installed for sluicing testing. The test system was 80 ft long, 8 ft deep and 12 ft wide. This served as a slice of the silo for testing. A report on this testing is available.

The pump selected was a Haselton vertical centrifugal pump with a cable to lower it into the sludge as it was sluiced. This pump had a 200 hp motor, and a 4 inch discharge line which was tied to a hose. During initial sluicing the pump was in a full recycle mode where it acted similarly to a slurry pump in that the discharge was in close proximity to the suction. By running in this method the waste was slurried into a homogeneous mix. When this was accomplished, the discharge was redirected into the receiver tank and the tank was emptied. The actual silo waste was transferred into four new 700,000 gallon tanks for storage before processing.

The primary lesson learned from this work was the full scale testing was critical to the success. We found several of the instruments that had been specified failed during slurry and transfer pumping. The coriolis meter failed quickly. The testing and the analysis of the results were useful to the designers for final design. These test reports are available.

2.2.6.2 Sluicing Transfer.

The Fernald system used the vertical Haselton pump for transfer from the silos to the storage tanks and from the storage tanks to the processing tanks. In both of these systems sluicing was used to drive the slurry to the pump suction. The pump was hung from a cable and was lowered as the material was sluiced so it always was located in the area of highest slurry concentration percent solids. The pump worked satisfactorily at up to 40 percent solids. Typically the slurry was transferred at 5 to 10% solids. These Haselton pumps worked well during the life of the retrieval operations, and no spares were used. The testing system was described in the slurry pumping section.

Testing of the system was critical. A test rig on the full scale system was added to test the surrogates before hot operation. This testing was essential for the eventual operation.

2.2.6.3 Pneumatic Retrieval

Silo 3 material was a dry fluffy powder which we believe is similar to the calcine waste stored at INL. This material was pneumatically retrieved from the silo. This was done with a pneumatic blower to provide the suction and a series of hoses and a hose management system where the operators would move

the end of the hose to the solids where the solids were conveyed into the hose. The hose was a 4 inch hose that was called an elephant snout. The solids were collected in a hopper with an off gas system. The waste was then loaded into soft sided bags and shipped to disposal.

A test system was fabricated in a machine shop close to the Fernald site in Cincinnati. This testing was necessary to allow processing to occur. The processing operated without incident and the silo was cleared ahead of schedule. We had a backup system using a remotely controlled front end loader, but it was not required.

2.2.6.4 Crawler

Fernald used a remotely controlled crawler to remove the silo walls when the silos were emptied. A commercially available remote system was used for demonstration of the empty Silo 4. This worked well.

A crawler was procured for use in retrieval from silo 3. It was not required because the pneumatic system worked well. See the section on pneumatic processing.

2.2.6.5 Tanks Mixing and Slurrying

Supernate was used to slurry the material in the 700,000 gallon hold tanks. The supernate from the system was pumped into a quite storage tank and allowed to settle out the solids. The clear liquor was then decanted off and used as slurry sluice water. When the operators did not allow the solids to separate adequately the sluicing efficiency decreased a lot. Fernald was very hesitant to add more water. Therefore, a process instruction was written requiring at least 12 hours before being allowed to be reused for sluicing. Once this was done, the sluicing performed better.

2.2.6.6 Recirculation System

When the Fernald ore residuals were mined from the storage tank it was transported by the Haselton pump to the EIMCO vertical clarifier. The slurry was pumped in at about 5 % solids. The clarifier is tunable and was operated at 50% solids in the discharge.

The material is pumped from the clarifier to a batching tank to feed into the grout mixer. The original design was for a positive displacement pump. The material is a refined ore, and is quite abrasive. A surrogate was developed and the system was pilot tested at ORNL. The selected positive displacement pump was a rotary lobe pump. The pump failed within 8 hours of startup. A total of four positive displacement pumps were tested and all failed. This problem was solved by adding a recirculation line to the clarifier and the system was operated by a hardened Haselton centrifugal pump. A side stream is used to feed into one of the three batching tanks. This system worked well throughout the campaign of a year operation.

The vertical clarifier was new technology for DOE. This worked quite well, and caused no upset conditions.

2.2.6.7 Transfer Out of Tanks

Fernald used a vertical horizontal pump supplied by Haselton as the means of removal of the ore from the silo or from the storage tank. In both cases the pump was mounted on a cable and was lowered into the waste. A sluicing nozzle with high volume recirculated supernate was used to sluice the solids and the slurry flowed to the low point at the pump suction. The operators used the remote nozzle controller to knock down the solids and move it to the pump suction. Once operation began the operators quickly became proficient in keeping the solids moving to the pump suction. To ensure acceptable operation the operators tried to keep the sludge to about 5% solids. The pump could pump much higher concentrations, but the consistency of the feed could not be controlled.

The sluicing water was the decanted liquor from the settled solids. Fernald found it necessary to feed into a quite tank to allow settling. Management did not allow make up water, since this added to the load that had to be treated at the end of the project. Whenever the operators tried to reuse water too quickly without allowing sufficient settling the system would not sluice new solids. A minimum set time of at least 12 hours was implemented, and when this was followed the sluicing performed satisfactorily.

The design engineers and operating engineers who operated this for Fernald are available and have been used for consulting for DOE applications. If there is interest in this these engineers can be brought in for consultations.

2.2.6.8 Characterization and Monitoring

Silos Mapping

The ore after the U was removed was placed in large silos very similar to the DOE million gallon tanks. The material was pumped into the silo and the motive water was recycled. However the material did not load into a smooth surface, and there was significant variation of the surface. This material had residual radium in the ore and the radium decayed into radon. This created a massive radon release, which was unacceptable to the EPA and the state of Ohio. DOE decided to install a layer of diatomaceous earth to cut down the emission of the radon by trapping it in the DE. In order to do this mapping of the surface was required. The DOE Robotics Crosscut program developed a program to take photographs and mapped the system. The DE was then fed into the silos to fit this curve. The average depth was approximately 5 feet.

Waste Tank Mapping

During removal the waste it was important to have a daily determination of the rate of removal to report to management. The operating engineers used drawings of the silo and the storage tanks to determine the height of various landmarks in the tank and daily drew a map of the surface based on the height as determined by three in tank cameras located at approximately 120 degrees around the tank. From this, any hills of material that had not been mined became the area of focus for the next day operation in order to try to remove the solids while trying to keep a level surface. The engineers became proficient in this daily mapping and the data was reported daily to DOE.

Several automatic methods were researched, but were not implemented because the simple system was adequate.

2.2.7 Experience in the U.K.

2.2.7.1 Pumps / Mixers

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

The Three Pump Plate was a device for cleaning skips and consisted of three centrifugal pumps mounted on a metal plate. The plate sat on top of the skip to be cleaned and two of the pumps used to stir up the contents of the skip and the third used to transfer the resulting slurry to a settling chamber. Use of the machine declined with the introduction of the Rotary Skip Wash.

The Pond Bay Sludge Retrieval facility was installed in the mid 1990s. It was intended to consist of four Warman submersible pumps in the main bay and a submersible pump and ejector in the withdrawal well. However, installation of the two north pumps in the main bay was not possible due to pipe work running beneath the access holes. The system was operated despite this between 1995 and 2000. At the time it was believed that the system was operating successfully and transferring sludge, but subsequent sonar surveys revealed that less than 5 m^3 of sludge had been removed from the bay.

In 1992, 180 m^3 of sludge was successfully transferred from the main settling chambers to a processing building. The equipment used consisted of a large and small desludging head, which both operated on the principle of mobilizing a local area of sludge within the head using water jets and transferring the slurry away using a Grindex transfer pump within the head.

The water ducts of the Windscale Piles were successfully desludged in 1995 using an ROV, attached to which was a submersible pump and suction hose. The retrieved sludge was transferred via an underwater pipeline to a skip in the main pond. There were problems with the reliability of the ROV during initial operations in Pile 1 water duct, which required the ROV to be retrieved and redeployed several times.

Jet Pumps UK Ltd has been adopted since 2007 for the company formerly known as Genflo Ltd. The original jet pump designer Tony Wakefield has retired from the company. The Wakefield design of Jet pumps has been used in several nuclear applications, for example for emptying sludge from tanks at Sellafield. Supernatant liquid was used as motive fluid to prevent the increase of secondary waste. The jet-pump is a natural fit to sludge emptying and dredging applications because there are no moving parts. Another important feature is the all-stainless construction with no plastics or elastomers to be attacked by nitric acid or radiation. In addition to nuclear and dredging credentials, the company have developed submersible systems for air circulation in mines, sewage clearance, the transport of chemicals in powder form, sand cleaning, beach replenishment schemes, by passing, environmental clean up and many other applications.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield – Multiple installations

How Well Did It Work?

Varied

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Generally full scale simulant based trials followed by plant trials

What is the Relationship Between the Technology and Characteristics of the Waste (ie Waste Properties, Rheology, and Other Physical Behaviors)?

Knowledge of characteristics of wastes was limited. Technology chosen to avoid specific characteristics of waste where possible.

What Were the Lessons Learned (High Level – No More Than 2-3)?

See the main description

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

More detailed reports may be available on each system.

2.2.7.2 Jet Ballast / Pulse Jet mixers

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

At Sellafield there are a large number of tanks with installed jet ballast, similar in design and size to the pulse jet mixer systems in RPP-WTP. The Sellafield tanks were designed and built from around the 1970's through to the present day.

Typical systems comprise of outer or peripheral tubes with a central tube. These are immersed in the tank liquor and controlled is sequence. They use compressed air to periodically blow the liquid from each tube out through a nozzle at the base. This creates a jet of liquid within the tank, agitating the tank content. The jet ballast tubes require a minimum amount of liquid to be present in the tube in order to operate correctly. Jet Ballasts, unlike more recent designs of PJM, they do not have a ‘suck’ phase of the cycle, and rely of the static liquor within the tube.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield – Multiple installations

How Well Did It Work?

30 years + operational experience. Typically work very well.

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Full scale simulant based trials

What is the Relationship Between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

Systems are very dependent on the characteristics of the waste and the design of the system, especially settling and behavior of the solids with respect to agglomeration.

What Were the Lessons Learned (High Level – No More Than 2-3)?

Small changes in properties of the wastes or operational performance of the plant can affect the overall performance of the system.

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

A number of reports have been published on the systems in journals and conferences over the years. For example, 7th World Congress in Chemical Engineering 'The influence of yield stress on the re-suspension properties of nuclear waste simulants' LJ Mason, SR Biggs, TP Tinsley, D McKendrick

2.2.7.3 Flygt Mixer

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

ITT Flygt claim to be the first to manufacture a submersible wastewater pump (1956). Used extensively in the mining, ore dressing, steel milling and ash handling industries. Several Flygt pumps have been supplied to Sellafield but have been unable to find any details.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield

How Well Did It Work?

Unknown

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Unknown

What is the Relationship Between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

Unknown

What Were the Lessons Learned (High Level – No More Than 2-3)?

Unknown

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

Unknown

2.2.7.4 Air Sparging

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

Air lifts have been used in the HA storage tanks to re-circulate concentrated HA Liquor for many years attracting the maximum TEI and TRL score. They provide a very vigorous agitation of the tank contents and in conjunction with the jet ballast system completely homogenize the tank content. Contaminated air is treated by a system of scrubbers

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield

How Well Did It Work?

Worked successfully on full tanks for almost 50 years

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Unknown

What is the Relationship Between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

Properties of settling behavior is fundamental to the performance of the system

What Were the Lessons Learned (High Level – No More Than 2-3)?

Changes in settling behavior due to phenomena such as agglomeration can have a big impact on performance. Operation of air lifts can lead to foaming when organics get entraining in the liquor.

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

Same as Jet Ballast

2.2.7.5 In Tank arm based and crawler systems

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

Robotic arm and crawler devices to deploy equipment within restricted areas. Custom design systems have successfully deployed within small diameter pipes e.g. 3", within tanks and other cells.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield

How Well Did It Work?

Successful

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Did the equipment deploy to the correct position

What is the Relationship Between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

N/A

What Were the Lessons Learned (High Level – No More Than 2-3)?

Understanding of restrictions within the cell is important

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

Unknown

2.2.7.6 High pressure nozzles / lances

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

A whole variety of propriety blockage clearing equipment using high pressure jets on the ends of umbilical hoses is available on the market. These can either be pushed along the pipe by means of a stiff umbilical or some self propel themselves along the pipe by means of rearward facing jets. If sufficient

space exists, jet washing / lancing of wide areas can be achieved e.g. in ponds or basins. A whole range of jet sizes and shapes is available, and these devices are widely used in the sewerage industry, but again they tend to cater mostly for larger diameter pipes, e.g. 3 inch and above.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield – multiple locations

How Well Did It Work?

Generally very successful. Tend to be used for specific blockage clearing activities as 'one offs.' Also used to remove cements and grouts from the outside of vessels or equipment.

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Removal of the blockage.

What is the Relationship Between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

Properties of the solid, especially its re-suspension properties are of primary importance.

What Were the Lessons Learned (High Level – No More Than 2-3)?

Deployment to the point of blockage has typically been the bigger challenge. Management and disposal of effluent also can cause difficulties.

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

Unknown

2.2.7.7 Mechanical Unblocking

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

Mechanical unblocking techniques consist of simple wire spring devices similar to drain clearing units for domestic use. These range from about 6 to 20mm diameter with power drives to continuously rotate and feed the spring forward/backwards through the blockage. Various cleaning tools can be attached to the front of the wire spring depending upon the nature of the material. A prototype unit using this principle has been developed to be remotely operated for clearing blockages in 12 mm diameter pipes for Sellafield. If there are no existing engineered entry points into the line then it would be necessary to cut into the line and return it to its original integrity following removal of the blockage. The operation of cutting into an active line will have dose implications to operators and decontamination of the unblocking equipment will be necessary.

Ultra High Pressure Ultrasonics

Brief description of technology (ie no more than 1-2 paragraphs)

A technique employing the power of sound waves to dislodge blockage material is High Power Ultrasound (HPU), alternatively known as Pulse Power Technology (PPT). This uses a High Voltage spark discharge between two submerged electrodes (similar to a car spark plug) to create a localized region of very high pressure waves which break up and disperse blockages. Originally developed for pipe decontamination, this method is now being developed for blockage clearance and in laboratory trials has shown some very promising results. The simplicity of the principle means that the spark head itself can be made very small, giving it an advantage in situations involving small bore pipes or difficult access. Care has to be taken in any environments where there may be hydrogen present, due to the potential ignition capability of the spark principle.

2.2.7.8 Ultrasonics

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

If access to the outside surface of the pipe where it is blocked is possible, then another technique which can be considered is the use of ultrasonics. A transducer is applied to the outside surface of the pipe and transmits sufficient energy through the pipe wall to disturb the blockage. The drawback of this technique is that it requires accurate determination of the location of the blockage and unrestricted access to the pipe exterior at the position of the blockage. Good 'coupling' between the transducer and pipe wall to ensure efficient energy transmission is also required.

A test programme was performed by Sellafield R&D in 1996 into the performance of ultrasonics in the clearance of pipe blockages. It was demonstrated that the technique showed potential for blockage clearance on site, but there were a number of issues which required further investigation, such as the effect of the ultrasound on welds and methods of remote deployment.

2.2.7.9 High Pressure Washing

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

There is a wide range of blockage clearing technologies using high pressure jets on the ends of umbilical hoses. These may be pushed along the pipe by means of a stiff umbilical or some self propel themselves along the pipe by means of rearward facing jets. A whole range of jet sizes and shapes is available, and these devices are widely used in the sewerage industry, however they tend to be most suitable for larger diameter pipes, e.g. 3 inch and above.

Water jetting was considered to be the most suitable technique for removal of a blockage in a liquor sampling pipe. Due to the low cost of the rodding and nozzle equipment, it was proposed to dispose of these items following the unblocking operation. The pipeline contained a posting port, which would allow deployment of the hose and nozzle without having to cut into the pipe and removal of the effluent via a peristaltic pump.

2.2.7.10 Chemical Cleaning / Dissolution

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

The use of chemical reagents to dissolve solids is a possible blockage removal technique. Remotely operated umbilical included a wash hose which was used to supply a low pressure chemical wash to the site of the blockage in order to dissolve it. Several of these units are kept in permanent storage in case they are needed for future use, and could be modified to suit different applications if required. This equipment was remotely operated to successfully clear an 8 m long blockage of solids approximately 25 m down a 50 mm NB pipe inside a hot cell.

Introducing liquor non-intrusively through an existing engineered wash point has the advantage of minimal operator dose uptake and the integrity of the pipeline is not altered. However, an intrusive method of introducing liquor, which involves cutting into the pipe and using a hose, allows higher pressures to be used. For any chemical dissolution technique, consideration must be given to the acceptability of the reagent used to downstream plants and processes. For example, the suitability of several reagents was investigated for the clearance of a blockage in the THORP HA steam strip and ammonium carbonate solution was considered to be the most suitable, but further work on the downstream effects in the plant was also recommended.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield

How Well Did It Work?

Successful at clearing the blockage

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Unknown

What is the Relationship Between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

Technique relies of the chemical agent being able to dissolve all or part of the solid, cause the blockage to breakdown.

What Were the Lessons Learned (High Level – No More Than 2-3)?

The process of dissolving i.e. how vigorous the ‘fizzing’ is, can be important to the success of breaking up the blockage.

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

Unknown

2.2.7.11 Spray Washing / Scabbling

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

See high pressure nozzles

2.2.7.12 Microwave / Laser Scabbling

Brief description of technology (ie no more than 1-2 paragraphs)

The system consists of a high power generator, magnetron, waveguide, and head. The microwaves are guided along the waveguide (essentially a steel duct) where they are then concentrated at the head. The head is deliberately placed close to the concrete where the microwaves are forced into the concrete. The microwaves essentially heat up the free moisture in the concrete which causes a violent explosion which breaks the concrete surface.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

The UK (Harwell) attempted to trial a microwave system for decontaminating the partially decommissioned LIDO enriched uranium thermal swimming pool reactor in 1993. The system failed to scabble the concrete. The microwave generator was selected solely on the basis that it was available.

R&D work in the early 1990s and the more recent work by Nottingham university show that microwaves can effectively scabble concrete. The Nottingham trials used Sellafield concrete (approx 30 year old) for the trials.

How Well Did It Work?

Mixed

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

The important material parameters are: the amount of free moisture content present in the concrete, the dielectric constant (materials ability to STORE the electromagnetic energy), and the loss factor (materials ability to convert the stored energy to HEAT). The microwave generators power and frequency are important.

What is the Relationship Between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

Unknown

What Were the Lessons Learned (High Level – No More Than 2-3)?

The above, particularly the UK LIDO work, shows that the key lesson is a clear overall understanding of how microwaves interact with concrete is needed before attempting to specify a piece of equipment and determining whether a certain type of concrete can be effectively scabbled or not.

Is There Key Documentation That is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

1. Concrete Breaking Using Microwave Energy-A Report on Investigations-The University of Nottingham/National Centre for Industrial Microwave Processing. Professor Sam Kingman.
2. Nuclear Science and Technology, Large scale demonstration of dismantling techniques under realistic conditions on the LIDO biological shield. EUR 17888, ISSN 1018-5593 Dated 1998 but trials done in 1993.

2.2.7.13 Characterization and Monitoring

Brief Description of Technology (ie No More Than 1-2 Paragraphs)

Flexible fibrescopes are commonly used to inspect pipes and then sample if solids are present. Usual sizes are of the order of 7 to 10mm diameter with lengths usually up to 15m. Smaller diameter units e.g. a 1.5 mm dia endoscope, have been successfully deployed down a 3 mm ID pipe.

A whole variety of propriety pipe inspection cameras are available on the market. These can either be pushed along the pipe by means of a stiff umbilical or alternatively some are mounted on self-powered drive units, which propel themselves along the length of the pipe. However these propriety camera units tend to be for use in larger pipes, e.g. 3 inch and above. For smaller pipes, either endoscopes have to be considered (with their limited max length of around 15 m) or alternatively Nexia Solutions has in the past developed its own in pipe cameras to suit the particular task at hand. For example a system was developed to deploy the camera head up to 30 m down a 50 mm NB pipe with approx. 10 ninety deg bends and included the requirement to steer it through a vessel.
Laser Induced Breakdown Spectroscopy (LIBS) to determine the elemental composition of materials. A LIBS fibre could be included in a fiberscope sample tube.

As blockage location is the first essential step in clearing it, we have also considered other non intrusive means of determining exactly where it lies within the pipe, e.g. Radscan radiation mapping, ultrasonic or radiographic analysis and thermal imaging of the exterior surface of the pipe if accessible. In addition sonic ranging, where a sonic pulse is sent down the pipe and reflected off the blockage to reveal its location can be considered if one end of the pipe is open and accessible

The Less-Intrusive Imaging and Process Sensing capability is concerned with the provision of suitable technology solutions to allow non-invasive process imaging or sensing. The ability to collate information regarding the internals of a process, storage vessel or container without having to gain physical access provides significant safety and cost benefits. Also, because of the nature of material found within Nuclear facilities, less-intrusive imaging and process sensing has a wide variety of applications including the internal imaging of process units, storage vessels, waste containers as well as active samples. The capability has provided the following solutions:

- Development of cosmic ray muon technology for the imaging of large and dense objects (e.g. silos).
- Development of a tennis ball sized deployable radiation mapping device (RadBall).
- Non-intrusive gamma transmission enabled level detection on the Vitrification Test Rig (VTR).
- Electrical Resistance Tomographic (ERT) Vortex detection in a nuclear crystallisation reactor (Thorp).
- X-ray enabled Post Irradiation Examination (PIE) of mock up fuel pins
- Real time 3-dimensional imaging of flow fields through an ion exchange cartridge.
- Development of non electrical radiation detectors for highly active plants.

Which Site, Tank(s), Basins, Vaults, Etc Was It Deployed?

Sellafield

How Well Did It Work?

Successful

What Technical Evaluation Criteria Were Used to Determine Whether It Worked (No More Than 2-3)?

Size of the pipe and requirement of the inspection / characterization

What is the Relationship between the Technology and Characteristics of the Waste (Ie Waste Properties, Rheology, and Other Physical Behaviors)?

N/A

What Were the Lessons Learned (High Level – No More Than 2-3)?

Breaking containment can be a significant challenge, along with disposal of any contaminated equipment. Potential for getting device stuck needs to be address – use of emergency pull cables can be considered

Is There Key Documentation that is Available Which Provides Technical Information Regarding the Technology, Especially Lessons Learned and Recommendations?

Unknown

2.3 Slurry Transport

2.3.1 Slurry Transport Breakout Session

A workshop breakout session was held on Wednesday (1/16/08) and Thursday (1/17/08). The attendees of this breakout session are listed in the table below. The discussions were focused on DOE-EM cleanup projects at each site where slurry transport was used in the past or is going to be used in the future. Notes on this information exchange were taken in the form of meeting minutes and are provided in Appendix YYY of this report.

Table 2.3. Slurry Transport Breakout Session Participants

Name	Affiliation	Wed (1/16/08)	Thurs (1/17/08)
David Roelant	FIU	X	X
Robert Cooke	Paterson & Cooke	X	X
Mark N Hall	Hanford WTP	X	X
David Harbottle	University of Leeds	X	X
Dan Krementz	SRNL	X	X
Gary Hofferber	Hanford K-Basin	X	X
Robb A Burk	WTP	X	X
Nigel Heywood	BHRG	X	X
John Conner	CHG	X	X
Don Hallbom	PSI Canada	X	X
Franz Nigl	PNNL	X	X
Ben Lewis	ORNL	X	X
Adam Poloski	PNNL	X	X
Steve Krahn	DOE-EM	X	
Tommy Caldwell	SRS		X
Kris Thomas	DOE-ORP		X
Steve Strand	Dow Chemical		X
Dominic Rhodes	Nexia Solutions		X
Tom Michener	PNNL		X

In addition to the discussion component of the breakout session, the group had knowledge of several past and on-going projects related to several DOE sites. Written submissions were requested from the breakout session attendees. The written components are intended to be a compilation of slurry transport information significant to the DOE-EM cleanup process.

Information on the Fernald site activities were provided by Phil McGinnis. The Fernald Closure project was supported by a DOE technology assistance effort. Mr. McGinnis led a team of engineers and chemists from ORNL, PNNL, INL, Mississippi State University, North Dakota EERC, and industry to test a piping flow loop. Testing in an existing flow loop at ORNL of the design proved the surrogate (provided by PNNL) would not stay suspended and caused line pluggage. Equipment evaluation tests were conducted using several pumps, and slurry instrumentation. Testing results were documented in a series of reports culminating in a final report. The executive summary of the report is found in Appendix 1. The full version of the report can be sent electronically upon request.

Information on three projects at the Savannah River Site was provided for DWPF, Saltstone, and Vacuum retrieval and transport (contribution from). The DWFP discussion includes two summaries on SRS tank farm sludge rheology from Terri Fellinger and Jonathan Bricker. Erich Hansen provides a summary of transport issues related to the Saltstone process. This includes a discussion about transport of grout mixtures using large scale peristaltic pumps. Lastly, Dan Krementz provided a summary of three phase flow transport issues that have arisen when using a vacuum retrieval and pneumatic transport process at SRS. These contributions can be found in Appendix 2.

Rick Demmer provided a written summary of Idaho National Laboratory slurry transport issues in Appendix 3. The summary includes issues associated with the transport of both INL liquid waste slurries as well as pneumatic transport of calcined waste.

John Connor and Mark Hall provided a written summary of transport issues from the Hanford tank farms in Appendix 4. The summary provides information on the pumps, and instrumentation that have been used at the Hanford site. Line plugging and unplugging issues at Hanford are discussed. The K-Basin cleanup project at Hanford also faced several challenging transport issues. Gary Hofferber provided information on a set of hose-in-hose simulant transport tests that were conducted in support of this project. The lessons learned from these tests were compiled in a conference paper. The abstract from this paper is provided in Appendix 4. Lastly, Robb Burk provided information that the Hanford Waste Treatment Plant has commissioned a set of pipeline transport tests with Newtonian and non-Newtonian fluids. The results from these tests are presented in a draft PNNL report, WTP-RPT-175 Rev A.

Lastly, Florida International University has recently completed a pipeline unplugging test with technology from NuVision. Test reports have been compiled into a report with David Reolant as the principal investigator. The executive summary form this report is provided in Appendix 5.

2.3.2 Slurry Transport Breakout Session Meeting Minutes

The meeting minutes from the slurry transport breakout session are provided in this appendix. Each bulleted item reflects a discussion point that was made in during the sessions. Each bullet has an initial listed in parenthesis to provide an indication on who made the discussion point. A listing of the session attendees and initials is provided below.

Table 2.4. Slurry Transport Breakout Session Participant Initials

Initials	Name	Affiliation
DRoe	David Roelant	FIU
RC	Robert Cooke	Paterson & Cooke
MH	Mark N Hall	Hanford WTP
DHar	David Harbottle	University of Leeds
DK	Dan Krementz	SRNL
GH	Gary Hofferber	Hanford K-Basin
RB	Robb A Burk	WTP
NH	Nigel Heywood	BHRG
JC	John Conner	CHG
DHal	Don Hallbom	PSI Canada
FN	Franz Nigl	PNNL
BL	Ben Lewis	ORNL
AP	Adam Poloski	PNNL
SK	Steve Krahn	DOE-EM
TC	Tommy Caldwell	SRS
KT	Kris Thomas	DOE-ORP
SS	Steve Strand	Dow Chemical
DRho	Dominic Rhodes	Nexia Solutions
TM	Tom Michener	PNNL

2.3.2.1 Hanford Tank Farm Discussion Notes (1/16/08)

- Cross site transfers (MH)
  - 6 pipelines exist
  - 4 have been plugged
  - Possibility of building new lines
- Transfer pump from tank to booster pump to receive tank waste followed by flush (MH)
  - ~three pipe volumes of water used during flushing
- Phosphate gels are primary concern for plugging (MH)
- Hose in hose system (MH)
  - Easy to handle with forklift to move
  - River Bend Co makes the hoses
  - Concrete barricades can be used for shielding

- Deign methods include (MH)
  - Use a wide variety of equations including Durand and Oroskar-Turain
  - Pick highest estimated value
- Design criteria (MH)
  - Design transfer below pressure rating of the piping
  - Jumpers are often the weak link (<200 psi)
  - Planned to move to 400 psi limitation
  - Complex geometries such as PUREX connectors are often used
  - Flow, pressure gradient, and temperature requirements are established
  - No heat traced lines are utilized
  - Tank waste mobilization by mixer pump heats waste to about 100ºF
  - Transfer to cold transfer lines causes precipitation usually in valve pit or in valves
  - Erosion rate is a key concern when operating at high velocities
- Average particle density used in the design of 2900 kg/m^3 from RPP-9805 document for a particle size cut of 74 microns (MH)
- Viscosity typically ranges from 6 to 9 cP based on modified Thomas relation in RPP-9805 (MH)
- In mining industry a density distribution usually exists with a small quantity of dense material (DHal)
- Deposition will be based on highest density material (DHal)
- 3-4 ft/sec seems low for a design velocity for yield stress materials (DHal)
- Should base design on a laminar to turbulent transition region which is affected by yield stress (DHal)
- Transferring at low pressure gradient in laminar flow will result in transport problems (RC)
- Under high pressure gradient conditions laminar flow can be stable due to the formation of a sliding bed of material that is pushed through the pipe by the pressure gradient (RC)
- Need to examine all min and max rheological/ and slurry property conditions (RC)
- Perform calculations at these extremes (RC)
- Only using a turbulent flow design equation will result in problems (RC)
- Plugged transfer lines were due to precipitation (JC)
- WTP will accept HLW sludge and LAW supernate from the tank farms (JC)
- Nearly all WTP piping is 3” schedule 40 (RB)
- A pipeline transport test facility has been built at PNNL to perform tests with WTP simulants & process conditions (RB)
- Pump Amps will be monitored during transfers (RB)

- Coriolis meters (RB)
  - Accuracy issues with bubbles
  - Recommend coupling them with Magnetic flow meters
- New coriolis meters are much more tolerant (NH)
  - Straight tube designs are more conducive to slurry transport
- Ultrasonic sensors can be utilized to measure bulk density (NH)
- Ultrasonic flow meters are typically inaccurate for slurry flows (NH)

2.3.2.2 Hanford K-Basin Discussion Notes (1/16/08)

- K-basin sludge consists of insoluble U, Fe, and sand (GH)
- 17 feet of water in basin (GH)
- Grating is over the basin allowing access to nearly all areas (GH)
- East basin contains fuel in open bottom containers (GH)
  - 3 foot in length, 2.5 inched diameter, 1/4 inch thickness, aluminum clad
  - Fuel corroded in basin producing metal oxide corrosion products
- Fuel came from N-reactor (GH)
- Sludge has been pumped to metal containers for holding at West basin (GH)
- Density ranges between 19 g/cc (for U metal), 9-12 g/cc (for U oxides), and 2.5 g/cc (for sand) (GH)
- Size ranges from submicron to < 1/4 inch sieve (GH)
- Criticality issues do not exist due to neutron poisons (GH)
- SEM images indicate the presence of agglomerated U-oxides (GH)
- Design equation used was the Durand equation (GH)
- 17 ft/sec needed to suspend large, high density particles in water (GH)
- 1 ¼" ID hose used to transfer 2200 ft from East to West basin (GH)
- Two submerged pumps were used for the transfer (GH)
- Four booster pumps were used each with 75 hp capacity (GH)
- Averaged 2 vol% particles during the transfer (GH)
- Utilized existing basin water for transfer to minimize volume/capacity limitations
- Goal is to D&D east basin (GH)
- 700 psi pressure gradient measured during the transfer (GH)

2.3.2.3 Hanford WTP Discussion Notes (1/16/08)

- Currently 30% to 50% through piping design on WTP (RB)
- Much of the equipment is already out for procurement (RB)
- Piping specs are complete (RB)
- In November 2006 a design guide for line sizing and slurry transfer systems was issued (RB)
  - Design was based on RPP-9805 particulate data
  - Results in 4 ft/sec design velocity
  - 4 ft/sec was applied to all piping regardless of diameter, this has been corrected
- Plant appears to be under instrumented but P&IDs will be further refined as design proceeds (RB)
- PNNL has created a newer estimate for particle size and density (WTP-RPP-RPT-153; RB)
- 25 kPa was largest shear strength measurement of tank waste and will be very challenging to restart (RB)
- Under normal operations, the piping system is designed for 30 Pa yield stress, 30 cP plastic viscosity Bingham plastic parameters (RB)

2.3.2.4 Hanford WTP Discussion Notes (1/17/08)

- Mark Hall was author on design guide (RB)
  - Oroskar-Turain equation was the basis
  - Two equations are used 1) empirical; 2) semi-empirical that can be extrapolated
  - Has viscosity term from Thomas relation
  - Separate the fine fraction (<74 micron; 200 mesh) from coarse fraction (>74 micron)
  - Fine fraction used for viscosity adjustment
  - Coarse fraction properties used for settling fraction
  - Have broad spectrum of waste so used D_{95} 210 micron 2.9 SpGr particles as coarse fraction basis
  - Approximately 80% fines; 20% coarse
  - A document, ICD-19, discussed the waste feed acceptance criteria for WTP
- A pipeline transport test facility has been built at PNNL to perform tests with WTP simulants & process conditions (RB)
  - ¼ way through critical velocity testing
- For test matrix (RB)
  - Three size fractions: 10, 50, 100 microns
  - Three densities: 2.5, 4, 8 g/cc for glass, alumina and stainless steel
  - Three carrier fluid rheological properties using kaolin as fines

- Simulate small amounts of settling material in a non-settling slurry
- Currently tested 110 micron glass beads in water
- As velocity decreases, the pressure gradient increases indicating the formation of a blockage
- "Dunes" were observed to form the pipe
- Bed was flushed at 150 to 100 psig
- Initial flush plugged the system completely
- Further tests will be performed with kaolin clay/water as a carrier fluid

- SRC model assumes uniform distribution of solids in turbulent flow (RC)
- SRC basis is D_{50} of particles above 75 microns for coarse fraction (RC)
- Bulk density of sludge is ~1.5-1.7 gm/cc (MH)
- Sludge consists of aggregates which are a complex combination of all materials in the aggregate (MH)
- Do agglomerates survive the transfer or are the dispersed? (MH)
- Do agglomerates recombine under low shear conditions at the end of the transfer? (MH)
- Sonication of samples for PSD measurements are not representative of the shear rates observed during pumping (MH)'
- Amount of energy input per unit volume is the key for deagglomeration as opposed to the shear rate (RC)
- Test system will use electrical resistance tomography (ERT) to image the pipe cross-section (RB)
- Dominic Rhodes has additional papers on UK experiences with ERT systems (DRho)
  - Multi-plane system allow to see down the axis of the pipe
  - ERT system has been installed on a UK Pu plant (THORP)
  - Data has been published in the open literature
  - Can now deploy the systems in metal walled vessels rather than plastic, non-conductive systems.
  - Also looking at acoustic devices, ultrasonic, clamp-on for slurry imaging

2.3.2.5 SRS Discussion Notes (1/17/08)

- Currently three processes with slurry transport at SRS (TC, DK)
  - Tank farm operations
  - Salt Waste Processing Facility (SWPF)
  - Saltstone facility

- For the tank farm operations (TC, DK)
  - PUREX process created two primary waste types
    - Iron based wastes
    - Aluminum based waste
  - Can fill tanks with water and mix until uniform
  - Kaolin clay is used as a waste surrogate
    - Must let the clay shear stabilize before use
  - used to for pressure gradient calculations
    - Hanks-Dadia model under turbulent flow
    - Buckingham equation used under laminar flow
  - Vacuum retrieval system used to empty the bottom of the tanks
    - Combination of slurry transport and pneumatic transport
    - Three phase flow exists in the regime
      - Air
      - Solids
      - Liquids
    - Flow analysis under these conditions is not well understood
- DWPF (TC, DK)
  - Erich Hansen and Terri Fellinger are the best contacts for this facility
  - Material properties are much different in the facility than in waste tanks due to
    - Shearing during retrieval
    - Transfer in pipelines
    - Dilution due to washing
    - DWPF treatment and frit addition
- Saltstone (TC, DK)
  - Erich Hansen is the best point of contact for this facility
  - Large scale peristaltic pumps are used

2.3.2.6 ORNL Discussion Notes (1/17/08)

- Ben Lewis is the point of contact (BL)
- Ben presented the transfer system results in a presentation at the workshop earlier in the day (BL)
  - Presentation focused on design of a cross-site transfer line

2.3.2.7 INL Discussion Notes (1/17/08)

- Rick Demmer is the point of contact (AP)
- Rick presented the transfer system results in a presentation at the workshop earlier in the day (AP)
  - Presentation focused on design sludge retrieval system for tank farm closure

2.3.2.8 Fernald Discussion Notes (1/17/08)

- Phil McGinnis is the point of contact (AP)
- Phil indicated that the slurry transport work at ORNL had occurred in the 2003-2006 timeframe. Reports have been published on this topic and will be discussed in the final report. (AP)

2.3.2.9 Pipeline Plugging and Unplugging Discussion Notes (1/17/08)

- Cooling of transfer lines at Hanford causes precipitation and forms chemical plugs (MH)
- Heat tracing of old lines has failed (MH)
- Sometimes pipes were preheated with hot water prior to transfers (MH)
- Cannot runs steam in double wall pipe annulus due to leak detector issues (MH)
- Lines are not insulated (MH)
- AEA/NuVision has tested a wave action system at FIU to unplug lines (DRho)
  - Two additional systems will be tested shortly
    - Sonic energy focused on a plug
    - High pressure water jets
- UK has used chemical dissolution with ammonium carbonate (DRho)
- Heel jets (high pressure jets placed on pipe elbows) have been used to unplug vertical pipe sections in the past at Hanford (MH)
- Vibration has also been effective in removing plugs from pipes (MH)
- K-Basin has mechanical plugs form in the past (GH)
  - Sludge vacuuming system
  - During filling operation the velocity of the flow was reduced and a plug formed
  - Plug was unplugged with a mechanical router
- Fernald transfer pump plugged during a loss of power at cold testing (BL)
- Bechtel has a design guide for plug removal at WTP (MH)

2.3.2.10 Industry Feedback Notes (1/17/08)

- Are we making the problem more complex than it really is? (DHal)
  - 1.5 to 2.5 m/sec is typical design flow rate for long pipe transfers

  - When transferring in small batches the shutdown procedure usually causes the problems
- There is currently interest in three-phase flow for low-water consuming transfers (Dhal)
  - a professor at Wollongong University is studying this type of flow
- Foam transfer has been used in the UK (DRho)
  - Foam degrades over time to minimize tank volume requirements
- SRC model is the current state of the art model for coarse particle transport (RC, NH)
  - Currently 2005 version is the most recent
  - Need to take course to get access to software
- Oroskar-Turain equation may not be reliable in certain ranges (NH)
  - Need to consider different correlations
  - Look at original paper to verify applicability
- Swirling flow in pipelines may be a useful technology to prevent bed formation (DRho)
  - Ribs are located on the inside of pipe walls
- Make sure that variable speed pumps are used; fixed speed pumps are old technology (NH)
- For flushing of process lines gradually increase flush velocity for 30 sec at startup (RC)
  - Flush velocity should be slightly greater than particle critical velocity
  - Do not wait to flush the lines; the bed can setup

2.3.2.11 Pump Technology Notes (1/17/08)

- WTP current design used Wilfley chemical pumps (RB)
  - Vertical motor, centrifugal pump design
  - 150-200 gpm flowrate
  - Not a “slurry” pump
- The Wilfley pump is not API 610 compliant and should not be selected for this application; several other pump manufacturers produce slurry pumps for these applications (NH)
- WTP pumps being re-specified to Hastelloy for high temperature HNO_3 service (RB)
- Polymer coatings used in conventional pumps may be of concern due to radiation damage (AP)
- Hazleton pump used at K-Basin (GH)
  - Hardened 5% Ni Fe alloy used
  - Specialty pumps limited to 1/8” particles
  - Used for floor vacuuming
  - capable of 100-200 gpm flow rates
  - designed for 60 gpm or 6 ft/sec in 2” ID hose

- Hanford tank farms use the following pumps (MH)
  - Centrifugal pumps for boosters
  - Mixer pumps are fluidic design
  - Moyno/Seepex pumps for retrieval
  - No aluminum or Teflon materials of construction
- SRS (TC, DK)
  - Have tried many pump designs
  - Submersible well pumps
  - Double diagram
  - Steam jet eductors
  - Centrifugal
  - Peristaltic pump used in saltstone facility
- Reverse flow diverters offer a slurry pump capability with no moving parts (MH)
- At K-basin a cheap submersible chemical pump was modified for slurry service by adding a ¼" gasket to separate the rotor form the stator and provide particle clearances (RB)
- For K-Basin transfer pump a handmade custom pump was used
  - Centrifugal, high head, high flow pump
  - Designed for particulate with curved vane impeller
  - 15 to 18 ft/sec at 70-80 gpm and 425 psi
- Peristaltic pumps have been used in industry for the transport of yellow-cake (RC)
- Jet pump coupled on the suction side of a centrifugal pump will steepen pump curve and prevent plugging in suction line (NH)
- Torr pump discussed in Nigel Haywood's presentation may be useful for sludge mobilization and retrieval
  - Design is used in the nuclear industry in the UK
  - Less dilution that jet pump
  - Originally designed in Russia to pickup sand

2.3.2.12 Miscellaneous:

Nigel Heywood indicated that a new generation of straight-tube Coriolis meters is available. Manufacturers mentioned were Kroner and Yokagawa. These straight-tube meters are much better with gas than the previous u-tube Coriolis meters (such as used in 242-A and I believe in closure/retrieval applications). I have found the Coriolis meter for 242-A to be excellent for process control – no problems with accuracy.

Also, two-phase flow (solids and liquids) can result in over-prediction of mass flow, solids and SpG if the flow is sliding-bed. The solids have a longer residence time in the device than the liquids.

Ultrasonic and radiometric sensors are available for flow and density. Doppler is notoriously inaccurate.

Mark Hall indicated that WTP is using Oroskar-Turian correlation for slurry critical velocity with a 30% safety margin added. This is based on RPP-5346 evaluation and is what we use in TF as well. However, they go into quite a bit more detail on the particle size and viscosity calculations and selection of parameters such as solids density. Their method will be documented in ICD-19 (draft) and a couple of WTP design guides which he indicated would be shared.

Nigel Heywood, when covering critical velocity correlations in the short-course lectures, made no mention of Oroskar-Turian critical velocity correlation (apparently there are 100s of them). However, he did indicate in the break-out session that the practice of evaluating a number of different correlations and adding a safety factor (as reportedly practiced by TF, WTP, K-Basins, and SRS) seemed sound. The latest/greatest correlation is from Saskatchewan Research Council, and you have to go to their course to get their software. Lead for SRC is Dr. Gillies (Shook and Gillies was one of the earlier correlations), who was scheduled to attend the workshop, but did not.

Robert Cooke (consultant who is apparently involved in the SRC course) stressed that 'you have to understand your system', i.e., particle size/PSD, densities, and the like.

David Harbottle (Ph.D. candidate from University of Leeds) indicated that their research showed that taking individual particle size/crystal density is conservative if there is agglomeration.

Cooke indicated that the amount of energy, not the rate of shear, is what affects particulate breakdown.

Savannah River (Tom Caldwell/Dan Krementz) indicated that for laminar flow they use the Buckingham equation and for turbulent flow they use some of the equations given in the short course. They have not experienced settling in pipes in SRS TF. Viscosity closely matches NaOH curves.

Oak Ridge (Ben Lewis): Their cross-site line plugged a couple of times and was unplugged by back-pulsing with water.

Dave Roelant (FIU) will provide a write-up of their latest research on pipeline unplugging, including experience from NuVision (ex-AEA, British researchers). Techniques include upslope beach erosion, a high-pressure, low-volume water lance, a sonic-boom technique from Hydrokinetics, Inc., and a rotating nozzle with a water jet directed backwards which roto-roots through the pipe (need somewhere for the water to drain though).

Dominic Rhodes (Nexia Solutions, ex-BNFL) suggested using foam to clear lines if volume is an issue. Over time the foam would break down to a minimal volume. They have used this technique.

Dominic Rhodes indicates that they are using Electrical Resistance Tomography (ERT) for solids monitoring in a production plant. Further details in his keynote talk Friday.

Robert Cooke indicated that if solids settle in a pipeline it typically forms ridges (dunes). Flushes need to be ramped up slowly (say over 30 sec) to avoid plugging the pipe completely. Starting at a lower flush rate will erode the solids by layers.

2.3.3 Slurry Transport Session Site Information Input

2.3.3.1 Fernald Site Information

EVALUATION OF SELECTED PUMPS AND INSTRUMENTATION FOR SUPPORT OF THE FERNALD ACCELERATED WASTE RETRIEVAL PROJECT

EXECUTIVE SUMMARY

The Silos 1 and 2 remediation plan at the Fernald Environmental Management Project (FEMP) near Cincinnati, Ohio, calls for almost 10,000 tons of radium-bearing, low-level waste material to be transferred to interim storage tanks, processed by concentrating the solids, mixing them with cement and other additives to form a grout, and solidifying the grout in containers. The containers will then be shipped off-site for final disposal. In support of this project, two slurry-pumping test-loop systems were used to evaluate the performance and wear of four proposed pump types: centrifugal, progressive-cavity, rotary lobe, and peristaltic. The test loops were also used to evaluate a variety of system-monitoring instruments and other equipment proposed for use on the full-scale system at FEMP. The test loops simulated proposed process operations using a surrogate material. This report summarizes all testing conducted at the Diagnostic Instrumentation and Analysis Laboratory (DIAL) at Mississippi State University and Oak Ridge National Laboratory (ORNL).

The Moyno progressive-cavity pump failed after approximately 78 hr of operation when the seal ruptured. Results from the Alfa Laval rotary lobe pump test conducted show that the pump was severely worn by the abrasive slurry, significantly compromising its performance. While the Watson-Marlow Bredel peristaltic pump performed well during most tests, its catastrophic failure because of severe plugging in the suction line and significant loss of pump lubricant makes it a poor choice for use in the full-scale processing system at Fernald since the pump failure occurred at a solids loading (50 wt%) that the pump would be likely to see at either the clarifier underflow or batch mixer slurry feed locations.

The testing showed that the Goulds or Hazleton centrifugal pumps could be considered for use in the full-scale processing system at Fernald. The Goulds centrifugal pump pumped slurries of K-65 waste with solids loadings of up to 30 wt% including Bentogrout without any noticeable change in performance. The viscosities of K-65 slurries (even those containing up to 6% Bentogrout) are not expected to exceed 100 cP if the total solids loading is kept to 45 wt% or less.

The magmeter accurately measures flow rate when properly sized, and slurry foaming is minimized. Because the foaming was an artifact of the rapid recirculation that occurred in the hot loop, it would not be expected to occur in the full-scale system at Fernald. Coriolis meters can accurately measure both flow rate and density/solids loading when foaming is eliminated and the meters remain in service no longer than 4 weeks. Such a replacement schedule would minimize the effects of erosion by the abrasive slurry. Coriolis meters cannot be used in environments with foam, however.

The microwave densitometer readings were precise and accurate at higher solids loadings. Implementation of a new calibration based on K-65 waste would likely improve the instrument's

accuracy over the full range of solids loadings expected in the full-scale system at Fernald. The Canberra gamma densitometer/attenuation meter accurately measures both slurry density and radium activity, although it may be affected by the presence of foam in the slurry as well as vibration from the piping. It also requires that the detector be cooled to cryogenic temperatures, requiring frequent filling with liquid nitrogen or the use of a mechanical cooler. Isolok samplers can be used to collect representative slurry samples. Material containment difficulties were solved by the insertion of a Delrin® sleeve in the discharge pipe of the Isolok sampler.

The process by which the technical assistance team conducted testing and evaluation generated the opportunity for quality decision making of the equipment required for the full-scale processing system at Fernald. A summary of the recommendations based on the evaluations and comparisons conducted regarding pumps and instrumentation that could be used in the full-scale processing system at Fernald follows: The testing showed that the Goulds Model JC or the Hazleton Type CBE centrifugal pumps could be considered for use in the Fernald system. Magnetic flowmeters can be used to accurately determine flow rates in the full-scale system at Fernald, while slurry solids loading can be accurately monitored on-line using microwave densitometers that have been calibrated for K-65. For locations at which both solids loading and radium concentrations must be measured, gamma densitometers/attenuation meters could be used. Isolok samplers can be used to collect representative slurry samples for sample analysis using off-line analytical techniques. Finally, based on the testing results, Everlasting diverter valves, vortice ells, and 5-D elbows would be expected to perform well if installed in the full-scale system at Fernald.

2.3.3.2 Savannah River Site Information

Fluid Flow at the Defense Waste Processing Facility (DWPF)

At the Savannah River Site, a batch of sludge feed is retrieved, prepared and combined into one of the million gallon feed tanks for transfer to the Defense Waste Processing Facility (DWPF). A typical sludge batch is approximately 500,000 gallons. The DWPF receives sludge from the Tank Farm, typically on a weekly basis, for subsequent processing. Approximately 6000-8000 gallons of sludge at typically 15-19 wt% total solids is transferred to the DWPF to make up each process batch. In the DWPF, acid is added to the sludge and the slurry is concentrated to approximately 20-25 wt% total solids in the Sludge Receipt and Adjustment Tank (SRAT). This adjusted slurry is then transferred to the Slurry Mix Evaporator (SME) where it is mixed with glass frit and concentrated further to approximately 45-50 wt% total solids. The slurry is sampled and analyzed to determine acceptability and then transferred to the Melter Feed Tank (MFT), which provides continuous feeding to the joule heated melter. The resulting glass is then poured into stainless steel canisters.

DWPF is currently processing the fifth batch of sludge. A unique set of chemical and physical properties have been noted for each batch processed to date. Although some of the noted physical properties impacted processing times, no transfer line pluggages have been encountered in the facility. Some of the physical property observations from the DWPF are as follows:

- Sludge Batch 1B – The sludge slurry was “tacky” and stuck to sample bottles in the analytical laboratory initially impacting analytical turnaround times.
- Sludge Batch 2 – Air entrainment/foaminess in the sludge slurry impacted SRAT and SME processing times and the ability to transfer between preparation tanks.

- Sludge Batch 4 (current batch) – Higher yield stress of the melter feed and potential feed loop control issues are impacting the ability to feed the melter.

Physical measurements are made of sludge batches prior to transfer to DWPF. These measurements are determined using a Haake M5/RV30 rotoviscometer. The M5/RV30 is a Searle sensor system, where the bob rotates and the cup is fixed. The torque and rotational speed of the bob are measured. The shear stress is determined from the torque measurement and is independent of rheological properties. Conditions that impact the measured torque are; slip (material does not properly adhere to the rotor or cup), phase separation (buildup of liquid layer on rotor), sedimentation (particles settling out of the shearing zone), homogeneous sample (void of air), lack of sample (gap not filled), excess sample (primarily impacts rheologically thin fluids), completely filling up the void below the bob (air buffer that is now filled with fluid) and Taylor vortices. The first five items yield lower stresses and the last three add additional stresses. The shear rate is geometrically determined using the equations of change (continuity & motion) and is that for a Newtonian fluid. This assumption also assumes that the flow field is fully developed and the flow is laminar. The shear rate can be calculated for non-Newtonian fluid using the measured data and fitting this data to the rheological model or corrected as recommended by Darby. In either case, for shear thinning non-Newtonian fluids, typical of SRS sludge wastes, the corrected shear rates are greater than their corresponding Newtonian shear rates, resulting in a thinner fluid. The bob typically used for measuring tank sludge is the MV1 rotor. Prior to performing the measurements, the rotor and cups are inspected for physical damage. The torque/speed sensors and temperature bath are verified for functional operability using a bob/cup combination with a National Institute of Standards and Technology (NIST) traceable Newtonian oil standard. The resulting flow curves are then fitted as a Newtonian fluid and this calculated viscosity must be within + 10% of the reported NIST viscosity at a given temperature for the system to be considered functionally operable.

In general, slurries processed in the DWPF are non-Newtonian, exhibiting rate dependent behavior and a non-zero yield stress. However, rheological properties can vary significantly depending on the sludge batch itself as well as other incoming waste streams. Feed from the salt disposition program is just being introduced into the facility this year. These are low solids streams but can impact the overall properties of the slurry in the DWPF. The rheological properties of sludge slurries are primarily affected by weight percent total solids, with secondary affects arising from particle size and shape and particle dynamics, such as agglomeration. The consistency of the slurry received at DWPF is an exponential function of weight percent total solids especially for HM or high aluminum sludges. Thus, at higher total solids loading, a small change in weight percent total solids can results in a large change in rheological properties. The proportion of insoluble and soluble solids is also significant. This property is primarily controlled by the degree of washing performed on the sludge during preparation of the sludge batch. Extensive washing can improve processing in DWPF by reducing off-gas quantities but can dramatically impact rheological properties of the sludge due to the larger fraction of insoluble solids and potential to introduce air entrainment.

Within the DWPF processing cells, the primary factors that influence rheological properties are total solids and pH. Thus, the extent of feed concentration is controlled along with the acid adjustment process, which drives the pH. The influence of the chemical composition on rheological properties is less understood. In fact simulant slurry compositions with similar elemental and anion species as radioactive slurry often yield very different rheological properties. Studies are ongoing in this area and include the impact of particle size, heat treatment, and other physical changes.

DWPF has a robust design and operating windows to accommodate the varying slurry transport properties. The operating ranges are defined below:

Table 2.5. Slurry Transport Parameters for DWPF

	Total solids (wt %)	Yield Stress (Pa)	Consistency (Pa-sec)
Sludge Slurry	13 – 19	2.5 – 10.0	0.004 – 0.012
SRAT	18 – 25	1.5 – 5.0	0.005 – 0.012
SME	40 – 50	2.5 – 15.0	0.010 – 0.040

Pumps within the DWPF are the primary equipment of concern in relation to rheological properties. Transfer pump design evolved over many years. The pump is typically above the tank liquid in the plenum region of the tank, with the pump impeller supported by a cantilever shaft with a radial bearing located outside the tank. The pump is designed to suction lift the contents of the process tank via a suction pipe extended into the tank bottom. To prime the pump, process water is used to till the impeller cavity. While the DWPF pumps share the same basic design there are some differences. For example, for tank sampling, sample pumps have a recirculation line which allows contents to be recycled into the tank of origin.

OVERVIEW OF THE KEY PARAMETERS, TECHNICAL APPROACHES, AND SUPPORT COMPONENTS RELATED TO THE TRANSPORT AND MIXING OF SLUDGE SLURRIES IN THE DEFENSE WASTE PROCESSING FACILITY

J.M. Bricker, J.R. Neuville, and T.L. Fellinger
Defense Waste Processing Facility
Savannah River Site
Aiken, SC 29808

Abstract

The primary function of Savannah River Site's (SRS) Defense Waste Processing Facility (DWPF) is to process and immobilize high level radioactive waste (HLW) into a durable borosilicate glass for safe, permanent geological storage. To date, the DWPF has processed approximately 2.5 million gallons of HLW. To create a batch of feed for the DWPF, several tanks of sludge slurry are retrieved, prepared, and combined into one of the million gallon DWPF feed tanks. A batch of feed nominally consists of 500,000 gallons. After a sludge batch has been prepared and qualified for processing in the DWPF, ~ 6000 to 8000 gallons of sludge slurry is transported on a weekly basis from HLW Concentration, Storage and Transfer (CST) facilities to the Sludge Receipt & Adjustment Tank (SRAT) located in the 221-S facility. In the SRAT, the sludge slurry is chemically adjusted, concentrated, and sampled. The adjusted sludge slurry is then transferred to the Slurry Mix Evaporator (SME) where it is mixed with glass forming frit, and concentrated further. The concentrated slurry/frit mixture is sampled prior to transfer to the Melter Feed Tank (MFT), which provides continuous feed to a joule heated melter. The resulting molten glass is then poured into stainless steel canisters. Each of the steps described above has a number of supporting unit operations (including mixers, pumps, heat exchangers, ventilation and purge systems, filters, etc.) which are required to process the feed. The intent of this paper is to define key parameters of interest, technical approaches, and support components associated with slurry transport from a vitrification plant processing point of view.

Saltstone Process (Erich Hansen)

The Saltstone Processing Facility (SPF) is designed to blend low level waste (salt solutions) with a mixture of dry cementitious material to make a flowable grout that can be pumped to the concrete vaults for final deposition. The salt solution is supplied by Tank 50 and processed at SPF via the salt feed tank. The salt solution is analyzed for density and wt% total solids. Bench scale grout mixtures are made from the actual salt solutions to determine the water to premix mass ratio (W/P) that will provide a grout with a gel times of greater than 25 minutes, bleed solution of less than 2 volume percent after 3 days of curing, and a set (less than 2mm) time between 3 to 6 days. Rheological characterization of the recommended grout is not a requirement for processing.

The SPF dry feed system consists of four storage silos, one containing Class F fly ash, one containing Grade 100 Blast Furnace Slag, and another containing Class I or II Portland cement. The fourth silo is for backup. The present composition of dry materials, call premix, for processing the waste, on a mass basis

is 10% Portland cement, 45% Slag, and 45% fly ash. The dry materials are weighted in a single mixing hopper, air blended and pneumatically transported to the day hopper. The day hopper feeds two screw augers, where the feed rate is controlled by loss of weight in the day hopper. The premix and salt solution is blended using a READCO mixer, which is a twin shaft, co-rotating mixer, deigned to mix dry materials with liquids on a continuous basis. The internal mixing blade configuration, in the READCO mixer, is parallel to the direction of flow, maximizing the applied shear for mixing. The internal mixing blade configuration can be changed to impact shear and throughput. The day hopper feeds the READCO mixer at 35 tons/hr and the salt solution is feed at the recommended W/P mass ratio (60 to 80 gpm, flow is measured). The grout leaving the READCO mixer gravity drops into one side of a dual slurry hopper. The grout level is maintained in the slurry hopper on the other side using radar level to control the duplex hose pump. The design of the hopper requires spraying/flushing of the hopper unit at a set frequency to clean the surfaces of the hopper during normal operations, introducing additional water into the system. The grout is pumped through a 3 inch, schedule 40 pipe that discharge into the vapor space of the concrete vaults. Pumping distances range between 1000 to 3300 feet of piping. The design for the slurry pump was based on using a Bingham Plastic model and 3300 feet of piping. The Bingham Plastic design parameters are a yield stress of 21.5 pascals and a plastic viscosity of 42.5 centipoise. This was based on testing of simulate grouts in the 1980's. The concrete vaults are 100 feet by 100 feet by approximately 29 feet tall. After processing is complete, the process lines are flushed with water and a urethane poly sphere pig, using air as the motive force, is used to remove any grout that remains in the process line. The flush water and pig are discharged into the active vault. To date, there has been no plugging issue with the slurry lines. There have been issues on the dry solids handling systems, including flowability out of silos to crusting of solids in the day tank. The READCO mixer is optimized for shearing, though not necessarily processing.

The instrumentation used to measure process parameter are load cells on the dry feed systems, magnetic flow meter for the salt solution, radar level meter to monitor and control the level in the slurry hopper, and a coriolis mass flow meter to measure grout flow.

There presently is no sampling/characterization of the dry feed materials or processed grout. The salt solution stored in Tank 50 is analyzed on a quarterly basis for WAC analyzes. Grout samples are prepared using these salt solutions and a TCLP analysis is performed, also on a quarterly basis, as required.

Three-Phase Transport (Dan Krementz)

There are multiple waste tanks throughout the DOE complex that have been emptied as much as possible by conventional pumping techniques. The remaining waste is a combination of liquid and solid waste. Conventional liquid-solid slurry technologies require great volumes of water to suspend and transport the waste at sufficient velocities and line diameters to avoid pipeline pluggage. When water is added to a waste tank, it adds to the overall waste volume, which is not desirable. Conventional pneumatic transport technologies do not apply to these applications due to the liquid content. These issues have led to investigation of alternative waste transport methods. Two specific technologies have been investigated for removing the heel from Tanks 18F and 19F at Savannah River Site (SRS).

SRS has contracted the services of TMR Associates of Denver, CO to clean the floors of tanks 18F and 19F with a remote vehicle named the "Sand Mantis". The Sand Mantis is designed to maneuver on the tank floor and mobilize waste using a low flow, high pressure water powered eductor (approximately

6 gpm motive fluid with supply pressures ranging from 17,000-33,000 psi). The motive force provided by the eductor propels the waste to a receipt tank in three-phase flow. This technology has been tested using a simulant consisting of a well-mixed combination of 65 wt% UOP IONSIV IE-95 20x50 zeolite resin, 25 wt% 70 mesh quartz sand and 10 wt% general purpose kaolin clay saturated in water.[1] The demonstration transfer line consisted of a 2" diameter transfer line with a 53' vertical rise, a 470' horizontal run and a 60' vertical drop.[2] In the vertical rise, slugs of solid-liquid slurry were observed progressing up the transfer line. In the horizontal leg of the transfer line, high velocity air flowed over slower moving solid-liquid slurry that behaved similar to pneumatic dense phase "sliding bed" flow. Approximately 2-3 gallons of motive fluid were needed to propel one gallon of solids simulant. The horizontal portion of the transfer line was never full of slurry, which contributed to the ability of the system to avoid line pluggage. The high pressure water at the eductor end also broke up any large agglomerations of simulant that may have been introduced into the initial leg of the line. As may be expected with these high pressures, erosion of the pipeline occurred during early testing.[2,3,4,5] Refinement of the design has led to a more manageable erosion rate. This technology has not yet been used in a waste tank, but testing of the equipment is in progress.

A demonstration of transfer using the same simulant was performed by Sycamore Construction of Mobile, AL using a more conventional pneumatic transfer approach. The transfer line consisted of a 4" diameter, 40' long vertical rise followed by 6" diameter, 600' long horizontal run. The system utilized a positive displacement air compressor to produce a vacuum that drew material from the source tank and delivered it to the destination tank.[6] When the simulant reached the destination tank, it was separated from the air using a proprietary separator and the air was recycled back to the source tank. The flow regime was similar to that for the Sand Mantis; slug flow in the vertical section and sliding bed flow in the horizontal section. No further testing of this technology is planned at this time.

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2.3.3.3 Idaho National Laboratory Information

INL Transport Issues (Rick Demmer)

1. What is the name of the technology?

   Idaho tank farm transport system.

2. Brief description of technology (ie no more than 1-2 paragraphs)

   The main tank farm at the Idaho National Laboratory (INL) is located at the Idaho Nuclear Technology Engineering Center (INTEC, formerly Idaho Chemical Processing Plant). There are eleven 300,000 gallon tanks used in the INTEC tank farm. The tanks are stainless steel, 40' diameter, right cylindrical vessels buried underground. The tanks are connected to processing equipment, and to other tanks via 3" stainless steel, schedule 80 pipe. Flow is typically gravity flow from processing systems to the tank farm and steam jet flow from one tank to another. Six of the tanks are now out of service, having been emptied of the acidic residue and solids and grouted in place.

   The calcination systems (New Waste Calcine Facility (NWCF) and Waste Calcine Facility (WCF)) were fluidized bed thermal treatment processes that were used to retrieve solutions from the tank farm and solidify them into a granular material. The most interesting solids transport systems at INTEC involved the pneumatic transport of NWCF product. However, these issues have little to do with the pipeline plugging problems addressed at the workshop nor normal DOE pipeline systems and will not be elaborated further.

3. Which site, tank(s), basins, vaults, etc was it deployed?

   The INTEC tank farm transport system manages wastes for the fuel reprocessing and waste treatment systems at the INL. The system consists of tanks WM-180 to WM-190. These tanks are single shelled, stainless steel tanks positioned underground in concrete vaults. The solutions were kept acidic during all phases of treatment, transport and even during retrieval and storage. This led to substantially less solids forming in the solutions, easier retrieval and less difficult solids management. Seldom did the solids level exceed 8" of height, were fairly free flowing and did not form a cake.

   The INTEC tanks were emptied and decommissioned using the same type of equipment generally used in the tank farm. Solutions were removed to failure of the existing steam jet. The remaining solids, which formed a depression around the original jet, were suspended and sluiced with additional water and directional nozzles placed in the tanks. An additional, larger steam jet (2" diameter) sitting on the bottom of the tank was then placed in the tank and shielded transport piping set up to transfer the last residue in the tank. Grout pours were engineered to push the remaining material to the jet as a final retrieval action.

4. How well did it work?

   Few transport issues were encountered in either the operation of the tank farm or the decommissioning of the tanks. The system benefitted from being constructed of heavy stainless steel 304L, with acidic solutions, and low solids concentrations. Two examples of issues will be discussed below in the lessons learned section. One of the most significant issues at INTEC was the plugging of density and level probes. These occurred throughout the INTEC operating history. The probes, particularly at the bottom of equipment, plugged with solids or "hard water" scale. But these were alleviated by using a high pressure pump and backflowing through the probes.

5. What technical evaluation criteria were used to determine whether it worked (no more than 2-3)?

   I know of were no formal critiera for the INTEC tank farm transport system. It was primarily a gravity flow system. Valve lineups were typically manually verified (at least in the old days) during transfers.

6. What is the relationship between the technology and characteristics of the waste (ie waste properties, rheology, and other physical behaviors)?

   The tank farm solutions were acidic (typically 1-3 M nitric acid) with high concentrations of salts and fission products. Solids/slurry data for two tanks are given at the end of this report.

7. What were the lessons learned (high level – no more than 2-3)?

   Two incidents of pipeline plugging were reported by the INTEC tank farm system engineers. The first was where a 3" globe valve in the tank farm valve box (a below grade box) did not open fully during operation. The slowed flow caused some solids to fall out ("slug") and flow became increasingly restricted. The valve was replaced with a ball valve (along with other valves of this type) and no further issues were encountered. The second incident occurred in a complicated pipe section (called a "trombone pipe" at the exit from a processing cell to the tank farm system. Solids had accumulated in this section from an increased concentration of solids resulting from decrepitation of silica gel in the off-gas processing equipment. Convoluted pipe sections such as the trombone are removed and replaced when difficulties are encountered and designs such as this are avoided in future systems.

8. Is there key documentation that is available which provides technical information regarding the technology, especially lessons learned and recommendations?

   There are reports on the operation of the INTEC systems that are available on-site. Little pipeline transport technical information has been retained over the years and the current facility contractor is primarily interested in D&D and not technology or lessons learned reports. There are a few people remaining at the INL that have the "anecdotal" information about these events and this equipment.

   Contacts: Rick Demmer, 208-526-1692; Frank Ward, 208-690-9587; Mike Swenson, 208-533-3376; Mike Patterson, 208-526-5525.

Table 2.6. INL Waste Slurry (solids heel materials) Properties for Two Tanks

Analyte	WM-180	WM-189	Units
Intersitital Water	80	70	%
Particle Size (med)	10	30	um
Particle Dist	2-70	2-200	um
Settling rate	Slow (1)	Fast (5)	g/hr
S Viscosity	2.2	1.9	cP
$NaNO_3$	62	29	%
$Al(NO_3)$	17	27	%
$K_3H_6Al_5(PO_4)^8$	-	13	%
KNO_3	7	-	%
$FePO_4$	-	5	%
Al_2O_3	5	-	%
$Zr(HPO_3)^2$	3	-	%

Table 2.6. (contd)

Analyte	WM-180	WM-189	Units
SiO_2	-	5	%
Sr(89+90)	1.00E-04	6.00E-05	Ci/g
Cs-137	1.40E-04	2.60E-04	Ci/g
Pu-238	2.20E-06	8.70E-05	Ci/g

2.3.3.4 Hanford Site Information

Liquid and Slurry Transport at the Hanford Tank Farms (John Connor, Mark Hall)

Pumps, Equipment, and Instrumentation

Centrifugal (vertical turbine) pumps are the standard for waste transfer pumps. Other pumps such as progressing cavity (positive displacement) may be used in waste retrievals (refer to the retrieval section). Suction inlets are typically within 10 inches of tank bottom but may be designed to draw from above the settled solids in some recent installations. A few pumps have a floating suction head for decanting. Transfer rates between tanks vary from 65 to 300 gpm depending on pump and system parameters. VFDs are installed on a few pumps. Several pumps are equipped with water lines that inject dilution water near the pump intake. Flush water is available either from the water supply or from a water truck.

Regarding material compatibility, aluminum, brass, bronze, copper, and Teflon should be excluded, or must not come into contact with the waste fluid. Transfer pump reliability history is documented in HNF-3128.

Mixer pumps (centrifugal pumps discharging horizontally-opposed fluid jets for supernatant mixing and solids mobilization) have been used in several tanks, although none are currently operable. The SY-101 mixer pump was used for several years to agitate solids and release trapped gas. The AZ-101 mixer pump testing for solids suspension will be discussed in the retrieval section.

Supernatant mixing is also performed by air lift circulator (sparging air in a draft tube) in the 242-A Evaporator feed tank, or through transfer pump recirculation (return stream to tank via a drop leg onto the liquid surface). Several other tanks have air lift circulators for mobilizing solids to enhance heat transfer from the solids to the liquid – however, they have not been used for many years.

The Tank Farm Contractor Operation and Utilization Plan (TFCOUP) documents the assumptions, results and analysis of the HTWOS model used to simulate the waste storage, retrieval, transfer and testament processes. Each update of the TFCOUP describes the most current tank waste processing simulations, inputs to simulations, limitations of the data used and necessary assumptions. The document is usually updated on an annual basis and defines the 518 batches of waste to be sent to the WTP. See Figure 1.

Material balance for transfers is by volume as indicated by level change in the sending and receiving tank. Coriolis mass flow meters have been installed for some SST retrieval transfers, in the 242-A Evaporator, and on two DST transfer pump jumpers. Flow meters, if available, are typically mag flow. Pumps are typically instrumented only for current. Pump discharge pressure is rarely available, and line pressure drop measurements are not available. Line pressure design limits are either 400 or 275 psig.

Most Tank Farms lines are carbon steel. The cross-site lines and a few other lines are stainless steel. Hose-in-hose sections have been used in a number of transfers going back to 1999. Hose-in-hose transfer sections consist of concentric reinforced flexible hoses. The primary hose is typically ethylene propylene diene monomer (EPDM).

Most transfers are through 3" carbon steel pipe (supernatant lines). Transfers from 242-A Evaporator utilize the 2" slurry lines for higher velocities and solids suspension. The new cross-site supernatant (3" stainless steel) transfer line have been in use for about 10 years. The cross-site transfer line length between 200 West and 200 East areas is approximately 7.5 miles. The new cross-site slurry (also 3" stainless steel) transfer line has not been used. The cross-site slurry line is equipped with a booster pumping station to maintain velocities of 4.5 to 6 feet per second depending on SpG and other waste parameters. System design descriptions for the cross-site transfer system and the double-shell tank transfer system are RPP-15136 and RPP-15137 respectively.

Line connections are either via valves or jumpers. Jumpers are rigid or flexible pipe sections between nozzles in a subsurface pit. Design of hose-in-hose transfer lines, piping jumpers and valves, and transfer pumps are covered by internal engineering standards.

Buried transfer lines are encased in steel pipe for secondary containment. Leak detection for transfer lines is via cable inside the secondary containment pipe or via drainage into a transfer structures (e.g., pits) located at low points in the line.

Heat trace is typically no longer used except on some over ground (hose-in-hose) transfers.

The latest DST Integrity Assessment report (RPP-28538) contains useful facility descriptions (including materials of construction), results of examination and analysis of the tanks and transfer systems, and lessons learned from construction and operating experience with older structures (e.g., SSTs).

Transfer to the WTP will be in accordance with the Interface Control Document for Waste Feed (ICD-19). Waste will be transferred to the WTP at flow rates between 90 and 140 gpm in a 3 inch ASTM A312, Grade TP 304L 40S pipe incased in a 6 inch secondary confinement pipe with leak detections. The transfers have an upper pressure limit of 400 psi due to jumper connections limits. The waste must have a critical velocity of at least 4 ft/s with a 30% margin. The waste must meet the physical properties listed in the ICD-19 before transfer. The waste feed are often saturated solutions and are required to be chemically stable (not precipitate solids) at ambient ground temperatures before transfer in the underground lines. A flush of not to exceed 3 pipe volumes (7500 gal) will be used to clear the 3 underground transfer lines. (See Figure 1)

Pipeline Plugging and Unplugging

Lines have plugged or become restricted from scaling, sedimentation, and from precipitation. The only unplugging technique typically attempted is pressurizing the line with water. Flushing with hot water or pressurizing the shortest section of line possible may be attempted. A caustic flush/soak has been used in a saltwell transfer line in an attempt to dissolve precipitated aluminum, with limited success. A number of lines have been abandoned due to plugging, including several of the old cross-site lines. The most likely cause of cross-site line plugging was precipitation. Sodium phosphate dodecahydrate needle crystals are the most problematic for plugging by precipitation. These precipitates are temperature dependent and capable of plugging at very low solids concentrations. Alumina compounds (Gibbsite and Boehmite) have been observed to form solids which can plug pipe lines. Cross-site transfer lines between the 200 East and 200 West areas have existed for about 40 years. In 1993, McKay reported that six of the lines had been built and four are permanently plugged.

RPP-17247 describes plugging experience and recommends dilution and flushing practices to avoid line plugging, and includes a number of useful references. This report is the basis for the internal engineering standard for dilution and flushing. A few recommendations are summarized here.

- Transfers with a high risk of precipitating solids should be evaluated using rules of thumb noted in these documents, laboratory cooling tests, and chemical modeling as appropriate.
- Generally, transfer velocities of 4 – 6 feet per second are recommended. If the velocity is less than 6 feet per second, then critical velocity analysis using the Oroskar-Turian empirical method (AICHE 1980) is recommended. This method was selected based upon literature review and comparison with other correlations (RPP-5346 further recommends adding a 30% safety factor to the Oroskar-Turian empirical correlation to account for uncertainty in the correlation and to prevent unstable flow conditions that can occur near the critical velocity).
- Transferring at a flow below the critical velocity should be limited to short durations (< 2 hours) and followed under process controls (such as monitoring of flow rate and pressure) and followed by a high-velocity line flush.
- Post-transfer line flushes should be a minimum of 1.5 line volumes and should also achieve 6 feet per second (or sufficient to mobilize solids as determined by critical velocity calculation).
- High-volume flush connections should be used to reduce the overall line pressure drop and increase the flush velocity. The use of "quick-disconnect" fittings for line flushes is not recommended.

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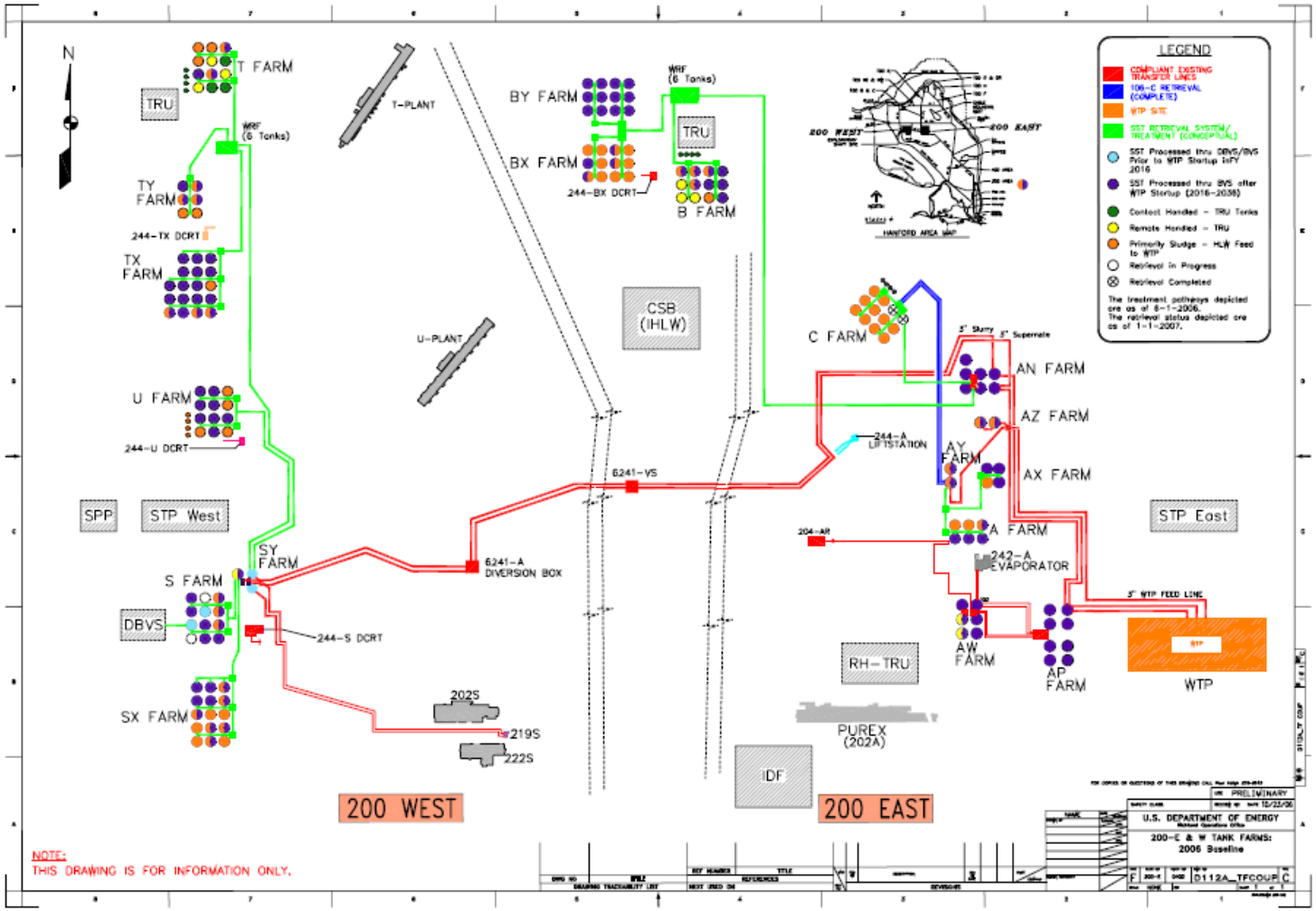

Figure 2.12. Waste Transfer System TFCOUP 6

WM2008 Conference, February 24 - February 28, 2008, Phoenix, AZ

LESSONS LEARNED IN OPERATING THE HOSE-IN-HOSE SYSTEM FOR TRANSFERRING SLUDGE AT HANFORD'S K BASINS - 8236

M.W. Peres
Fluor Government Group
PO Box 1050, Richland, WA 99352
USA

ABSTRACT

In May 2007, the Department of Energy and the Fluor Hanford K Basin Closure Project completed transferring sludge from the K East Basin to new containers in the K West Basin using a Hose-in-Hose system. This project presented a number of complex and unique technical, operational, and management challenges that had to be resolved to complete the required transfers and satisfy project milestones. The project team (including DOE; regulators; and Fluor management, operations, maintenance, engineering and all other support organizations) found innovative solutions to each challenge.

This paper records lessons learned during the operational phase of the sludge transfer via the Hose-In-Hose system. The subject is limited to the operational phase and does not cover design, development, testing or turnover. A discussion of the situation or problem encountered is provided, along with the lesson learned as applicable to a future program or project.

Figure 2.13. Abstract from lessons learned at K Basins

2.3.3.5 Florida International University Information

In the past, some of the pipelines at DOE sites have plugged during high level waste (HLW) transfers, resulting in schedule delays and increased costs. Availability of a pipeline unplugging tool/technology is crucial to ensure smooth operation of the waste transfers and to ensure tank farm cleanup milestones are met. Florida International University (FIU) had previously tested and evaluated various unplugging technologies through an industry call. Based on mockup testing, two technologies were identified that could withstand the rigors of operation in a radioactive environment and had the ability to handle sharp 90° elbows. These technologies were NuVision Engineering's Fluidic Wave-action Technology and AIMM Technologies' Hydrokinetics. A second phase of testing is currently being conducted for both technologies with the objective to qualify at least one technology for subsequent deployment at a DOE facility. In this report, the testing and data analysis of NuVision's technology are presented.

The current phase of testing and qualification is comprised of a heavily instrumented 3-inch diameter full-scale pipeline, facilitating extensive data acquisition for design optimization and performance evaluation, as it applies to three types of plugs typical of DOE HLW. One of these plug types is a kaolin-water mixture typically used in emulating slurry mixes. The other two plugs are crystallized salt plug simulants recommended by Hanford Waste Treatment Plant (WTP) engineers. Three different test bed lengths (285, 621, and 1797 ft) were utilized to determine the effectiveness of NuVision's technology with respect to pipe length. Erosion rates were determined for each plug type and at each test bed length. An amplification of the inlet pressure was observed at the blockage area which demonstrated the need for

a complete analysis of the pressure pulse propagation through the pipeline. Wave speeds have also been analyzed to determine correlations between the amplification factors, unplugging rates and equipment control parameters utilized by NuVision.

Although some correlation is observed between erosion rates and other test parameters, the parameters that directly influence it are not easily discernable. The amplification factors were correlated to the process control parameters (i.e. an increase in drive time for the same drive pressure will increase the amplification factor – an increase in suction time will decrease the amplification factor). The process control parameter, in turn, directly affects wave speeds. It was also noted that the cavity size affected the amplification factor and resulting wave speeds.

Cross-site lines at Hanford can extend almost eight miles. With access locations at either end, maximum pipe lengths to plugs could reach as far as 19,000 ft from an entry point. It is for this reason, the experimental test data was extrapolated to 19,000 ft. Extrapolated test data include maximum pressure, unplugging rates, energy input, and wave speeds. During the experimental testing, variations in the process control parameters were observed, and became more extreme at the longer test bed length. These variations are believed to be due to the do to changes in the environmental conditions during testing. The variability in process control parameters makes it difficult to extrapolate test results to longer pipeline lengths.

An alternative method to predict some parameters of the testing at the scaled up pipe lengths is to use a simplified 2-D model of the transient flow in the pipe. Results show that the modeling can be used to predict maximum pressures at the three test bed lengths.

2.3.3.6 Pipeline Transport Testing at PNNL AP Poloski

The U.S. Department of Energy (DOE) Office of River Protection's Waste Treatment and Immobilization Plant (WTP) will process and treat radioactive waste in the underground storage tanks at the Hanford Site. Piping, pumps, and mixing vessels will transport, store, and mix the high-level waste (HLW) slurries in the WTP.

The testing performed at PNNL address the need to perform critical-velocity tests on physical simulants. Test results are summarized by Poloski et al. (2008). Critical velocity is defined as the point where a moving bed of particles begins to deposit on the bottom of a straight horizontal pipe during slurry-transport operations. Deposition testing of slurries containing particles with well-characterized size and density suspended in fluids with known rheological properties was performed. The test results provide information to evaluate the conditions that can lead to plugging in pipeline transport operations.

The simulant test particles ranged in density from 2.5 to 8 g/cc while the nominal particle size ranged from 10 to 100 μm. Seventeen tests were conducted with these test particles suspended in three carrier fluids with target Bingham plastic yield-stress values of 0, 3, and 6 Pa.

An experimental flow loop was constructed of 3-inch schedule 40 stainless steel piping with a mixing tank, slurry pump, and instrumentation for determining flow rate and pressure gradient. At the beginning of a test, the slurry flow velocity was nominally set to 8 ft/sec. The flow was then incrementally decreased and a steady-state pressure gradient was obtained at each flow condition. A rise in pressure

gradient as the flow rate drops indicates that the pipe cross-sectional area is filled with moving sediment. This point is referred to as the "critical velocity." The laminar-to-turbulent transition velocity is referred to as the "transition velocity."

To clear the sediment bed from the system, a flush system modeled after the WTP design (as of August 2007) was installed and tested. This system consists of a pressure vessel where an initial charge of water is placed. The pressure is then increased to a target value, nominally 100 to 110 psig. A valve is opened, and the high-pressure water flush removes deposited slurry particles from the pipe loop. Results from this testing provide an indication of slurry stability as a function of fluid rheological properties and transport conditions. The flush system effectively removed sediment beds from the piping with the following observations:

The pneumatic flush system must be opened slowly to erode the sediment bed from the top down. If the pneumatic flush system is opened quickly, the sediment bed is simply pushed to the nearest corner, and a granular plug develops and completely fills the pipeline cross-sectional area. During testing a granular plug was slowly forced through the system by cycling pressure to the line on and off. In the transparent section, the velocity of this granular plug was observed to be on the order of inches per second. It took several minutes to remove this granular bed. Cycling the valves open and closed during these situations appeared to accelerate recovery operations. Operation in this mode resulted in water hammer as the flow stopped and started. Interestingly, the most difficulties occurred with the low-rheology tests. Higher rheology likely offers a higher apparent viscosity on the particles in the sediment bed with an associated increase in shear stress that promotes plug removal.

At flush-to-line volume ratios of 1.5 to 1.7a small amount of particles were still observed in the transparent section. A minimum flush-to-line volume ratio of 2 would likely produce only a trace amount of particles in the piping.

Since typical critical velocity correlations are limited to Newtonian fluids with particles greater than 100 micrometers in size and the WTP expects to process large quantities of non-Newtonian slurries with these properties, the PNNL testing investigates deposition velocity calculations for non-Newtonian slurries. The PNNL test results indicate that a design approach for critical velocity of both Newtonian and non-Newtonian fluids should consist of at least three criteria: 1) the critical deposition boundary, 2) the transitional deposition boundary, and 3) the laminar deposition boundary. These boundaries define the transitions between several slurry-transport flow regimes. Four of the major regimes are shown schematically in Figure S.1.

The three boundaries between these four regimes are strong functions of slurry rheological properties. By plotting yield stress on the abscissa and flow velocity on the ordinate, a "stability map" showing these stability regimes can be obtained. An example stability map is shown in Figure S.2. Point W on the abscissa (i.e., rheological properties equivalent to water) represents the Newtonian critical deposition velocity for turbulent flow. This point can be predicted by the Oroskar and Turian (1980) and Shook et al. (2002) equations. As the non-Newtonian character of the slurry increases, adding yield stress, the increased apparent viscosity of the slurry adds drag on the particles and reduces the flow velocity needed to suspend the particles in turbulent flow. The boundary between the stable turbulent and unstable turbulent flow regimes is referred to as the critical deposition boundary and is depicted by path WX (shown in blue).

However, the flow velocity required to reach turbulent flow increases with slurry rheological properties because viscous forces dampen the formation of turbulent eddies. At point X, the flow becomes dominated by viscous forces rather than turbulent eddies. Since the turbulent eddies necessary for particle transport are not present, the particles will settle when flow velocity crosses this boundary, called the transitional-deposition boundary. Along path XY, the stable turbulent and unstable laminar flow regimes are defined.

As the yield stress continues to increase the required pressure gradient for flow increases. At point Y, the required pressure gradient is adequate to push the particles through the pipeline even along the pipeline wall. Often yield stress forces are large enough to suspend the particles in the stagnant core region of a non-Newtonian flow. Along path YZ, the rheological properties continue to increase, which lowers the flow velocity required to achieve the pressure gradient required to push the particles through the pipeline. Hence, for particles in slurries to remain suspended, **pipelines must be operated in Regime A or Regime D**. The PNNL test report presents the equations that define where these boundaries fall with respect to particle and rheological properties.

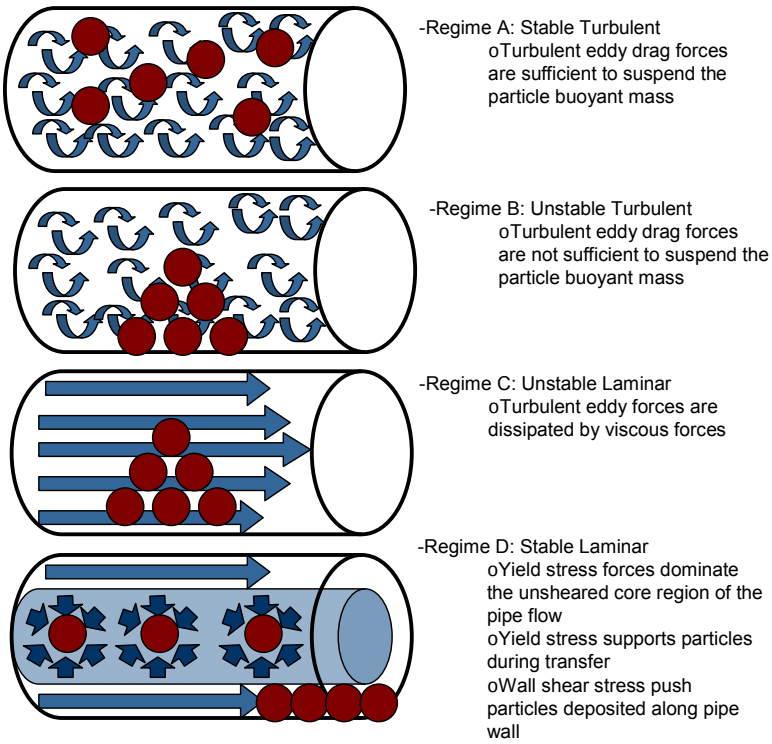

Figure 2.14. Four Slurry-Transport Flow Regimes

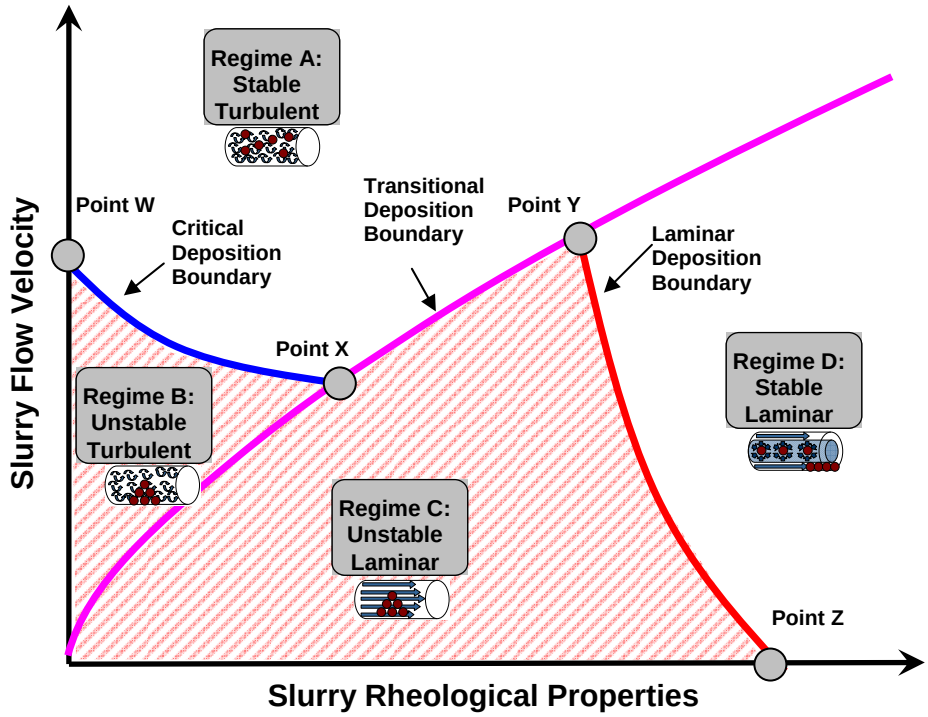

Figure 2.15. Example of a Stability Map

References

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Shook CA, RG Gillies, and RS Sanders. 2002. *Pipeline Hydrotransport with Applications in the Oil-Sand Industry*. SRC Publication No. 11508-1E02, Saskatchewan Research Council, Saskatoon, Canada.

Note: PDF versions of WTP reports can be found at the following URL: http://www.pnl.gov/rpp-wtp/

2.4 Slurry Processing

The Unit Operations Breakout Sessions of the "Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop" met twice during the Workshop. This was a "voluntary attendance" section devoted to discussing the issues, problems and lessons arising from processing slurries. A format for this breakout session was mutually agreed to follow the overall headings of the chemical processing unit operations with their specific applications within the DOE Site's processing systems.

Table 2.7. Slurry Processing Breakout Session Participants

Name	Affiliation	Wed (1/16/08)	Thurs (1/17/08)
Steve Berry	Energy Solutions	X	?
Mike Schliebe	Fluor Hanford	X	?
John Pareizs	SRNL	X	?
Connie Herman	SRNL	X	?
David Dickey	MixTech	X	?
Matt Gebhardt	WTP	X	?
Johathon Bricker	SRS	X	?
Terri Fellinger	SRS-DWPF	X	?
Phil McGinnis	ORNL	X	?
Graham Jonsson	Nexia	X	?
Sam Schicks	Parsons	X	?
Herb Sutter	Consultant	X	?
Michael Poirier	SRNL	X	?
Don Gagel	Consultant ORNL	X	?
Rick Demmer	INL	X	?
Paul Bredt	PNNL	X	?
John Neuville	SRS-DWPF	X	?
Dominic Rhodes	Nexia Solutions	X	?
Tom Michener	PNNL	X	?

In the first session the following major divisions were determined to be the major unit operations (applicable to DOE) for discussion and further development:

- Mixing – Homogeneous mixing, specialized sampling (e.g. accountability, process control, WAC)

Uniform transfer (e.g. melter feed to melter, whole batches), heterogeneous mixing (well stirred, off the bottom), and Computational Fluid Dynamics modeling (this mixing section overlapped with the breakout section on pulse jet mixers); SRS-SS-1, Commercial (Com)-DD-1

- Solid Liquid Separations – Centrifugation and filtration: SRS-MP-1
- Waste Immobilization – Grouting, vitrification and steam reforming: SRS-CH-1, SRS-EH-1, SRS-JB-1
- Chemical Reactions – Leaching, oxidation, reduction, dissolution and precipitation
- Evaporators – Scaling, precipitation and foaming; SRS-WW-1
- Process Control – Instrument line unplugging in processes, solids interference with instrumentation for liquids, On-line assay and NDA

- Ventilation (off-gas processing) – Gas generation, retention and release (H2, CO2, VOC), aerosolization and ties with process chemistry and retrieval; RL-SB-1

There was significant discussion in selecting these divisions. They seem to cover most of the unit operations involving slurries throughout the DOE Complex. Names were assigned for "homework" to identify key DOE sites providing information for these operations. The homework assignments were: identify design criteria the sites used when processing slurries in these operations, identify particular operations were in process or were successfully applied at the DOE Sites, state lessons that had been learned and what guidance references (i.e. DOE Orders) were available. The completed homework assignments are shown above and are labeled with the site and the initials of the contributor (ie, SRS's Sam Shicks first assignment becomes SRS-SS-1). These assignments are collected under these designations in the appendix.

The next breakout session dealt with providing a summary of slurry processing activities that had taken place at each site. It was a frank and open discussion of some "gripes" that the process engineers generally had working with slurries and some improvements that could be made. We also highlighted lessons that have been learned from the DOE Sites. A great deal of the discussion centered on the frustration that we share in "rushing" systems into operation without adequate development. While we understand the high cost of research and development (and that R&D doesn't contribute to the bottom line) the challenge of working with slurries dictates more development for proper startup and operation. Rushing complicated systems into operation without good development usually results in costly upgrades.

This discussion centered around three general areas. They are shown below, along with the names of those who wrote additional information (found in the appendix):

- There is high value in thorough cold testing of unit operations of slurry processing.
- Paper studies are inadequate for slurry processing
- More testing necessary for slurry processing operations
- Sources such as the Rand Corporation, IPA, DuPont, Hanford Pilot Plant Workshop have highlighted the need for extensive cold testing with slurries; Com-DD-2
- Pilot Plant testing is a necessary (though costly) development stage
- DWPF good example of pilot plant
- Operate pilot plants with correct geometry and scale
- Avoid oversimplification of feeds and simulants (see Appendix C "Simulant Development, Approval, Validation, and Documentation" guideline)
- Test abnormal and off-normal conditions
- Retain pilot plants for future feed adjustment tests.
- Integrate unit operations in pilot plants (understand downstream issues from individual processes)
- Understand what cold testing means
- Do not oversimplify rheological models (see Appendix ?? "Slurry Rheology")
- Identify criteria and requirements up front

- Understand the limits of simulant and pilot testing (identify bounds of tests)
- Recognize that radiologically produced materials are different and have different characteristics than non-radiological simulants; INL-RD-1 (see Appendix D "Guidelines for Performing Chemical, Physical, and Rheological Properties Measurements")
- Involve experts and SMEs throughout the testing and startup stages with frequent design reviews; Com-HS-1

Another area of discussion among process engineers is where to turn for understanding of the historical and technical accomplishments within the DOE Complex. This was expressed in the desire to develop an "encyclopedia" of processes that produced the waste solutions we are trying to treat. A good start would be to gather the descriptive training manuals for processes such as PUREX. A knowledge center, like the D&D folks are putting together with Florida International University, that collects process information and identifies SMEs for future reference would be another good step (INL-RD-2). These sessions wrapped up with the agreement that the DOE sites have technical challenges in processing slurries, and that workshops such as this were tremendously valuable; they are valuable for the instruction from slurry experts, and certainly for the interaction and discussion between the various site engineers.

2.4.1 Slurry Process Session Site Information Input

2.4.1.1 SRS PJM Mixing and Limiting Test Parameters – Sam Shicks

Pulse Jet Mixer (PJM) Design Unit Operations for Nuclear Waste Processing of Suspended Solids Slurries

There are a variety of PJM systems currently in use in the nuclear waste processing industries. PJMs have been tested and proven to work well for off-bottom suspension and blending of slow settling solids suspended in Newtonian fluids. Applications for this use should be recognized for PJM consideration since PJMs provide maintenance free service. Pulse jets can be designed for jet velocities up to 40 fps which can be evaluated for off-bottom suspension of potentially dense particulate solids and validated for plant life erosion wear allowances. PJMs have also been used to re-suspend sludge solids in cases where the settled sludge solids behave as Non-Newtonian fluids during the settled sludge state.

This section is a discussion of the PJM design and scale-up strategy from a perspective used by Parsons for the Salt Waste Processing Facility (SWPF). The objective of this discussion is to note some principles involved in the design and scale-up strategies for the PJM systems designed for SWPF.

Design Basis

Designing for the off-normal condition provides a realistic basis for PJM design. The off- normal condition is the state of settled sludge or the presents of dense particulate solids on the tank bottom. Blending is referred here as the state where there is no significant separation between fine slow settling particulate solids and off bottom suspension is referred here as the condition where no dense particulate solids remain on the tank bottom for more than a few seconds.

The same PJM that blends well during normal conditions may have difficulty re-suspending from the off-normal condition and may not be capable of off-bottom suspension of dense particulate solids. If a PJM is designed to return from the off-normal state, then it will be more than adequate to maintain the normal state and can simply be regulated down after reaching that state. Dense particulate solids may be present. Therefore, jet flow rates and velocities must be sufficient to perform off bottom suspension of such solids for pump out removal.

PJM Operation (Sequential Pulsing)

Pulse jet mixer systems typically contain more than one pulse pot inside the tank. Pulsing the pulse pots one at a time is referred here as sequential pulsing. Pulsing more than one pulse pot at a time is referred here as simultaneous pulsing. Sequential pulsing of PJMs has been tested and proven to provide more efficient agitation and has several advantages over simultaneous pulsing and should be considered.

Limited Air Use for Sequential Pulsing

A considerable amount of instantaneous air is required to produce a pulse and if more than one pulse pot is pulsed simultaneously, then that amount only increases by a factor equal to the number pulsed. This places a large demand on the air supply system which must be sized for the intermittent flow requirements. A design for such a system would require a large receiver at the point of use and a very robust compressed air utility system. Compressed air systems are generally sized for greater than 100 pounds of pressure to minimize pressure drop in distribution lines. This minimizes line sizes since most plants don't have the room to route large bore piping for air systems. The air is typically regulated down at the end user location. Sequential pulsing reduces the intermittent load requirements on a plant air supply system.

Reduced Ventilation for PJMs

Venting the pulse tubes creates a surge in air flow through the PJM ventilation system. The net effect creates a super-imposed back pressure on the same vent manifold which services the remaining pulse pots. PJM systems operated with three-way valves are more efficient than those operated with air jet pairs since compressed air is not required to pull vacuum (See Figure 2). However, the same are vented to a common vacuum header and are affected by downstream back pressures. Sequential pulsing reduces the intermittent ventilation loads on the ventilation system and minimizes superimposed back pressure effects on the vent manifolds for systems designed to operate using three-way valves.

Limited Tank Surge for Sequential Pulsing

When a pulse pot discharges, the level in the tank rises. This is compounded when more than one pot fires at the same time. Less tank surge means less lifting of the mass of liquid inside the tank and more mixing within the tank liquid. It's easier to deform something if it is held in place than it is if it is moving since part of the energy added to deform it must be used to move it. When pulse pots are fired sequentially, the tank surge is balance by the filling of the remaining pulse pots so that the net tank surge is hardly noticeable when the system is operating. This also puts less demand on the tank ventilation system. Some nuclear process tanks are covered and vented to provide a dynamic vapor barrier which is negative to the cell containing the process tank. Tank surge only adds to the design capacity of the tank ventilation system.

Near Steady State Mixing

Sequential pulsing provides a quasi-steady state effect in the tank, one that can be measured. Although there is an intermittent action taking place in the mixing process, this action is repeatable. This works best when there is little or no delay between sequential pulses and sets some criteria for the volume and number of pulse pots since the first pulse pot must be filled before the remaining are pulsed. On the contrary, if pulse pots are fired simultaneously, then there is a period of delay necessary to fill the pots and this delay period increases disproportionately with the scale of the PJM system. Such variance in the mixing action would be difficult to scale up from. Sequential pulsing reduces the number of test parameters since there is no delay between pulses. Sequential pulsing acts like a repeatable forcing function on the tank system.

Nuclear Safety

Accidental blow through events are such that occur when there is a failure of the PJM control system which causes air to be sparged into the tank. A PJM system must be designed to mitigate such an event in the nuclear waste processing industry. Sequential pulsing provides for much easier design mitigation since less air is required which can be flow limited using a restricting orifice upstream of the system. Process vessel ventilation HEPA systems must be sized to handle the generated aerosolization from the flow limited air sparging into the tank and can be much smaller than if the pulse pots were designed to pulse simultaneously.

PJM System Design

PJM systems on any scale should be designed using a validated computer program since the design of any pulse jet mixer system can only be completed with a detail understanding of the air and fluid dynamic responses within the pulse pots and dynamic liquid levels in the mixing tank. An example of some key performance variables that were simulated using the Parsons Pulse Mixer Program are shown below.

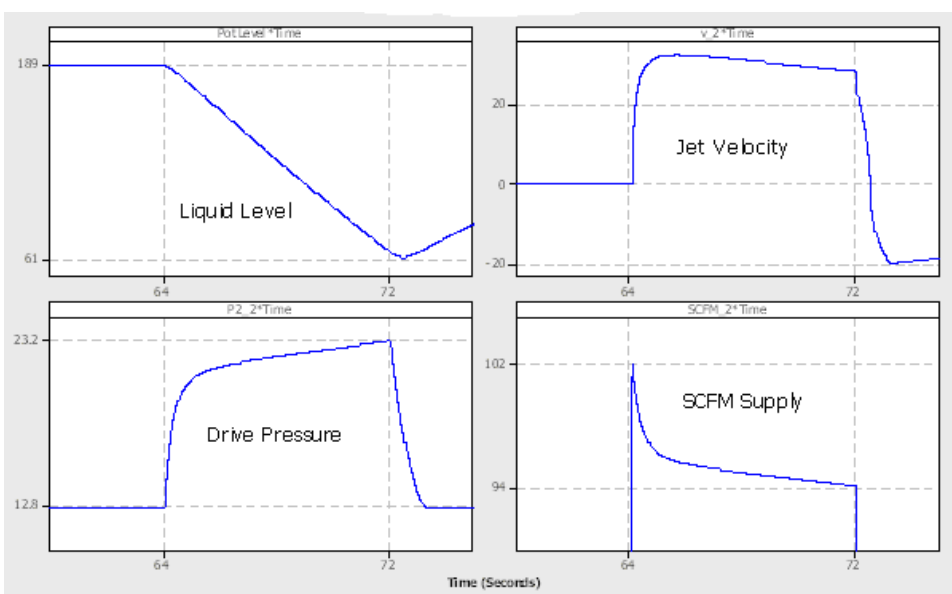

Figure 2.16. Performance Parameters for Parsons Pulse Mixer Program

A computer program serves as an iterative design tool which provides the necessary feed back to the system design engineer from which the design engineer can evaluate the size of the pulse pot and jet nozzles in addition to the air supply and vent header. The performance regarding the average jet velocity can be determined before the system is built and can be incorporated into any test matrix to determine the proper control setting that will be used to run scaled tests. Compressed air and ventilation systems do not scale up. In fact these systems increase by several magnitudes and must be sized separately for each PJM system. For larger systems, the drive pressure competes with the flow rate leaving the pulse pot. There is no closed solution for the standard PJM design. A computer program is a must.

The full scale compressed air system should be designed with preventative features which can indicate a potential blow through event before it occurs. A properly designed air delivery and ventilation system is the first step towards preventing a blow through.

Restricting the Air Supply

Restricting the air supply by placing an appropriately sized orifice downstream of the pressure control valve will prevent a drop in the supply air system pressure and provide the same drive pressure to each pulse pot. The orifice should be sized to account for 90% of the air line loss from the pressure control valve to the pulse pots. This however, is less efficient but also provides the advantage of identical pulses to each pulse pot.

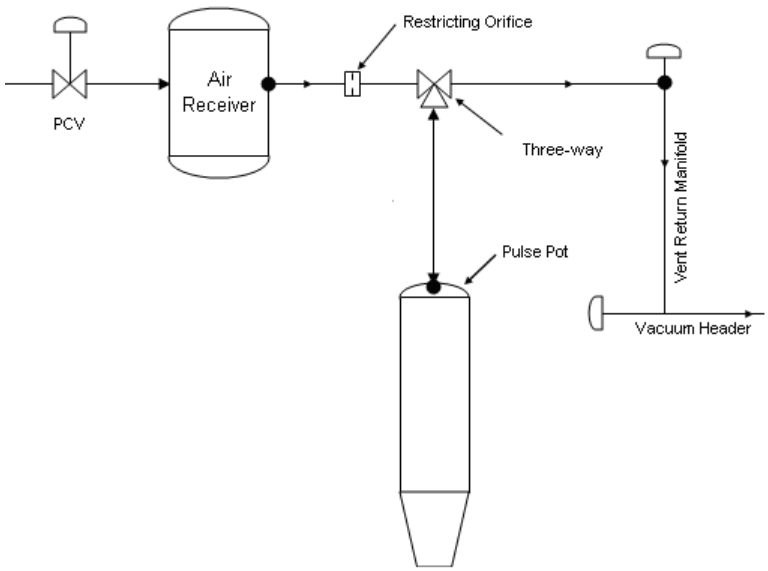

Figure 2.17. Pulse Mixer Schematic

Pulse Pot Design

A fully submerged pulse pot is more efficient since the drive pressure ramps up very quickly. A partially submerged pulse pot will contain free air space on the top. The later is less efficient but provides a unique response in the drive pressure profile (See Figure 2.16) which can be measured from the three-way valve enclosure cell. The restriction orifice located in the air supply line and the size of the jet nozzle creates the unique drive pressure profile. If in the event of a control system failure, the measured drive pressure response can be used to prevent an over-blow event and the system can be shut down for

repair. The minimum liquid level inside the pulse pot should be at least one second before the transition to the nozzle occurs. The pulse pot should be sized to provide sufficient capacity to support the design drive time and average jet flow rates. Sequential pulsing requires that the first pot in the sequence has filled before the remaining pots have completed their drive. If "N" is the number of pulse pots and "DT" is the drive time, then the time to vent and fill the pulse pots must be less than (N-1)DT. Once again, this can only be determined using a computer program.

PJM Ventilation

The PJM ventilation system should be vented to a designated vent system equipped with HEPA filter. Consideration should be given for including demisters cooler and heaters to condition the air before it enters the HEPA filter system. The line sizes for the ventilation system should be robust especially if more than one PJM system is supported by the same ventilation system. A computer program is used for calculating the transient ventilation loads.

Reliability and Maintenance

Full scale PJM designs should consider reliability issues. The most prone component to fail is the three way valves. These valves should be located in a shielded cell which support contact maintenance and located at elevations which exceed the maximum vacuum lift by the ventilation system. Flush down systems are required. Pulse jet nozzles should be designed with plant life erosion wear allowance. Use of hardened exotic steels should be considered as coating or inserts at the nozzle flow areas. Tanks should be equipped with ¾ inch thick stainless steel erosion wear plates beneath the jet nozzles. Erosion testing using an approved simulant should be considered to validate the erosion wear allowances for the design maximum pulse jet velocities.

Performance Testing for PJM Scale-Up

Performance testing of PJMs should be based on re-suspension and blending from the off-normal condition. Off bottom suspension of potentially dense particulate solids should also be considered. Test plans should be based upon a sound technical basis. A technical basis should be a dimensional analysis of the operating variables which have a known effect on the measured variable. The result of this analysis should be a hypothetical model which states that a functional relationship exists between a set of dimensionless scale up parameters, one of which should include the dependent variable. The technical basis should also state the method that will be used to define the functional relationship using valid test data. Existing models should be considered in the technical approach.

Test parameters should be minimized during testing. The mixing performance of a PJM is driven by the average jet velocity and flow rate and limited by the working level, liquid volume, geometry and physical properties of the slurry and particulate solids. *Scale up should not be based on the drive pressure which is a means to achieve the average jet velocity and flow rate*. The air delivery system is sized independently using a computer program.

A reduced scale test matrix should consider independent tests performed at two working levels for a single PJM configuration such that at each working level, the average jet velocity and flow rate can be varied by controlling the supply air pressure setting. All other variables should be held constant.

Measuring the blend time of settled sludge solids is a useful means for evaluating PJM performance parameters. Blend times can be adequately measured from an off-normal condition. If solids concentration is measured in an opaque slurry then an acoustical sensor should be used and not an optical sensor. Optical sensors have been proven to be limited to less than 3 weight percent. The Markland 502-TP submersible acoustical probes work well for slurries up to 20 weight percent suspended solids. The blend time can be measured by submersing three suspended solids sensors in the tank at three elevations with one sensor located just beneath the liquid surface. The blend time is noted when all three sensor are reading the known total suspended solids concentration in the tank.

Design Working Level

The aspect ratio is referred here as the ratio of the working level divided by the internal tank diameter. This ratio should be less than one for best mixing performance.

Hydraulic Diameter

The hydraulic diameter is four times the total liquid surface area excluding the area occupied by the pulse pots, divided by the wetted perimeter. The wetted perimeter is the perimeter of the pulse pots plus the inside perimeter of the tank wall. Turbulent mixing is reduced when the hydraulic diameter is reduced.

Average Jet Velocity and Flow Rate

The average jet velocity is calculated by dividing the volume of liquid inside the pulse pot which is discharged into the tank by the drive time and by the nozzle exit area. For sequential pulsing, the drive time does not include the vent drive time which is the short period following the end of the drive time and liquid continues to exit the pulse pot. However, the pulse volume is based on the maximum and minimum levels inside the pulse pot during the drive time plus the vent drive time. This is the true average jet velocity. The average jet flow rate is simply the average jet velocity multiplied by the nozzle flow area.

Scaled PJM Testing for SWPF

Pulse jet mixers have been tested and proven to work well for off-bottom suspension and blending of slow settling solids using SRS HLW/MST sludge simulant blended at (5-8 wt % suspended solids). The same simulant properties are considered Newtonian when blended. Testing performed at off-normal conditions provided the best method for scale up testing. Such conditions allowed the solids to settle and compact at the tank bottom. In this state the mixture no longer behaved as Newtonian fluid. Operating parameters used to re-suspend the sludge slurry and blend the solids evenly in the tank could be evaluated against the time to reach this state. Results provided accurate scale-up models for predicting the same behavior in a larger scale tank. Off-bottom suspension of dense particulate solids was evaluated in separate tests using dense particulate solids simulants.

Scaled PJM testing was performed in two different sized test tanks, a 1/5 and a 5/8 scale system were tested. The scale is based on the internal diameter of the test tank. The PJMs were configured with one pulse pot in the center of the tank and either 3 or 6 pots oriented at approximately 2/3 radial distance from

the center pot. Tank bottoms were dished and perimeter jet nozzles were oriented to point down and perpendicular to the bottom dish. Pulse pots were 80% submerged and pulsed one at a time in a sequential cross pattern with no delay between pulses. This provided a quasi-steady state mixing effect in the tank and limited tank level surge due to pulsing. Average pulse jet velocities were varied by adjusting the air supply pressure at fixed drive times.

Measurements showed that slow settling solids will form a suspended solids layer beneath the liquid surface with increasing solids concentrations near the tank bottom. The fluid in the tank beneath the solids layer behaved as Non-Newtonian. The presence of the suspended solids layer prevented jet velocities beneath the layer from reaching the liquid surface. Eventually, for sufficient pulse jet velocities, the suspended solids layer was pushed up to the liquid surface, after which blending did occur and the fluid behaved as Newtonian. Test demonstrated that the working level was a limiting factor which increased the time to raise the suspended solids layer to the surface. It was found that tests completed at high working levels could only be blended if the average jet velocity was sufficient. In some tests, the suspended solids layer never reached the liquid surface indicating that the shear strength of the same layer exceeded the shear stress induced by the velocities beneath that layer and that the working level was a contributing factor.

The performance of the PJMs, were evaluated based on the duration of time necessary to evenly blend the suspended solids in tank, beginning from the same off-normal condition. This was determined to be the Blend Time. The blend time was correlated using the dependent dimensionless parameter and is referred here as the (Turnovers to Blend):

$$\frac{\sigma \quad t}{V}$$

where σ = average pulse jet circulation (ft^3/second)
t = Measured blend time (seconds)
V = Measured tank liquid volume (ft^3)

As a result of all tests performed, the following it is clearly understood:

a. Tests performed at higher working levels experienced **greater** (turnovers to blend).

b. Test performed with 7 pulse pots experienced **greater (**turnovers to blend).

c. Tests performed with 4 pulse pots experienced **lower** (turnovers to blend).

d. Tests performed after extended settling periods experienced **greater** (turnovers to blend).

e. In all cases, the (turnovers to blend) was **reduced** when **σ** was increased.

f. The (turnovers to blend) is dependent on the, jet velocity and flow rate and is independent of the nozzle diameter which is defined by both.

The most surprising result of this study is items b and c. Since the pulse pots were pulsed sequentially, additional pulse pots did not provide more agitation. Longer blend times were due to the fact that the mixing was a result of turbulent flows. Therefore, the presents of additional pulse pots reduce the tank hydraulic diameter, which thereby reduces the turbulence as defined by the Reynolds number.

The result of the parametric study produced two scale-up models for calculating the blend time for any prototypical scale using similar simulants.

The first model is a four parameter model which accounts for various working levels, and the tank hydraulic diameter (i.e. 4 pots or 7 pots) is shown below.

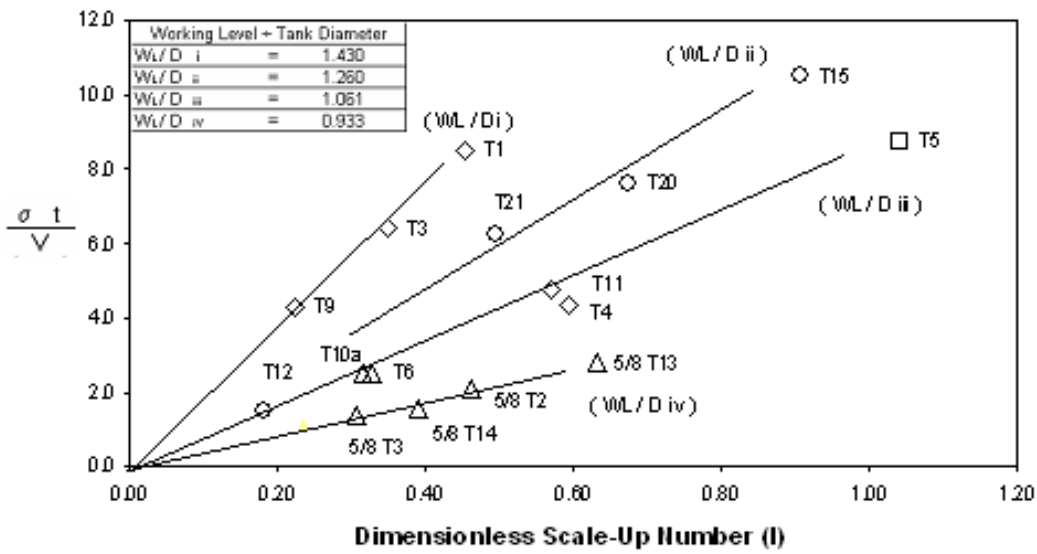

Figure 2.18. Four Parameter Model for Mixing

The four parameter model is represented by a family of parameter fit curves. Each curve represents a combination of 4 and 7 pot tests performed at different working levels. Each curve represents the slope for the working level (W_L) divided by the tank internal diameter (D_i). The curve with the smallest slope represents tests performed in the 5/8 scale test tank.

The Dimensionless Scale-Up Number for (Model I) is

$$\left(\frac{\upsilon}{\sqrt{W_L\, g}}\right)^{-8.89}\left(\frac{\upsilon}{\sqrt{D_H\, g}}\right)^{4.61}$$

Or alternatively

$$\frac{g^{2.14}\quad W_L^{4.445}}{\upsilon^{4.28}\quad D_H^{2.305}}$$

Both are dimensionally equivalent where υ is the average jet velocity and D_H is the tank hydraulic diameter, D is the internal diameter of the tank and g is the acceleration of gravity. The parametric equation for Model I is shown below.

$$\frac{\sigma\ t}{V} = \left(24.7\ \frac{W_L}{D} - 21.3\right)\left(\frac{g^{2.14}\quad W_L^{4.445}}{\upsilon^{4.28}\quad D_H^{2.305}}\right)$$

The second model is a five parameter model which accounts for greater sludge compaction and is represented by the single linear trend shown below.

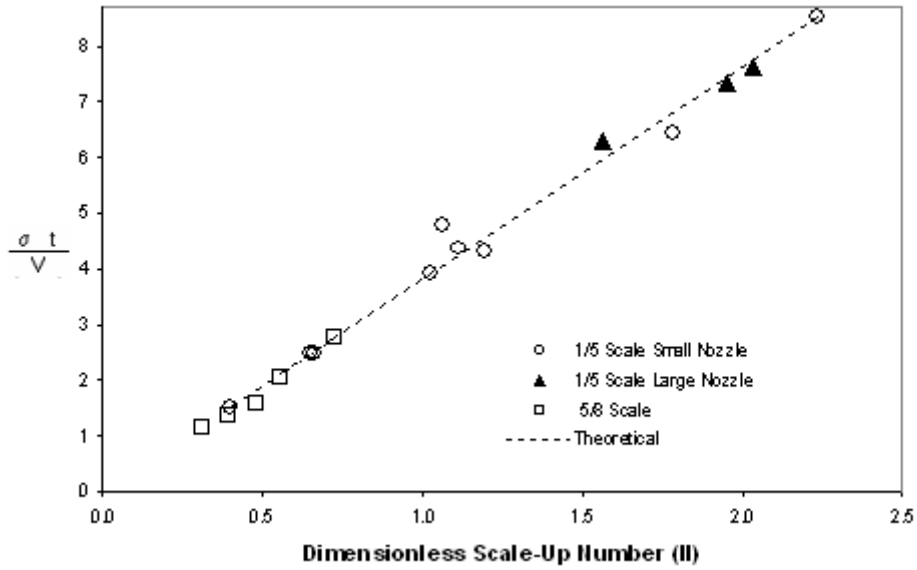

Figure 2.19. Five Parameter Model for Mixing

The Dimensionless Scale-Up Number for (Model II) is

$$\left(\frac{W_L}{D}\right)^{3.20}\left(\frac{W_L}{D_H}\right)^{5.88}\left(\frac{\nu}{\sqrt{S_L g}}\right)^{4.01}\left(\frac{\nu}{\sqrt{D_H g}}\right)^{-7.68}$$

where S_L is the level of the suspended solids layer before the blend test started. The remaining variables are the same as previously described.

Both models fit the test data well and can be used to calculate the blend time for both model scales and are used as the basis for the SWPF full scale PJM design. The parametric equation for Model II is shown below.

$$\frac{\sigma\ t}{V} = 3.82\left(\frac{W_L}{D}\right)^{3.20}\left(\frac{W_L}{D_H}\right)^{5.88}\left(\frac{\nu}{\sqrt{S_L g}}\right)^{4.01}\left(\frac{\nu}{\sqrt{D_H g}}\right)^{-7.68}$$

2.4.1.2 Mixing Operations for Processing Slurries – Dave Dickey

Mixing operations for processing nuclear waste slurries fall in different categories. Processing categories include low-viscosity liquid blending with or without slowly settling solids, low-viscosity solids suspension with more rapidly settling solids, and mixing of non-Newtonian or other concentrated slurries. For each of these categories, the mixers may be conventional rotating equipment, continuous jet pump mixers, or pulse jet mixers. Each type of application has different process requirements. Some applications may involve combinations of requirements at different times in a batch operating mode. Involved in those requirements are known and unknown characteristics of the different types of mixing equipment.

Uniform Liquid Blending

Liquid blending is usually the easiest of the process requirements and the best understood, but not necessarily well documented or even measured for applications involving nuclear waste slurries. Blending is likely to be important when a chemical, such as an acid or caustic, is added to the waste to change the pH or initiate a reaction. The time required to distribute the additive throughout the vessel will determine how quickly the process can begin to take place. In other situations, knowing the time required for uniformity after the addition of a new batch of waste to the existing heel from the previous batch may be important. The most difficult question of liquid blending involves the time required to blend after a stagnant start, as in a storage tank or after a design basis event (DBE). In each case a change in composition must be blended to an adequate degree of uniformity, if for no other reason than to obtain a representative sample.

The two most important factors in achieving uniformity are the absence of "dead spots" and the time required to blend. Determining the absence or existence of dead spots depends on the measurement method. If a sampling method is used to test for uniformity, the location of the sample point(s) may or may not detect appropriate conditions in a vessel. Unfortunately in many nuclear waste applications, the sampling locations are in the bulk of the vessel, while a dead spot may be at the bottom or a corner. Laboratory tests in a scale-model transparent vessel may be the only of determining if or where a dead-spot exists. Similarly, determination of a blend time depends on the method of sampling or observation used. Even if a vessel has no dead spot, some location will be the last to become uniform, or will be the least uniform. In turbulent mixing, that location of last blending can move from location to location with successive tests. Only visual observation of a color removal test in a transparent vessel can identify that final location. Sampling methods can only approximate the time to uniform blending. However, the solution to the problem of a slow blending location can be overcome by using an extended blend time, whereas a true dead spot may remain unmixed. Most vessels with low-viscosity fluids and slowly settling solids will eventually attain a uniform liquid composition or temperature. Settling solids may never become uniformly distributed even with uniform liquid blending and should be considered separately.

Blend times and blending characteristics for rotating mixers in vertical cylindrical tanks are well understood and documented. The remaining questions involve the effects of large quantity additions, rate of addition, the effects of recirculation, unusual geometry, and obstructing elements in a vessel. Typical effects of large quantity additions, rate of addition, and external recirculation are not significant when compared with the more rapid effects on blending by a rotating mixer. The effects of unusual geometry and internal obstructions on liquid blending are less certain and subject to testing. Again, dead spots are more critical than longer blend times.

Questions about the performance of jet mixers are less certain because of limited experimental data and the likelihood of their use in unusual geometries. The motion created by continuous free jets is understood for many applications. The uncertainties involve recirculation patterns and the effects of obstructions. Testing can be done in geometrically similar models. Sampling is necessary when jets are used in actual installations. Again, the uncertainty due to restrictive sample locations and the possible effects of dead spots must be considered. Further research and development is justified to the extent that unusual geometries or new jet mixers are being considered.

Pulse jets only add to the uncertainties of estimated blend times. First, the pulse velocity, quantity, and duration all influence how much fluid in motion. Second, pulse jet mixers provide their own obstruction effects and special geometries. Limited sampling and observation data for blend times provide some information about effects of PJMs. However, the blend time questions are secondary, only because even less is known about how solids suspension can be achieved with PJMs. More observations and data are needed to understand the blending capabilities of PJMs for low viscosity blending.

Low Viscosity Settling Solids

Most slurries with settling solids involve low viscosity liquids, although high concentrations of solids may result in effectively high-viscosity fluids, which will be discussed in the next section. The effects of settling solids must be considered in both pipelines and vessels. All but the smallest, least dense, dust-like particles settle, although some very slowly. The problem with most nuclear wastes is that small quantities of very dense or oversize particles may cause all of the problems. These particles are likely to accumulate over a period of time, either plugging pipes or collecting in the bottom of vessels. Some particles most likely to settle are also likely to be radioactive, and thus potentially a cause of safety problems. The uncertainties of waste properties and composition make conservative particle suspension capabilities a necessity.

To a first approximation, experimental investigations and research studies suggest that pipeline suspension and in-tank suspension are similar, at least with respect to particle suspension characteristics. Turbulent and transitional particle settling, in turbulent fluid flow, is more dependent on particle density than particle size, as compared with terminal settling velocity in a stagnant fluid. Thus, high density particles, like oxides and salts of heavy metals can be more difficult to suspend than lower density particles with similar settling velocities. This effect is especially significant in nuclear waste, since uranium, plutonium, and similar element compounds are in the heavy particle category. The remaining uncertainty is how small quantities of difficult to suspend particles behave when mixed with larger quantities of less difficult to suspend particles. Several unanswered questions exist for all types of particle distributions. Is the observation of small quantities of dense particles the worst case? Is the effect of more easily suspended particles a positive or negative influence on the more difficult particles? Or does the relative concentration of particles have different effects? The selection of a simulant can be a critical factor in testing and evaluation. These uncertainties and settling effects occur in both pipe flow and agitated vessel suspension. More investigation is needed.

Again the understanding of solids suspension is much better for rotating mixers than for jet mixers. Rotating mixers are commonly applied in vertical, cylindrical tanks with baffles. Axial flow impellers work best when the downward flow from the impellers effectively sweeps across the bottom of the tank. Correlations for solids suspension provide reasonable estimates of their performance. Testing in geometrically similar stirred tanks with scale-up is the best approach to evaluate specific situations. Uncertainties still exist for particle size and composition distributions. Application of rotating mixers in the preparation of slurries of glass formers usually permits sufficient conservatism to assure uniform suspension. Uniformity has been tested and sampling methods used successfully.

Practical experience with continuous jet mixers in nuclear waste applications provides valuable information about both the suspension and resuspension of solid particles. Continuous jet mixers have been used successfully in many storage tanks. Although, because of geometry and access limitations,

complete resuspension in storage tanks is not always possible. Velocities and flow patterns with continuous jet mixers can be predicted or computed, as with CFD, to reasonable accuracy. The effects of local velocities will help characterize effects on solids suspension.

The solids suspension capability of pulse jet mixers for settling solids in low viscosity applications is a significant unknown at this time. Most of the pulse jet mixer experience has been with slowly settling particles, where good liquid motion was sufficient to maintain suspension. Little is known about the effects of PJMs on solids suspension with difficult to suspend particles. The same unknowns about mixed solids and low concentrations of difficult to suspend particles add to the uncertainty of PJM performance. Test plans and equipment construction is underway to generate some relevant data.

Non-Newtonian Slurries

At sufficiently high concentrations, particle-particle interactions in slurries make them behave as if they are non-Newtonian fluids. The "sufficient" concentration is partly a volume percentage effect and partly a particle size distribution effect. High volume percentages and wide particle size distributions both contribute to the non-Newtonian fluid characteristics. In some situations with clay-like particles, the non-Newtonian characteristics can cause shear-thickening. Shear-thickening gives the slurries a higher apparent viscosity when pumped or agitated than when poured slowly. In other situations, which are more common with nuclear wastes, the slurries take on a yield-stress, with shear-thinning characteristics beyond the yield point. These shear characteristics are more typical of ketchup, which resists flow when stagnant and then thins to a lower viscosity as it is moved by pouring, pumping, or agitation.

The critical issue with yield-stress fluids, whether mixed with a rotating or jet mixer, is stagnant regions. Any agitation of a yield-stress fluid moves the fluid closest to the mixer effectively, with penetration to only some distance. If the penetration extends to a distance equal to or greater than the distance to the tank wall then everything moves. If the penetration does not reach the limits of the tank contents, a "cavern" is formed. Within the cavern, the fluid moves, but beyond the agitated limits of the cavern, everything is essentially stagnant, potentially a huge dead spot.

With rotating mixer agitation, the region around the impeller is the first volume to move and a single cavern is formed if insufficient agitation is present to overcome the yield stress. With continuous jets, the effects are similar to a rotating mixer, with a cavern formed in the volume where sufficient motion is present. With pulse jet mixers, the displaced volume is around each mixer with the cavern moving with the changing liquid level. In applications where installed mixers have not provided sufficient agitation, the addition of spargers has increased the moving volume. Motion throughout the tank is usually a requisite for sufficient agitation.

Modeling of Slurries

The critical modeling consideration in most agitation problems, especially solids suspension, is geometric similarity. Even small changes in tank, mixer, or obstruction geometry have been shown to affect solids suspension. Without accurate geometric similarity, questions will remain concerning the effects of support structures, tank internals, and pulse jet mixers. Small-scale tests in transparent tanks have been the standard for observing "just suspended" conditions of solids suspension. The definition for "just suspended" is that the solids do not rest for more than about one-second on the bottom of the tank.

This just suspended condition can be observed visually, but must be calibrated to use sampling or probes. Sampling and probes may introduce the effects of other obstructions. Testing is always conducted with actual particles and liquids to characterize the effects of settling on agitation performance accurately.

Computational fluid dynamics has not been an effective predictor of solids suspension performance without experimental validation. The computer models cannot simultaneously model small particles and large flows. The particle models used for computation are based on terminal settling velocity in a stagnant liquid, not suspension in turbulent flow. A CFD model can be used to investigate a limited range of changes from a validated condition.

2.4.1.3 SRS Slurry Handling Input Processing Solid Liquid Separations – Michael Poirier

Solid-Liquid Separations

SRNL has been asked to size filters to remove solid particles from a number of waste streams. In sizing the filters, one must consider particle removal and filtration rate.

To select the pore size for particle removal, one needs to know the particle size of the solid particles. Our preferred particle size method is the Microtrac particle size analyzer. It is relatively easy to determine the particle size of nonradioactive slurries. With radioactive slurries, we can measure the particle size with the Microtrac, but in some cases the dose from the sample is too high to allow the analysis to be performed. In these cases, we collect a slurry sample on a coupon, allow the coupon to drain, and analyze the coupon with scanning electron microscopy (SEM). The SEM generates pictures. We identify particles on the pictures and measure the size of these particles. We collect pictures at different magnification and generally analyze about 500 particles.

Once the particle size is known, one selects the filter pore size. Ideally, one would want all particles to be greater than the filter pore size. However, that may not always be practical. A more practical approach may be to select a pore size that is smaller than 99, 95, or 90 % of the particles.

The effect of particle size and filter pore size on filtration rate or filter flux cannot be determined without testing. Ideally, this testing should be performed at pilot-scale or full-scale.

Tarleton and Wakeman investigated the effect of particle size and pore size on filter fouling.[1] They found feed solutions with smaller median particle size to reduce filter flux more rapidly. They observed that the smaller particles in the feed are responsible for the fouling or cake layer in or on the membrane, but the median particle size is a poor indicator of fouling potential. They suggested comparing the 10th percentile particle size to the 90th percentile filter pore size. When this ratio is significantly less than 1, pore fouling is reduced.

These guidelines provide an initial section of filter size, but testing with actual feed material or a good simulant is needed for final filter size selection.

1. Tarleton, E. S.; Wakeman, R. J. "Understanding Flux Decline in Crossflow Microfiltration: Part I-Effects of Particle and Pore Size", Trans ChemE, Part A, 1993 (71), 399-410.

Retention of Pilot-Plant Facilities

Having pilot-plant facilities available after startup can help facilities solve problems that occur during startup.

Following start, the filters at the SRS Effluent Treatment Facility (ETF) were operating well below design throughput, because two pilot-plant facilities were available, they allowed SRNL to conduct testing needed to identify the problem of the low throughput and develop solutions to the problem.

Understanding of Upper and Lower Boundaries of Operation

At SRS and at other DOE sites, there is a tendency to test "bounding conditions" or to make the test conditions "conservative". We had a case in which this approach gave us serious problems. We were developing the submersible mixer pump for use in the SRS Tank Farm. Because our dissolved salt solution has a viscosity of 3 – 5 cp and sludge slurries can be Bingham plastic, we provided the vendor with a maximum viscosity and yield stress expected. We did not the pump needed a minimum viscosity of 3 cp to operate. Because of this lower limit, we could not test the pump with water.

Test Normal and off Normal Conditions

Many processes operate very well at steady state, design conditions. Often problems will occur following an unexpected shut down. In testing the rotary microfilter, personnel performed shut down and restart tests to evaluate the impact of an unexpected shutdown on the equipment. For the Actinide Removal Process, SRNL personnel investigated the impacted of an extended shutdown on the ability of the process to resuspend sludge/MST slurries.

Integration of Unit Operations

When testing a unit operation, one needs to look at the effect of its product on downstream facilities. One also needs to examine the impact of upstream processes on the unit operation. Some examples of experiences follow.

SRNL conducted testing to support the design of the Hanford Waste Treatment Plant. One area of testing looked at the impact of recycle streams on each of the processes.

In the testing of coalescers for the MCU process at SRS, the coalescers were tested independently of the centrifugal contactors.

2.4.1.4 SRS Feed Prep in Bulk Vit-Design Criteria – Connie Herman

Bulk Vitrification (BV) combines low activity waste (LAW) and glass-forming chemicals within a large disposal container and melts the contents using electrical resistance heating. The baseline BV process includes a mixer/dryer to convert liquid waste into a dried, blended feed for vitrification. This is done by blending and drying liquid LAW and glass-forming minerals (GFMs) in a 10,000-liter vacuum dryer at temperatures ranging from 60 to 80°C. The feed is brought to a suitable dryness, consistency, and particle size for transport to the BV container for subsequent melting at approximately 1250° to

1350°C by electrical resistance. The current design uses a "feed-while melt" process in which melting is initiated in a small quantity of staged batch, and additional batch is gradually loaded in increments until the container is filled with waste glass.

Laboratory, engineering, and full-scale tests of the BV melting and feed preparation steps have been performed. In FY07, a full-scale, integrated dryer/melter test was completed using a feed that included modified glass forming chemicals and additions of a carbohydrate source. Bench and engineering scale tests of the dryer operations were critical to determine the behavior of the modified chemicals and carbohydrate source before moving to large-scale dryer tests. (Shimskey 2007; Tedeschi 2007). This testing eliminated several materials that would have caused dryer failure and demonstrated the ability to generate a desirable granulated feed material. However, the FY07 full-scale test showed that scale was an important factor and <50% of the dryer product was in a granulated form with the balance being a fine powder. This fine powder occluded the off gas pipe of the BV melter which needed to be cleared multiple times during the test. (Witwer 2007). Dryer operational and design changes are underway to increase the quantity of granules produced during drying. Melter off gas design changes are underway to reduce feed powder entrainment and buildup on the off gas pipe.

Shimskey RW, WC Buchmiller and MR Elmore. 2007. Dryer Operation Impacts of Proposed MIS Mitigation Changes, PNNL-16659, Rev. 0. Pacific Northwest National Laboratory, Richland, WA.

Tedeschi AR. 2007. Supplemental 130L Scale Dryer Development Test Report, RPP-RPT-31688, Rev. 0, CH2M HILL Hanford Group, Inc., Richland, WA.

Witwer KS, et al. 2007. FS-38D Test Report, 30686-RT-0003, Rev. 0. AMEC Nuclear, Ltd and Pacific Northwest National Laboratory, Richland, WA.

Assembly of Design Requirements and Criteria by SME

When designing a production facility, it is imperative to include the appropriate subject matter experts as early as possible to ensure that any critical design parameters will be incorporated. These parameters could be specific to the process, the facility equipment or potentially upstream or downstream facilities/processes. When slurry mixing and transport are involved, the physical and chemical properties of the material being transported will be important and must be assessed against this criterion. The more that is known about the material, the better the system or facility can be designed. A key example at the Savannah River Site is the high level waste sludge preparation process. The sludge must be washed to remove some of the soluble salts that are incompatible with the DWPF glass matrix and/or melter. However, the process of washing and concentration changes the rheological properties of the sludge. Both the Tank Farm and the DWPF have rheological operating limits because of their respective pump limitations. Recognizing the limitations, while also involving subject matter experts in the glass arena, allows the most efficient preparation of the high level waste sludge for DWPF. The combination of the rheological and glass processing limits continue to be successfully used to find an optimum washing endpoint for the DWPF. Another example at the SRS is the salt treatment process. When the process was selected, very few subject matter experts from the DWPF were involved. Therefore, the potential impact of the introduction of a flammable solvent in DWPF was not identified. Due to the flammability concerns, significant upgrades had to be made, as well as changes to the operating flowsheet, to

accommodate the incorporation of the solvent material. While the problem may not have been completely avoided, more time would be available to solve the problem or potentially find alternative solution.

2.4.1.5 SRS Saltstone Process – Erich Hansen

The Saltstone Processing Facility (SPF) is designed to blend low level waste (salt solutions) with a mixture of dry cementitious material to make a flowable grout that can be pumped to the concrete vaults for final deposition. The salt solution is supplied by Tank 50 and processed at SPF via the salt feed tank. The salt solution is analyzed for density and wt% total solids. Bench scale grout mixtures are made from the actual salt solutions to determine the water to premix mass ratio (W/P) that will provide a grout with a gel times of greater than 25 minutes, bleed solution of less than 2 volume percent after 3 days of curing, and a set (less than 2mm) time between 3 to 6 days. Rheological characterization of the recommended grout is not a requirement for processing.

The SPF dry feed system consists of four storage silos, one containing Class F fly ash, one containing Grade 100 Blast Furnace Slag, and another containing Class I or II Portland cement. The fourth silo is for backup. The present composition of dry materials, call premix, for processing the waste, on a mass basis is 10% Portland cement, 45% Slag, and 45% fly ash. The dry materials are weighted in a single mixing hopper, air blended and pneumatically transported to the day hopper. The day hopper feeds two screw augers, where the feed rate is controlled by loss of weight in the day hopper. The premix and salt solution is blended using a READCO mixer, which is a twin shaft, co-rotating mixer, deigned to mix dry materials with liquids on a continuous basis. The internal mixing blade configuration, in the READCO mixer, is parallel to the direction of flow, maximizing the applied shear for mixing. The internal mixing blade configuration can be changed to impact shear and throughput. The day hopper feeds the READCO mixer at 35 tons/hr and the salt solution is feed at the recommended W/P mass ratio (60 to 80 gpm, flow is measured). The grout leaving the READCO mixer gravity drops into one side of a dual slurry hopper. The grout level is maintained in the slurry hopper on the other side using radar level to control the duplex hose pump. The design of the hopper requires spraying/flushing of the hopper unit at a set frequency to clean the surfaces of the hopper during normal operations, introducing additional water into the system. The grout is pumped through a 3 inch, schedule 40 pipe that discharge into the vapor space of the concrete vaults. Pumping distances range between 1000 to 3300 feet of piping. The design for the slurry pump was based on using a Bingham Plastic model and 3300 feet of piping. The Bingham Plastic design parameters are a yield stress of 21.5 pascals and a plastic viscosity of 42.5 centipoise. This was based on testing of simulate grouts in the 1980's. The concrete vaults are 100 feet by 100 feet by approximately 29 feet tall. After processing is complete, the process lines are flushed with water and a urethane poly sphere pig, using air as the motive force, is used to remove any grout that remains in the process line. The flush water and pig are discharged into the active vault. To date, there has been no plugging issue with the slurry lines. There have been issues on the dry solids handling systems, including flowability out of silos to crusting of solids in the day tank. The READCO mixer is optimized for shearing, though not necessarily processing.

The instrumentation used to measure process parameter are load cells on the dry feed systems, magnetic flow meter for the salt solution, radar level meter to monitor and control the level in the slurry hopper, and a coriolis mass flow meter to measure grout flow.

There presently is no sampling/characterization of the dry feed materials or processed grout. The salt solution stored in Tank 50 is analyzed on a quarterly basis for WAC analyzes. Grout samples are prepared using these salt solutions and a TCLP analysis is performed, also on a quarterly basis, as required.

2.4.1.6 SRS Transport and Mixing of Sludge Slurries in DWPF Jonathon Bricker

The following is the summary of an SRS Report on transport and mixing of sludge slurries:

OVERVIEW OF THE KEY PARAMETERS, TECHNICAL APPROACHES, AND SUPPORT COMPONENTS RELATED TO THE TRANSPORT AND MIXING OF SLUDGE SLURRIES IN THE DEFENSE WASTE PROCESSING FACILITY

J.M. Bricker, J.R. Neuville, and T.L. Fellinger
Defense Waste Processing Facility
Savannah River Site
Aiken, SC 29808

Abstract

The primary function of Savannah River Site's (SRS) Defense Waste Processing Facility (DWPF) is to process and immobilize high level radioactive waste (HLW) into a durable borosilicate glass for safe, permanent geological storage. To date, the DWPF has processed approximately 2.5 million gallons of HLW. To create a batch of feed for the DWPF, several tanks of sludge slurry are retrieved, prepared, and combined into one of the million gallon DWPF feed tanks. A batch of feed nominally consists of 500,000 gallons. After a sludge batch has been prepared and qualified for processing in the DWPF, ~ 6000 to 8000 gallons of sludge slurry is transported on a weekly basis from HLW Concentration, Storage and Transfer (CST) facilities to the Sludge Receipt & Adjustment Tank (SRAT) located in the 221-S facility. In the SRAT, the sludge slurry is chemically adjusted, concentrated, and sampled. The adjusted sludge slurry is then transferred to the Slurry Mix Evaporator (SME) where it is mixed with glass forming frit, and concentrated further. The concentrated slurry/frit mixture is sampled prior to transfer to the Melter Feed Tank (MFT), which provides continuous feed to a joule heated melter. The resulting molten glass is then poured into stainless steel canisters. Each of the steps described above has a number of supporting unit operations (including mixers, pumps, heat exchangers, ventilation and purge systems, filters, etc.) which are required to process the feed. The intent of this paper is to define key parameters of interest, technical approaches, and support components associated with slurry transport from a vitrification plant processing point of view.

Background

DWPF has been producing radioactive canisters for approximately 12 years and during that time has processed four sludge batches (Sludge Batch (SB) 1A, SB1B, SB2, and SB3) and is currently processing the fifth sludge batch (SB4). SB1A through SB4 have been a blend of two main types of sludge slurries stored at the HLW CST facilities called PUREX[1] and HM[2] (H-Canyon Modified Purex Process). Based on the volumes of each type of sludge slurry blended for a particular batch, a unique set of chemical and physical properties have been noted. This high degree of variability of chemical and physical properties of the prepared sludge batch can lead to processing, sampling, and transport issues in the DWPF. Table 1 provides a list of issues encountered in the DWPF during the processing of each sludge batch. In addition to those issues, transport of sludge slurries throughout the facility is complicated by the complex canyon configuration. In

2.4.1.7 SRS Evaporation Process Principles – Bill Wilmarth

Radioactive Waste Evaporation

Evaporation Process Principles

Evaporation is an operation used to remove a liquid from a solution, suspension, or emulsion by boiling off some of the liquid. It is thus a thermal separation, or thermal concentration, process. The evaporation process is defined as one that starts with a liquid product and ends up with a more concentrated, but still liquid and still pumpable concentrate as the main product from the process. There are actually a few instances where the evaporated, volatile component is the main product, but this will not be discussed that here.

In most cases it is essential that the product is subject to minimal thermal degradation during the evaporation process, requiring that temperature and time exposure must be minimized. This and other requirements brought on by the physical characteristics of the processed product have resulted in the development of a large range of different evaporator types. Additional demands for energy efficiency and minimized environmental impact have driven development toward very innovative plant configurations and equipment design in the commercial sector. In the DOE-EM environment, nuclear design features typically dominate the selection of evaporator methodology and operational characteristics.

Criteria for Selection of Evaporator Plant Concept

During the design of evaporation plants, numerous, sometimes contradictory, requirements have to be considered. They determine which type of construction and arrangement is chosen, and the resulting process and economic data. The most important requirements are as follows:

- Capacity and operational data, including quantities, concentrations, temperatures, annual operating hours, change of product, controls automation, etc.
- Product characteristics, including heat sensitivity, viscosity and flow properties, foaming tendency, fouling and precipitation, boiling behavior, etc.
- Required operating media, such as steam, cooling water, electric power, cleaning agents, spare parts, etc.
- Capital and other financial costs
- Personnel costs for operation and maintenance
- Standards and conditions for manufacture delivery, acceptance, etc.
- Choice of materials of construction and surface finishes
- Site conditions, such as available space, climate (for outdoor sites), connections for energy and product, service platforms, etc.
- Legal regulations covering safety, accident prevention, sound emissions, environmental requirements, and others, depending upon the specific project.

Evaporation Processes Used in Support of the Environmental Management Mission

Evaporation was employed by the early designers and operators of the Savannah River Plant (now called Savannah River Site) and Hanford Reservation to separate water from radioactive waste generated in support of weapons material production. Acidic radioactive wastes generated from plutonium and uranium separation facilities were adjusted with sodium hydroxide or sodium carbonate and sodium nitrite and stored in large carbon steel storage tanks where the waste was allowed to separate into a metal sludge and salt supernate. The early designers and operators of the Hanford and Savannah River Plant quickly realized that considerable cost savings (e.g., fewer storage tanks) could be achieved by evaporating the waste to reduce the total storage inventory.

The SRS HLW tank farm evaporators began operations in the early 1960's (1960 in F Area and 1963 in H Area). The SRS evaporators are horizontal bent tube evaporators. Horizontal bent tube evaporators are best applied for small capacity evaporators, when headroom is limited and for severely scaling services similar to those found in the SRS HLW evaporators1. The design goal of the SRS evaporators was to achieve as high a volume reduction as possible; therefore, the evaporators' systems were designed to produce an immobile "salt cake" by successive evaporations of the concentrated supernate. Fresh supernate was evaporated well past saturation and the concentrated liquor was allowed to cool and separate into salt crystals and a decantable saturated salt supernate as shown in Figure 2.20. The decantable salt supernate was pumped back to the evaporator and further concentrated. The successive evaporation of the decantable saturated liquid was repeated until the HLW storage tanks were filled mostly with salt cake and a small fraction of saturated salt supernate. The waste volume was typically reduced to approximately 1/3 of its original volume. The original sludge fraction was separated from the salt supernate and stored in other HLW tanks.

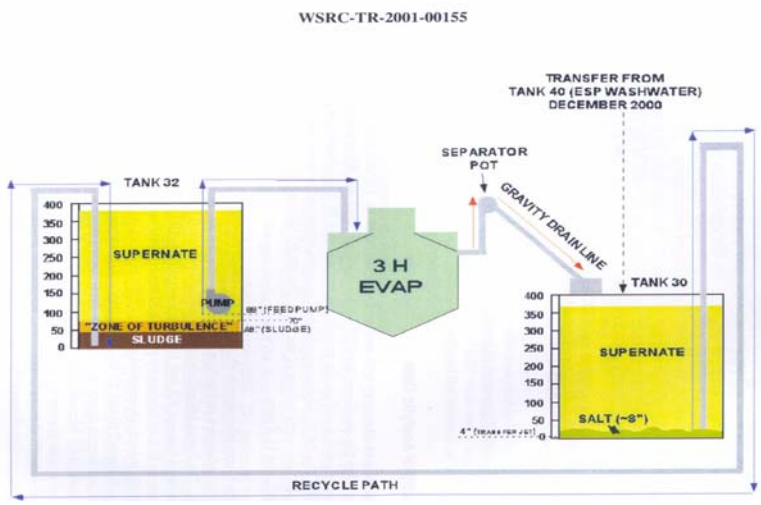

Figure 2.20. SRS 3H Evaporator System Diagram

The Hanford reservation is currently using a vacuum forced circulating evaporator (242-A, startup in 1977) to concentrate HLW. A simplified schematic diagram of the 242-A Evaporator is shown in Figure 2.21 An additional forced circulating evaporator; the 242-S, was shutdown in 1980. Prior to the introduction of forced circulating evaporators on the Hanford site, "In-Tank Solidification" of the waste or evaporation was accomplishment by taking advantage of the radiolytic heat generated from the waste and allowing the

waste to self-boil in the tank. The condensate from these in-tank evaporation systems was treated by ion exchange (if necessary) and pumped to site seepage basins ("cribs"). A small steam heated pot type evaporator (242-T) was also used to evaporate waste in Hanford T Area.

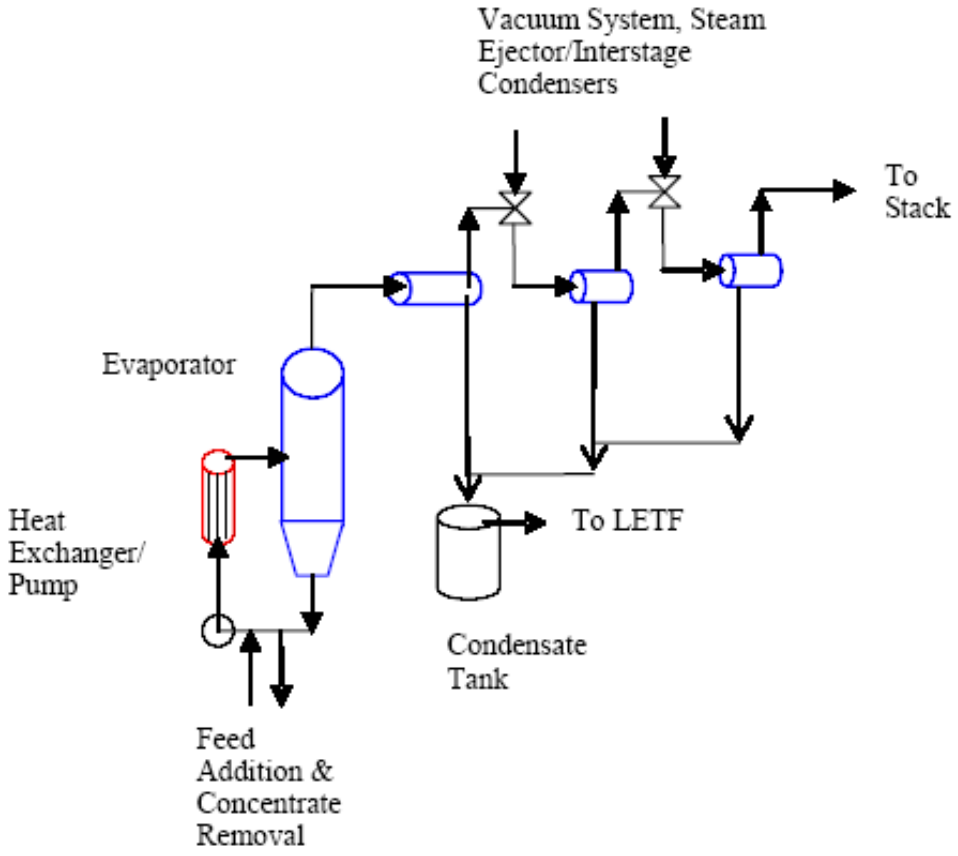

Figure 2.21. Simplified Diagram of the Hanford 242-A Evaporator

The Hanford River Protection Project Waste Treatment Plant (WTP) is currently being designed and constructed near the 200 East Area on the Hanford Reservation. The WTP is designed to pretreat Hanford tank waste to remove strontium, transuranics (TRU), and cesium and then vitrify the treated waste into a LAW borosilicate glass matrix that will be stored on site. The Sr, TRU, and Cs, as well as the insoluble portion of the tank waste, will be vitrified into a HLW borosilicate glass matrix and sent to the US Spent Nuclear Fuel and High Level Defense Waste Repository at Yucca Mountain. Figure 6 shows a simplified flow sheet of the WTP process. Since most of the waste is comprised of water, large forced circulating evaporators will be used to remove water from the WTP and send it to the Hanford Liquid Effluent Treatment Facility (LETF). Additionally, a thermo-siphon evaporator is used to recover nitric acid that is used to elute the cesium removal (ion exchange) columns. Figure 2.22 shows a process flow diagram of the WTP flowsheet.

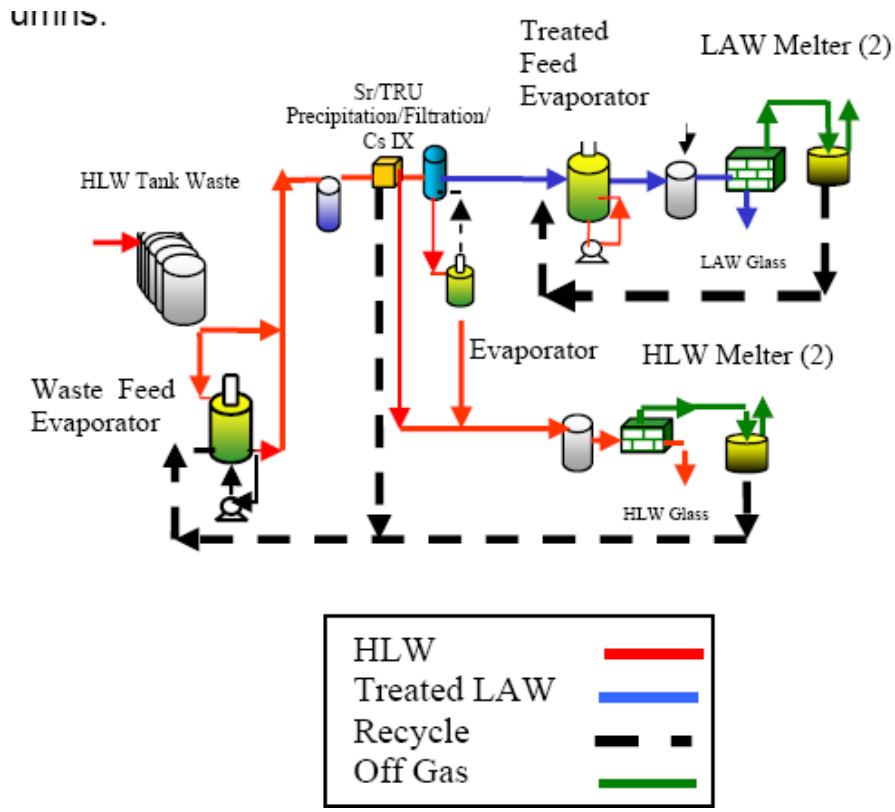

Figure 2.22. Diagram of the WTP Flowsheet which highlights the use of Vacuum Evaporator

Design Features of Evaporator Technologies

Atmospheric Waste Evaporators

For atmospheric evaporators (also called bent tube evaporators), the heat for the evaporation process is supplied by the tube bundle as shown in Figure 2.23. Heat is transferred from the bundle to the waste. The tube bundle actually consists of the tube bundle, an inlet and outlet tube sheet, and an inlet and outlet steam chest. The tube bundle has several tubes (233 for a typical SRS evaporator) located inside the evaporator pot. The ends of the tubes are attached to the inlet and outlet tube sheets. The individual tubes are curved downward from the inlet toward the outlet. This promotes draining condensate from the tubes and allows the tubes to expand when heated without placing undue stress on the evaporator pot.. The tube sheets are attached to and form part of the evaporator pot wall. The inlet and outlet steam chests are located on the outside of the evaporator pot and attached to the inlet and outlet tube sheets. The inlet steam chest directs 150 psig steam into the tube bundle. The outlet steam chest directs condensate from the tube bundle to the backpressure control valve. The condensate outlet pipe originates within the outlet steam chest near the bottom of the chest. The pipe exits the chest at the top and directs condensate to the back pressure valve.

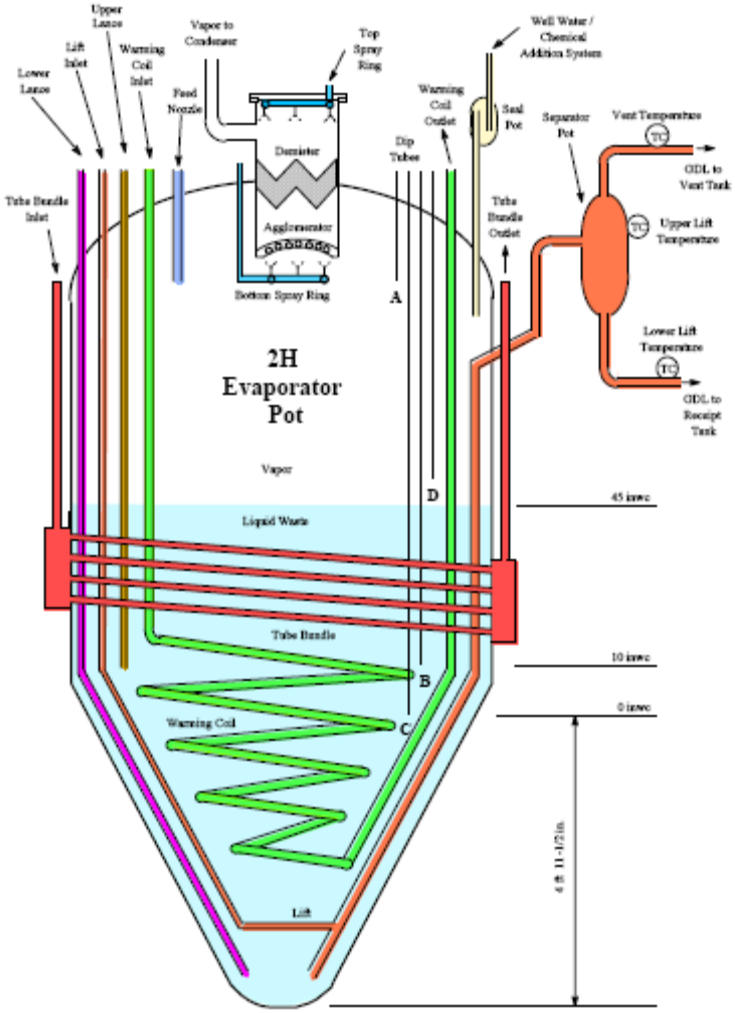

Figure 2.23. Diagram of the SRS Bent-Tube Evaporator Design

During normal operation of the evaporator, feed is constantly added to the evaporator, overheads are being produced constantly and concentrate is being lifted from the evaporator simultaneously. The three streams involved in the concentration process, waste feed, overheads, and concentrate must be balanced to maintain evaporator level and specific gravity within limits. Any change in one stream will have an effect on the other two streams.

Forced Circulation Evaporators

Forced circulation evaporators (Figure 2.24) are used if boiling of the product on the heating surfaces is to be avoided due to the fouling characteristics of the product, or to avoid crystallization and are the benchmark for application at Hanford. The flow velocity in the tubes must be high, and high-capacity pumps are required.

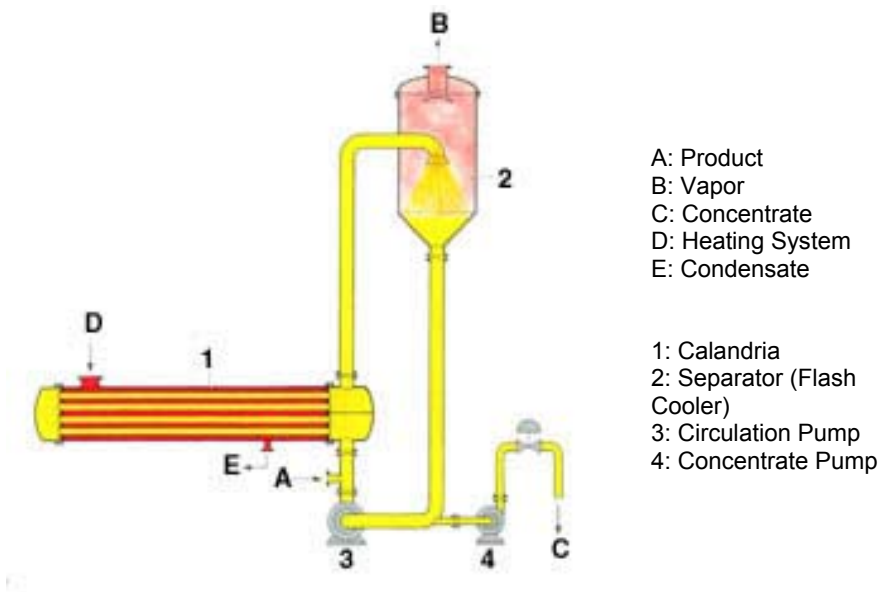

Figure 2.24. Schematic Drawing of a Forced Circulation Evaporator

The circulating liquid is heated when it flows through the heat exchanger and then partially evaporated when the pressure is reduced in the separator, cooling the liquid to the boiling temperature corresponding to this pressure. The liquid is typically heated only a few degrees for each pass through the heat exchanger, which means the recirculation flow rate has to be high.

This type of evaporator is also used in crystallizing applications because no evaporation, and therefore no concentration increase, takes place on the heat transfer surface. Evaporation occurs as the liquid is flash evaporated in the separator/flash vessel. In crystallizer applications this is then where the crystals form, and special separator designs are used to separate crystals from the re-circulated crystal slurry. The heat exchanger (in evaporator parlance sometimes called the "calandria") can be arranged either horizontally or vertically depending on the specific requirements in each case.

Falling Film Evaporators

A **falling film evaporator** is an industrial device to concentrate solutions, especially with heat sensitive components. This technology has not been used extensively in the EM environment; but, the technology does provide an alternative to forced circulation evaporators. In general evaporation takes place inside tubes. But there are also applications were the process fluid is located on the outside tube. The process fluid to be evaporated flows as a continuous film downwards controlled by gravity. The fluid will create a **film** along the inner tube walls, progressing downwards (**falling**) – hence the name. The Fluid distributor has to be designed carefully in order to maintain an even liquid distribution for all vertical in parallel aligned tubes through which the solution falls.

In the majority of applications the heating medium is placed on the outside of the tubes. High heat transfer coefficients are required in order to achieve equally balance heat transfer resistances. Therefore most commonly condensing steam is used as a heating medium. Separation between the liquid phase (the solution) and the gaseous phase takes place in the tubes Increasing vapor velocities downwards to the

bottom of the exchanger increases the sheer force acting on the liquid film and therefore also the velocity of the solution. The result can be a high film velocity and a progressively thinner film resulting in increasing turbulent flow. The combination of these effects allows very high heat transfer coefficients.

The tube side heat transfer coefficient is determined by the hydrodynamic flow conditions of the film. For low mass flows or high viscosities the film can be found of laminar type. Heat transfer is controlled purely by conduction through the film. Therefore in this condition heat transfer decreases with increased mass flow. With increased mass flow the film becomes wavy laminar and with further increased flow turbulent. Under these conditions the heat transfer increases with increased flow. Evaporation takes place at very low mean temperature differences between heating medium and process stream. Typically between 3-6K, therefore these devices are ideal for heat recovery in multi stage processes. A further advantage of the falling film evaporator is the very short residence time of the liquid and the absence of superheating of the same. Not considering the vapor separator, the residence time inside the tubes is measured in seconds, making it ideal for heat-sensitive product such as milk, fruit juice, pharmaceuticals and many others. Falling Film Evaporators are also characterized by very low pressure drops. Therefore they are often used in deep vacuum applications.

Historical Issues in Evaporator Operations

Evaporator Scaling

During July of 1997, [1,2,3] operation of the SRS 242-16H evaporator (2H) was stopped due to low flow through the evaporator concentrate discharge line (gravity drain line or GDL). Video inspection and later characterization revealed the low flow to be caused by accumulation of two solid phases (a nitrated aluminosilicate, $Na_8Al_6Si_6O_{24}(NO_3)_2 \bullet 4H_2O$ and sodium diuranate, $Na_2U_2O_7$. Inspection of the 2H evaporator also revealed the heat transfer surfaces and pot were coated with a sodium aluminosilicate (NAS) and sodium diuranate scale[8,9]. Studies by SRNL revealed that simulated salt supernates could form nitrated aluminosilicates in the presence of silica. The aluminosilicate scale is very similar to that observed in the Bayer aluminum process and paper industry[4,5,6] and was produced by reaction of the aluminate supplied by the canyons and the silicate from the DWPF recycle, specifically the glass formers (frit) carryovers. Additionally, work by SRNL also indicated that if the feed to the evaporator were saturated with soluble uranium, sodium diuranate would precipitate in the evaporator. The accumulation of the sodium diuranate phase, which selectively precipitated with the aluminosilicate phase, caused criticality concerns in the 2H evaporator. Eventually, in October 1999, the 2H evaporator was shutdown due to the presence of a large amount of sodium aluminate scale that contained sodium diuranate.

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[1] W.R. Wilmarth, M.C. Thompson, C.J. Martino, V.H. Dukes, J.T. Mills, C. Boley, and B.L. Lewis, "Nitric Acid Cleaning of a Sodalite – Sodium Diuranate Scale in High Level Waste Evaporators," WSRC-MS-2001-00741, Sep. Sci and Tech., **38**, 3249, 2003.
[2] W.R. Wilmarth, C.J. Coleman, J.C. Hart, and W.T. Boyce, "Characterization of Samples from the 242-16H Evaporator Wall," WSRC-TR-2000-00089, March 20, 2000
[3] W.R. Wilmarth, C.J. Coleman, A.R. Jurgensen, W.M. Smith, J.C. Hart, W.T. Boyce, D. Missmer, and C.M. Conley, "Characterization and Dissolution Studies of Samples from the 242-16H Evaporator," WSRC-TR-2000-00038, Rev. 0, January 31, 2000.
[4] Barnes, M.C.; Addai-Mensah, J.; Gerson, A.R.J., Crystal Growth, 200 (1999), 251-264.
[5] Gasteiger, H.A.; Fredrick, W.J.; Streisel, R.C., J. Eng. Chem. Res., Vol 31, 1992, 1190.
[6] Buhl, J.; Löns, J., J. Alloys and Compounds, 235 (1996), 41.

As a result of the formation of aluminosilicates when elevated concentrations of silica are a concern, SRS changed the operational requirements for the site's High-Level Waste evaporators. Wastes containing high silicon concentrations, e.g., DWPF recycle would be concentrated in the 2H Evaporator. The criticality hazard for the 2H Evaporator was reduced by depleting the U-235 content of the waste below acceptable levels. Waste containing aluminate would be processed in the 2F or 3H Evaporator and acceptance criteria were established to monitor for the possible formation of sodium aluminosilicate.[7] Currently, SRS performs nitric acid cleaning of the 2H Evaporator pot prior to the volume of scale reaching an administrative limit. These restricts limit the 2H availability.

Waste Foaming

Foaming and antifoaming in radioactive waste systems has been studied for a number of years. Advanced antifoam agents have been successfully developed by Illinois Institute of Technology (IIT), tested at a pilot scale, and in radioactive wastes and simulants at SRTC for the DWPF and Salt Disposition Program. The advanced antifoam IIT747 is currently being used at the DWPF. The methodology used to test the foaminess and antifoam effectiveness for the DWPF and Salt Disposition Programs were also used in support of the WTP evaporator R&T program.

Radioactive waste foams are typically stabilized by very small insoluble particles. The solid particles stabilize foaminess in two ways: by adsorption of biphillic particles at the surface of the foam lamella (liquid-gas interface) and by layering of the particles trapped inside the foam lamella. During bubble generation and rise, solid particles organize into a layered structure due to confinement in between each bubble.[8] Peak foaminess occurred well after salt crystals precipitated from the solution thus indicating a particle stabilized foam mechanism as described above. However, Hanford radioactive waste has been shown to foam when boiling is first initiated and no insoluble particles are present in the waste.[9] This indicates the presence of a surface active agent that may be causing foaming at the onset of boiling. Analysis of the data from the evaporation of actual Hanford radioactive tank waste (241-AN102) indicates that foaming occurred at approximately 5 M Na. Additionally, foaming of Hanford waste in the 242-A evaporator has been excessive, causing plant shutdown especially after the waste becomes saturated and solids begin to precipitate.[10]

Toxic Vapor Production

In June of 2001, elevated levels of mercury vapor were discovered in the 3H Evaporator service building during a routine survey. These elevated levels were not expected based on the process model for the system and experience with operating other waste evaporators on site. Liquid samples of the

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[7] W.R. Wilmarth, "Technical Requirements for Dispositioning Tank 40H Decants, SRT-LWP-2001-00032, Rev. 1, March 20, 2001.
[8] S.K. Bindal, A.D. Niklov, D.T. Wasan, D.P Lambert, and D.C. Koopman, 2001,"Foaming in Simulated Radioactive Waste", Environmental Science Technology, **35** pp. 3941-3947.
[9]M.L. Crowder, C.L. Crawford, H.H. Saito, T.B. Calloway, L.V. Gibson, M.A. Burdette, and S.L. Crump, May 2001, "Bench Scale Evaporation of a Large Hanford Envelope C sample (Tank 241-AN102), WSRC-TR-2000-00469 Rev. 1, Westinghouse Savannah River Company, Aiken Sc 29808.
[10]M.D. Guthrie, February 1996, "242-A Campaign 95-1 Post Run Document", WHC-SD-WM-PE-055 Rev. 0, Westinghouse Hanford Company, Richland, WA 99352.

evaporator overheads were analyzed to better understand the mercury vapor results. Analysis of the liquid overheads samples determined the presence of both elemental and organo-mercury species. The level of organo-mercury species in the liquid was unexpected.

Controls were put in place to protect the workers from the elemental mercury vapors and a program was developed to better understand the organomercury species. At the time, there was no routine method available to detect organomercury vapors. An outside laboratory,[11] with extensive experience in measuring low levels of organomercury in vapors and liquids, was contracted to assist with developing a sampling plan to measure concentrations of organomercury in the 3H Evaporator service building.

Once the mercury vapor was detected in June 2001, the Industrial Hygiene staff, in conjunction with Site Medical, reviewed the available data to determine if the health of any site employees had been placed at risk due to elevated mercury vapor levels. Affected employees were given the option to submit bioassay samples (both blood and urine). Several employees did voluntarily submit samples that were tested. In all cases, the test results showed mercury levels that were within the expected ranges for the U.S. population.

Following the discovery of mercury vapor in the 3H service building, several actions were taken to mitigate the situation. Administrative Controls were put in place requiring additional personal protective equipment (PPE) when working in areas where the mercury vapors were found and in areas where the vapors might be found. Routine monitoring of mercury concentrations in the air was initiated. A mercury hazards review was initiated to develop a better understanding of how the mercury vapor was moving through the system and to determine what actions could be taken to minimize the release of mercury. A temporary modification was installed in December 2001 to divert the overheads receipt tanks vents directly into the building exhaust system. Design was initiated to permanently modify the ventilation system for the overheads tanks and to provide ventilated sampling stations.

The 3H Evaporator was shut down for an extended outage between May and September 2002 during that time laboratory studies confirmed the formation under simulated waste conditions.[12] Once full system operations were resumed in November 2002, a series of liquid and vapor samples were collected in the 3H Evaporator service building. Vapor samples collected during this period showed dimethylmercury (a more volatile organomercury) vapor concentrations that were within occupational exposure limits for the operations being performed, but were above limits established for continuous occupancy. As a result of these sample results, additional administrative controls were established.

Sampling continued in December of 2002. During this sampling, elevated levels of dimethyl mercury (DMHg) were found at an alternate overheads sample location. The concentration found at this location was higher than the Occupational Safety and Health Administration (OSHA) ceiling limit. Based on this result, immediate actions were taken to add additional administrative controls and personal protective equipment requirements for sampling activities at the alternate sample locations. Individuals involved in the sampling activities during December submitted bioassay samples for medical surveillance (laboratory analysis of blood and urine). The sample results showed no indications of mercury exposure and no

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[11] N.S. Bloom, E. van der Geest, E.M. Prestbo, W. Wilmarth, and D. Thaxton, "Formation and Degradation of $(CH_3)_2Hg$ in Nuclear Waste Tanks," Proceedings of the 7th International World Mercury Conference, RMZ.
[12] W.R. Wilmarth and S.W. Rosencrance, "Studies of Mercury in High Level Waste Evaporator Systems," WSRC-TR-2003-00238, June 6, 2003.

levels of mercury in their system above normal levels. In addition, 63 people who had been previously involved in evolutions in the 3H Evaporator service building voluntarily submitted a total of 74 bioassay samples for mercury analysis. The results of these analyses also indicated no mercury exposure and no levels of mercury in their system above normal levels.

The sampling program was expanded in February and March 2003 to include the 2H and 2F Evaporators and various locations of the Effluent Treatment Facility. No additional personnel exposures resulted from these sampling activities since breathing air was supplied to affected personnel and additional administrative controls were enforced. The results of this sampling showed elevated levels of DMHg at the 2H evaporator and at some locations at the Effluent Treatment Facility. Modifications to reduce concentrations at these locations have been completed. However, specific maintenance and operations activities still require additional administrative controls to ensure personnel exposure is avoided.

Crystallization is the (natural or artificial) process of formation of solid crystals precipitating from a uniform solution or melt, or more rarely deposited directly from a gas. Crystallization is also a chemical solid-liquid separation technique, in which mass transfer of a solute from the liquid solution to a pure solid crystalline phase occurs.

The crystallization process consists of two major events, nucleation and crystal growth. Nucleation is the step where the solute molecules dispersed in the solvent start to gather into clusters, on the nanometer scale (elevating solute concentration in a small region), that becomes stable under the current operating conditions. These stable clusters constitute the nuclei. However when the clusters are not stable, they redissolve. Therefore, the clusters need to reach a critical size in order to become stable nuclei. Such critical size is dictated by the operating conditions (temperature, supersaturation, etc.). It is at the stage of nucleation that the atoms arrange in a defined and periodic manner that defines the crystal structure — note that "crystal structure" is a special term that refers to the relative arrangement of the atoms, not the macroscopic properties of the crystal (size and shape), although those are a result of the internal crystal structure.

The *crystal growth* is the subsequent growth of the nuclei that succeed in achieving the critical cluster size. Nucleation and growth continue to occur simultaneously while the supersaturation exists. Supersaturation is the driving force of the crystallization, hence the rate of nucleation and growth is driven by the existing supersaturation in the solution. Depending upon the conditions, either nucleation or growth may be predominant over the other, and as a result, crystals with different sizes and shapes are obtained (control of crystal size and shape constitutes one of the main challenges in industrial manufacturing, such as for pharmaceuticals). Once the supersaturation is exhausted, the solid-liquid system reaches equilibrium and the crystallization is complete, unless the operating conditions are modified from equilibrium so as to supersaturate the solution again.

Equipment for Crystallization

1. *Tank crystallizers*. Tank crystallization is an old method still used in some specialized cases. Saturated solutions, in tank crystallization, are allowed to cool in open tanks. After a period of time the mother liquid is drained and the crystals removed. Nucleation and size of crystals are difficult to control. Typically, labor costs are very high.

2. *Scraped surface crystallizers*. One type of scraped surface crystallizer is the Swenson-Walker crystallizer, which consists of an open trough 0.6m wide with a semicircular bottom having a cooling jacket outside. A slow-speed spiral agitator rotates and suspends the growing crystals on turning. The blades pass close to the wall and break off any deposits of crystals on the cooled wall. The product generally has a somewhat wide crystal-size distribution.
3. *Double-pipe scraped surface crystallizer*. Also called a *votator*, this type of crystallizer is used in crystallizing ice cream and plasticizing margarine. Cooling water passes in the annular space. An internal agitator is fitted with spring-loaded scrapers that wipe the wall and provide good heat-transfer coefficients.
4. *Circulating-liquid evaporator-crystallizer*. Also called *Oslo crystallizer*. Here supersaturation is reached by evaporation. The circulating liquid is drawn by the screw pump down inside the tube side of the condensing stream heater. The heated liquid then flows into the vapor space, where flash evaporation occurs, giving some supersaturation.The vapor leaving is condensed. The supersaturated liquid flows down the downflow tube and then up through the bed of fluidized and agitated crystals, which are growing in size. The leaving saturated liquid then goes back as a recycle stream to the heater, where it is joined by the entering fluid. The larger crystals settle out and slurry of crystals and mother liquid is withdrawn as a product.
5. *Circulating-magma vacuum crystallizer*. The magma or suspension of crystals is circulated out of the main body through a circulating pipe by a screw pump. The magma flows though a heater, where its temperature is raised 2-6 K. The heated liquor then mixes with body slurry and boiling occurs at the liquid surface. This causes supersaturation in the swirling liquid near the surface, which deposits in the swirling suspended crystals until they leave again via the circulating pipe. The vapors leave through the top. A steam-jet ejector provides vacuum.
6. *Continuous oscillatory baffled crystallizer (COBC™)*. The COBC™ is a tubular baffled crystallizer that offers plug flow under laminar flow conditions (low flow rates) with superior heat transfer coefficient, allowing controlled cooling profiles, e.g. linear, parabolic, discontinued, step-wise or any type, to be achieved. This gives much better control over crystal size, morphology and consistent crystal products. For further information see oscillatory baffled reactor.

2.4.1.8 Hanford Gas Generation Issues Related to Waste processing – Sam Bryan

Gas Generation from Hanford Waste

PNNL has had considerable experience in gas generation measurements and assessment of flammable gas accumulation within Hanford related processes. Information has been gained from the results of laboratory studies with simulated wastes (Bryan and Pederson 1994, 1995); laboratory studies with actual waste core samples (Bryan et al 1996a, 199b, 2000, Bryan and King 1998a, 1998b; King and Bryan 1998; King et al 1997, Pederson and Bryan 1996), ; studies of thermal and radiolytic reactions in the gas phase (Bryan and Pederson 1995, 1996); gas solubility evaluations (Norton and Pederson 1995, 1996); and in-tank gas composition data (Mahoney et al 1999; 2000).

The thermally activated and radiolytic rates of gas generation were established for samples drawn from multiple Hanford Tanks, including A-101, AW-101, S-102, S-106, SY-103, and U-103. Studies with actual wastes provide an important complement to mechanistic investigations using simulated

wastes (Barefield et al. 1995, 1996; Bryan et al. 1992, 1994, 1996; Delegard 1980; Meisel et al. 1991, 1993). These studies have established that gas generation rates are sensitive to a number of parameters, including the identity of organic solutes and the concentrations of nitrite, hydroxide, aluminate, and various minor and trace components (Bryan et al. 1992, 1994, 1996; Delegard 1980; Herting 1992a, 1992b; Person 1996). Based on thermal and radiolytic activation parameters measured by Bryan et al (1996a, 199b), a predictive model has been developed to estimate the flammable gas generation yield from Hanford site waste storage tanks (Bryan and Pederson 1996, 2000; Hu 1997, 2000, 2002).

Gas Retention in Hanford Double Shell Tank (DST) Wastes

Within the nonconvecting layer of Hanford wastes, much of the gas that is produced is trapped as bubbles (Gauglitz et al. 1996). The gas retention and release behaviors of Hanford double shell tanks (DSTs) on the Flammable Gas Watch List (FGWL), AN-103, AN-104, AN-105, AW-101, SY-101 and SY-103, were characterized in detail using the ball rheometer and void fraction instrument (VFI) (Meyer et al 1997, Mahoney et al 1999, 2000). The objectives of this work centered on understanding the gas retention and release behavior to enable predictive modeling to reduce future occurrences of these events.

Gas Generation from WTP Related Wastes and Processes

In support of Bechtel National, Inc., Research and Technology Department to provide design tools for estimating rates of hydrogen gas generation by waste streams in the Waste Treatment and Immobilization Plant (WTP), PNNL was commissioned to study the gas generation behavior from WTP (Bryan et al 2004). A model for predicting hydrogen generation rates in Hanford's stored tank wastes has been developed by Bryan and Pederson (1996) and Hu (1997, 2000, 2002). However, as the waste is processed in the WTP, its properties and conditions are subject to change. These included the effects of beta/gamma and alpha radiolysis and of adding hydroxide, permanganate, air, sugar, and glass-forming materials at various stages in the processing prior to the vitrification step. While many issues could be resolved through technical evaluations of existing information (Bryan et al 2004; Camaioni and Stock 2003), some issues required experimental testing (Bryan et al 2002, 2003, 2004; Hallen et al 2000, 2002a, 2002b). The radiolytic effects on organic ion exchanger systems was studied to gain understanding of the dose consequence on ion-exchanger capacity and ion-exchanger lifetime, as well as a safety concern for flammable gas generation due to radiolysis effects (Arm, et al 2003; Brown, et al 1995; Bryan, et al 1994; Carlson, et al 1995; Kurath, et al 1994).

Gas Generation from Hanford K-Basin Sludge

The path forward for managing Hanford K Basin sludge calls for it to be packaged, shipped, and stored at T Plant until final processing at a future date. An important consideration for the design and cost of retrieval, transportation, and storage systems is the potential for heat and gas generation through oxidation reactions between uranium metal and water. A series of three studies commissioned by Fluor Hanford was performed at the Pacific Northwest National Laboratory (PNNL) to aid the design and understanding the needs for packaging, shipping and storage of Hanford K Basin sludge. The first test series (Series I; Delegard et al. 2000) focused on gas generation from K East (KE) Basin floor and canister sludge (size-fractionated and unfractionated samples collected using a consolidated sampling technique. The second series (Series II; Bryan et al. 2001) examined the gas generation behavior of KE Basin floor, pit, and canister sludge. Mixed and unmixed and fractionated KE canister sludge materials

were tested, along with floor and pit sludge from areas in the KE Basin not previously sampled. The third study in the series (Series III; Schmidt et al 2003), describes work to assess corrosion and gas generation from irradiated metallic uranium particles (fuel particles) with and without sludge addition. The sludge material used in all three test series was saturated with water, consistent with sludge management plans. The work for all three test series was conducted under the direction of the Spent Nuclear Fuel (SNF) Sludge Handling Project managed by Fluor Hanford.

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2.4.1.9 Lessons Learned Cold Testing – Dave Dickey

Erosion Issues with Pulse Jet Mixers

Although comprehensive calculations of erosion wear based on literature references have been done for PJM's (24590-WTP-MOC-50-00004), the particle size basis is the 11 μm median value for the waste stream. The justification is conservative if this median particle size is appropriate for design. The primary reference, (FanAiming, et al.) for the erosion wear is also based on small particles (200 mesh, less than 74 μm). That reference predicts a factor of two (2) difference between impingement erosion and parallel erosion. Because large particles are less likely to follow the fluid with the change in direction and more likely to strike the impinged surface, the impingement factor could be much larger. Similarly, if the impact of particle diameter on erosion has an exponent of about two (2), as from the Karabelas reference, small quantities of upper 95% or 99% mean or median particle sizes could increase erosion. Considering the upper 95% mean particle size of 140 μm (24590-WTP-RPT-M-05-001) instead of the median size of 11 μm, erosion rates could be more than 30 times greater than currently predicted.

When the erosion wear estimates based solely on paper studies were called into question by the EFRT for the Hanford WTP, an Issue Closure study, M2 – Mixing Vessel Erosion, was initiated. From several knowledgeable sources, the only adequate predictions of erosion wear should be based on experimental investigations with appropriate particle simulants at similar impingement angles. The initial tests found a significant increase in erosion rates over those predicted by the paper study. Wear plates with hard metal coating are now being considered to improve the wear characteristics of vessel bottoms under PJM discharge points.

The EFRT report concluded that, "it (was) not clear that the operator of the WTP (could) provide reliable warning of erosive wear during radioactive operations. Even if a problem were discovered before it became catastrophic, it (was) not clear how the facility operator could respond without a lengthy interruption of waste processing." Hopefully, an experimental investigation will avoid erosion problems with the bottoms in PJM vessels.

Fernald Closure Project – Phil McGinnis

DOE had a Technology Assistance effort to support the Fernald Closure project. I lead a team of engineers and chemists from ORNL, PNNL, INL, Mississippi State University, North Dakota EERC, and industry to test the piping flow loop. We found the design for the facility incorporated piping which was too large, in a desire to be conservative. Testing in an existing flow loop at ORNL of the design proved the surrogate (provided by PNNL) would not stay suspended and caused line pluggage. The design was modified before installation and this problem did not arise in the operation.

The clarifier used for the Fernald processing was a vertical clarifier from EIMCO which took feed at 4% solids and provided a uniform feed of 50% solids. The material from the clarifier was to be pumped in batches to the grout feed tank for treatment. The original design called for use of a Rotary Lobe positive displacement pump. ORNL set this up in the existing flow loop and the pump failed within 8 hours of testing due to erosion and the use of tight tolerance in the pump. A series of 6 pumps were tested at ORNL and MSU in order to determine an acceptable solution. The positive displacement pumps using progressive cavity, rotary lobe, peristaltic, and gear all failed. This problem was solved by using a hardened centrifugal pump with a recycle back to the feed of the clarifier. A diverter valve in the recirculation line was used to divert a portion of the feed into one of the three grout feed tanks while the majority of the sludge continued in the recirculation mode. The clarifier capacity was sufficient to accept this additional load.

Another issue was the measurement of the density and the radionuclides concentration of the feed. These parameters were important for calculation of the batch size and the amount of fixative to be added to the mixer. The coriolis meter failed quickly due to the thin walls, We tested several instruments, finally selecting a microwave unit which is now being selected for use at Hanford. The radionuclide problem was solved by developing a gamma attenuation monitor which was developed at INL and built by Canberra.

This testing cost on the order of $1 to $2 M. The documented cost savings for the can loading was at least $75M. The cost of avoidance of lost time would have been much more than that.

A Quantitative Assessment of R&D Requirements for Solids Processing Technology

Edward W. Merrow – RAND / R-3216-DOE / PSSP – July 1986

Summary

Plants that process or produce solid materials, such as metal-ore processors, resins plants, calciners, and synthetic fuels plants, consistently have poorer performance than plants that process liquids and gases. The failure of many solids processing plants to perform efficiently has been uniform over time-new plants show no tendency toward better performance. The present study was undertaken to attempt to identify the reasons for poor performance of solids processing plants and thereby to suggest priorities for research and development (R&D).

We began by expanding an existing database on solids processing facilities to include information on 40 commercial plants, including data on the R&D programs that preceded the design of the facilities. We then performed a statistical analysis of plant performance and the relationship between plant performance and the R&D programs. This approach has several significant advantages over the more conventional method of simply soliciting expert opinion:

- It assures that the R&D priorities will be determined by the problems actually experienced, rather than by academic interest.
- It is considerably less subjective than expert opinion, and the results are therefore less likely to be biased.
- It is reproducible and tied to an explicit figure of merit, plant performance.

Our analysis revealed that the key performance problems in solids processing plants result from physical and mechanical difficulties rather than problems of process chemistry. The R&D needs in the process chemistry areas are being identified and tackled successfully, but problems associated with materials handling, corrosion and abrasion, and solids behavior are not being resolved. The failures in the area of waste handling are particularly pronounced and have led to a number of complete plant failures.

The failure of solids plants performance is closely correlated with incomplete knowledge of heat and material balances, that is, the equations that establish heat and mass flows throughout a plant for design purposes. Poor knowledge of these balances in solids processing is related to difficulties in applying prior experience to new facilities, and this in turn reflects a poor state of basic knowledge about solids.

2.4.1.10 Idaho Lessons Learned-Impact of Radiation on Process – Rick Demmer

Understand Impacts of Radiation on Process (ROVER) Rick Demmer

Irradiated materials do not always behave in the same chemically and physically as unirradiated, dummy or cold pilot scale solutions. Even though the solutions are chemically identical, changes occur in the “real” materials that have been through a reactor.

One example of this was the ROVER head end/dissolution process operated at the Idaho Chemical Processing Plant (ICPP) uranium reprocessing facility from 1982 through 1985. This happened to be a positive example as the irradiated fuel was less difficult to process than the cold pilot scale facility, using unirradiated fuel, indicated.

The ROVER reactors were prototype rockets that would be used for propulsion in spacecraft. Several spacecraft reactors were built and tested at Los Alamos between 1955 and 1972. The specific type of reactor fuel that was reprocessed at the ICPP was a graphite fuel with uranium carbide embedded in a graphite matrix fuel rod that was hexagonal, measuring about 1" on a side and was about 6' long.

Because of the high enrichment and low burnup of the ROVER fuel it was very advantageous to recover the uranium. Highly enriched uranium was very valuable in the 1970s and 1980s. As the ICPP was designed to recover uranium from an aqueous, acid dissolved metallic reactor rods. ROVER rods were not compatible with the typical dissolution and processing scheme. Development was performed on the fuel matrix and a process was designed to combust the graphite and uranium carbide in a burner and leach the ash for uranium. It could then be chemically adjusted and fed directly to the ICPP separations process.

Pilot plant tests with unirradiated fuels determined that approximately 8 hours would be required for each fuel rod batch to complete the combustion and leach process. The "hot" processing was scheduled for about five years to accomplish these tasks. A period of "cold" testing was completed on the full scale equipment and the process was validated. But as irradiated material was introduced it became obvious that it completed the combustion and leach much quicker than the unirradiated material. In fact, batch processing times were nearly one-quarter of the cold material. While some of this may be attributed to gaining experience and progressing along the "learning curve", the engineers were astonished that the irradiated material was actually being processed more rapidly. No one knew precisely why, but it was a happy occurrence.

Of course, no good deed goes unpunished and the ROVER good fortune was no exception. The first problem with ROVER was that a system of gloveboxes specifically designed for the chemical analysis of the dissolved solutions was not capable of being used with the irradiated fuel. Even though the fuel material had aged since the nuclear reaction, thereby eliminating the short lived highly energetic radionuclides, the material was about 10 times more radioactive than physics models had predicted. One week after "hot" startup, the radiation fields were far too great to allow chemical analysis in the expensive set of gloveboxes. Since all the cold trials and training had been performed in the gloveboxes, and no preparations had been made process the sample remotely, there was a very rapid, though successful change in plans.

A second problem that occurred in processing the ROVER fuel was that not all the material being processed was fuel. Some excursions had occurred during testing at Los Alamos and some rocket engine debris had to be retrieved from the desert. Among the material in these broken assemblies were rocks and sand. These did not process as readily as the graphite fuel matrix. Fortunately, the fluidized ash processing equipment was robust enough to allow these items to exit without serious problems.

A different issue with irradiated fuel that arose at the ICPP was the presence of undissolvable solids. During the nuclear reaction, fission fragments accumulate. Some of the fission fragments are noble metals, also called "Fissium". Fissium is mainly composed of ruthenium, which produced fine black

solids that quickly settle and are relatively easy to suspend. Some fuels (EBR-II) have insoluble Fissium solids purposely doped into the fuel matrix. Undissolvable solids produced in the nuclear reaction were a small but troublesome part of the ICPP dissolution process. These solids could plug equipment, disturb extraction processes and ultimately become sludge in the tank farm. Fortunately, they were typically a small portion (less than 1%) of the overall mass of the fuel assembly.

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2.4.1.11 External Technical Reviews – Herb Sutter

The National Academies of Science (NAS) 2007 report, Assessment of the Results of External Independent Reviews for U. S. Department of Energy Projects, concluded that projects benefit from the effort expended in preparing and conducting external independent reviews and independent project reviews. This benefit increases as the size, complexity, and inherent risks of the project increase.

External and internal reviews can provide pertinent information for DOE-EM to assess technical risk associated with projects and develop strategies for reducing the technical risk, and provide technical information needed to support critical project decisions. Technical risk reduction increases the probability of successful implementation of technical scope.

Three key objectives of reviews are:

1. To determine if the technology, process, system, or design under review will meet project objectives and requirements,
2. To identify any issues (showstoppers) preventing successful implementation of the technology, process, system, or design under review, and
3. To identify issues or data needed to support critical or other project or program decisions.

EM has conducted a variety of technical reviews including:

- Contractor Review: – CRs are requested, organized and conducted by the contractor. The External Flowsheet Review was a detailed and successful CR recently conducted on the Hanford Waste Treatment Plant Project.
- External Technical Reviews – ETRs are independent reviews requested by the project or EM management. They are organized by EM-20 and may focus on a single technical issue or a complete project. ETRs have been conducted on a number of projects including the Demonstration Bulk Vitrification System Project at Hanford and the Tank 48 Project at Savannah River
- Technical Readiness Assessment – TRAs are independent reviews requested by the project or EM management. Initial TRAs were organized by the projects, but EM-20 has recently assumed responsibility for organization. TRAs employ an evaluation formalism developed by NASA and used

by the DoD to evaluate the technical maturity of all elements of a project. Guidance under development by EM-20 is expected to require TRAs prior to CD-1 and CD-2. TRAs have been conducted on the Waste Treatment Plant and K-Basins Projects at Hanford and Tank 48 Project at Savannah River.

Lessons Learned

1. Early and Often: Reviews, especially external reviews, should be conducted early on and throughout a project, at a minimum during conceptual design/technology down select and prior to, or in, the early stages of final design. The K-Basins TRA resulted in a complete rethinking of the technical approach. Fortunately the review was conducted early on in the project before project funding and direction were locked down.
2. Structure, Reproducibility, and Objectivity: The process should be structured (see EM-20 guidance for ETRs and TRAs). Independent reviews are the best way to ensure objectivity.
3. Relevant Environment and Project Requirements: One of the temptations in technology development is to accept a technology that has worked well in another project as mature and ready for insertion without examining the differences in project requirements and relevant environments. Often these differences prove fatal to technology application. Technologies must be tested in a "relevant environment" and meet project specific standards of performance. The specification of the relevant environment requires that the input to, and output from, the technology system/process be clearly defined. This means, for example, that the physical and chemical properties of the waste being treated must be thoroughly characterized including waste variability, and that the required characteristics of the treated waste be specified. It is also necessary to determine which waste properties are critical for the makeup of realistic simulants. If the wastes are variable, testing should be carried out on a complete range of wastes and simulants. Clear definition of system/process requirements is critical. The WTP TRA encountered a number of requirements that were overly restrictive. Trying to meet these, in some cases, virtually impossible requirements would have required major commitments of time and money for technology development. Reevaluation of the requirements led to much simpler and less costly technology solutions.
4. Mature All Technology Systems: Waste processing typically consists of a number of technology systems linked together. The tendency is to focus technology development on a core technology and ignore peripheral technologies. For example, Fluidized Bed Steam Reforming (FBSR) was assessed for treatment of Tank 48 waste. Most of the technology development work for this application had focused on the reactor system. However, little attention had been paid to the product handling system. Maturation of the product handling system does not appear to be difficult, but it clearly will require additional testing and development. Failure to mature peripheral systems can lead to major problems in process operation.
5. Assess Independently, Plan as a Team: Independent assessment is recommended. The assessment team should consist of technology experts not connected to the project i.e., DOE and contractor personnel involved in the project should not be part of the assessment team. However, once the assessment is completed, the development of technology development plans and strategies should be a combined DOE/contractor exercise. ORP is using a Technology Steering Group (TSG) consisting of high level DOE and Contractor personnel to oversee technology development and implementation. The TSG has greatly improved the quality of the planning and the speed with which the plans and strategies are approved and implemented.

6. Develop A Plan To Reduce Project Risk: An assessment is of little use if it sits on the shelf and does not result in detailed plans and strategies to complete technology development and reduce project risk.
7. Documentation: Assessor "due diligence," document examination is vital to the process. It was not unusual for the assessors to determine that statements made during the assessment Q/A sessions were not backed up by project documentation. Technology development plans should be well documented.
8. Test a Prototypical System: All technology components should be tested, preferably in a complete, prototypical system. The WTP LAW melter off gas system was tested in prototypical configuration during melter development. This simplified assessment of off gas technology by allowing evaluation of the entire system as a single technology. If prototypical test results were not available, each off gas component would have had to be evaluated separately.
9. Flowsheet Evaluation is a Challenge: The WTP flowsheet consists of many technology systems and many interfaces. Although each system was assessed during the TRA, no useful way was found for assessment of the entire flowsheet as a unified whole to determine if project goals can be met. To some extent this is more a design question than a technology maturity question. Nevertheless, much thought and development needs to go into flowsheet evaluation

2.4.1.12 Idaho Lessons Learned-Knowledge Center – Rick Demmer

A Knowledge Center is being developed for the DOE tank farm processing community. DOE-EM (21) along with NuVision and representatives from the DOE sites will soon develop a system to access the wealth of information available from decades of tank farm use and processing. Conceptually this is an interactive, web-based system where the DOE engineering community may retrieve tank/processing documentation or ask questions that will be directed to subject matter experts throughout the DOE Complex. A similar D&D Knowledge Center is currently being developed by Dr. Leo Lagos, Applied Research Center of the Florida International University. The tank/processing knowledge center is funded to begin development in FY-08.

There are sources currently available to help provide help in tank and processing related issues. One source is the DOE Office of Science and Technology Information. If you have a question on a processing aspect you may go to the OSTI energy citations database (http://www.osti.gov/energycitations/) and search for information. The OSTI is a reasonably thorough catalogue of DOE site reports, citations and references. Another resource available would be the subject matter experts at the Pipeline Workshop.

A general internet search also produces fairly reliable information and turns up contacts that can be networked to find true subject matter experts in this field. One of the advantages that we DOE site types have is access to very powerful search engines maintained at the site libraries. Keyword searches often return hundreds of references; the problem then being weeding the list down to a manageable few that give the requested information. Working with the INL site library (for instance) gives access to the most sophisticated searches (far more powerful than internet searches) and ready access to references through interlibrary loans.

2.5 Special Breakout Sessions

2.5.1 Retrieval – Mobilization to Overcome Shear Strength

On day two, there was a special session within retrieval related to mobilization needed to overcome shear strength that was moderated by David Boger. The understanding or lack thereof of physical properties of the waste became the main topic of much of the discussion during this special session. One of the questions posed by Professor Boger from the University of Melbourne was "*what properties of the waste should you know before waste removal begins*?"

This question resulted in much discussion about the reality of tank waste retrieval as it exists at the Hanford and SRS sites today. The premise of the question was that if we know more about the waste and its physical behavior, then we will be more successful in fielding appropriate technologies for removing it and getting it out of the underground storage tanks for transport and processing for waste disposal. The general conclusion from the participants from the sites were that roughly ten percent of the waste volume is where all the trouble really is, in terms of waste retrieval without major concerns to waste transport or waste processing and treatment. These statements were based upon what the sites have experienced in the past during sluicing of single shell tanks during the era of operations. As noted, to date, there has not been significant experience with tanks that have been emptied and closed. The reality of funding priorities at the sites is that until there are significant problems that occur clearly demonstrating these problems, then the cost of obtaining representative samples (or determining properties either in-situ or via core samples) is prohibitive.

The site baselines have been selected, and if there were additional sampling, or the affordability to sample "today", it would likely have minimal impact on current retrieval baseline technologies. The overlying feeling of the site staff was that getting the waste out of the tanks is difficult, and if we can do that, then other things can be done in real time to ensure that waste can be transported to the next step of the process. One of the problems with knowing more information about the waste is that representative samples would have to be taken and analyzed to understand the waste properties. Costs to perform representative sampling are prohibitive, and any samples taken really do not tell what the in-situ properties of the waste truly are. In general, the sites continue to move in a direction of not performing major sampling campaigns until there is a true problem that could stop retrieval operations. While the consensus of the group was that additional physical properties would be beneficial, there is minimal support to performing theses property measurements.

2.5.2 Transport – "Open Channel Flow" and "Critical Velocity w/ Viscosity Adjustment"

A special breakout session on gravity flow systems was held and moderated by Robert Cooke. Mark Hall described the design guide for determining slopes for process drain/overflow lines within the Waste Treatment and Immobilization Plant (WTP). He began with a review of the process lines affected by the guide as well as anticipate waste properties. He explained that three slopes are to be implemented in the WTP with rise to run ratios of 1:100, 1:50, and 1:20. He then outlined the technical approach of how the system flow rates and velocities are determined. An effective pipe diameter is then determined. The

diameter and slurry propertied (e.g. particle size, density, and viscosity) are then input into equations for conventional pipeline transport. The result is a prediction on the critical velocity of the open channel system.

2.5.3 Processing – Pulse Jet Mixers (PJM)

A special breakout session on pulse jet mixers was held and moderated by Art Etchells. This session was attended by sixteen workshop participants.

Perry Meyer described the work PNNL is doing on mixing with PJMs for Bechtel and the WTP project at Hanford. He began with a review of the work on non-Newtonian fluids and the correlation derived for getting complete tank mobility. This could be achieved by PJMs alone but the cost in additional air was prohibitive. A hybrid design of PJMs mixing the lower part of the vessel and sparged air mixing the upper part of the vessel was developed and will be implemented. He discussed the current work on the suspension of discrete particulate solids looking at off bottom suspension and vertical distribution as measured by cloud height. This presentation is included in the proceedings.

When asked representatives from Nuvision the current suppliers of PJM technology said they knew of no equivalent information in their history.

Sam Shicks of Parsons gave a preview of his talk on re-suspended a bed of fine fluffy solids with devices similar to the PJMs dubbed Air Pulse Agitators (APA) by Parsons. This work was done in support of the proposed Salt Waste Processing Facility (SWPF) at Savannah River. He was able to use the real material in two scale tanks and measured the time it took to achieve concentration uniformity of the solids. A correlation of velocity and various geometric parameters was obtained. Attendees from SRNL will work with Parsons to better characterize the settling bed and the final slurry. His work was presented later in the workshop and is included in the proceedings.

There was a general discussion of what data and models are available for various process results for PJMs and the need to develop consistency and a good technical basis. Some of the information that exists is considered proprietary by the possessors.

The only action item that came out of the session was a workshop on PJM mixing would be most useful involving expertise from both the UK and the USA. Art Etchells is to follow up on setting such a workshop up this year.

2.5.3.1 Pulse Jet Mixing Work at PNNL

Extensive work on pulse jet mixing systems in support of the Hanford Waste Treatment Plant has been conducted at PNNL. This ongoing work is primarily experimental, with an emphasis on establishing the technical basis for mixing system designs, operation, and safety-related functions such as flammable gas management. Test equipment has included a prototypic 12.5ft. diameter (12,000 gallons) vessel with four PJMs operated by pneumatic jet pump pairs. Also, clear acrylic vessels ranging from 2ft. – 8ft. are utilized. Some of these systems are operated by pneumatic drive systems (utilizing vacuum instead of jet pump eductors) while others utilize closed-loop pump systems that mimic pulse jet operation by producing an intermittent jet.

Important Mixing Parameters

Pulse jet mixing is governed by geometric, operational, and physical parameters. Geometric parameters include the number of jets (N), jet nozzle diameter (d), vessel diameter (D) or volume (V), pulse tube volume (V_{PT}), and the radial positions of the jets. The jet discharge angle and the impingement angel with the dish bottom can also be important as these affect the primary flow distribution patterns. The *jet density* $\phi_d = N(d/D)^2$ is a useful parameter which combines the effects of multiple jets, allowing relative comparison of different mixing system designs.

Important operation parameters include the jet velocity (u) and relative fill level (H/D). The velocity pulse is characterized by the discharge drive time (t_D), and the cycle time (t_C). Alternatively, the unsteady mixing effects are described by the *pulse volume fraction* $\phi_P = NV_{PT}/D^3$ and the *duty cycle* $DC = t_D/t_C$.

The important physical parameters for cohesive slurries are the bulk slurry density (ρ), the yield stress (τ_y), and the high shear consistency (κ). If the slurry has time-dependent properties (such as increasing shear strength when stationary) then the shear strength (τ_s) and time constant (t_τ) may be important. The important properties for non-cohesive slurries are the liquid density (ρ) and kinematic viscosity (ν), the solids density (ρ_s) or the density ratio $s = \rho_s/\rho$, the solids size (d_s) or size ratio (ds/D). For solids with a distribution of sizes, typically d_{95} is used. The solids volume fraction (ϕ_s) is also important. Recent testing has indicated that the settling velocity (u_s), which depends on both liquid and solid properties, is a very useful parameter, especially for mixing slurries with a complex size and density distribution.

Scaled Testing Approach

The scaled testing approach employs both geometric and kinematic similarity principles. Geometric similarity requires all length scales to be reduced by the scale factor (SF). Kinematic similarity is partially satisfied by holding the pulse volume fraction (ϕ_P) and duty cycle (DC) constant. The use of dynamic similarity depends on the type of problem being addressed. For non-Newtonian rheology, the primary dynamic similarity parameter is the yield Reynolds number, $Re_\tau = \rho u^2/\tau_y$. Hence the same velocity is used for testing at different scales. For testing non-cohesive slurries where gravitational effects are important, the Froude number $F = u^2/(s-1)gD$ is the important dynamic parameter for the bulk flow. However dynamic similarity applied to the particle phase requires an additional relation. One such relation is the velocity ratio u/u_s. It is difficult, in general, to hold both the Froude number and velocity ratio independent of test scale. Hence testing is performed with velocity as a free parameter, resulting in a scale relationship.

Pulse Jet Mixing of Non-Newtonian Slurries

For slurries with non-Newtonian rheology, the primary mixing requirement is to overcome the yield stress and mobilize the slurry. A mixing cavern can form when there is insufficient jet momentum to overcome the yield stress everywhere in the vessel. This is illustrated in Figure 1. For downward firing PJMs, the mixing cavern height is approximately given by $Hc/D = c(Re_\tau\,\phi_j)^{1/2}$ where the constant is dependent on the geometry. To mix the entire vessel contents requires the cavern height to equal the fill height. For a given yield stress and mixing system geometry, the relationship gives the required jet

velocity. If that velocity is insufficient to produce a high enough cavern then the number of PJMs or nozzle diameter must be increased. For yield stress values in excess of ~10-20 Pa, it can be difficult to achieve good mixing in the upper region of the vessel. This finding led to the testing and development of PJM-sparger hybrid mixing systems. Since PJMs are affective near the bottom of the vessel where the jet velocity is highest, and sparge bubbles naturally rise to the surface, the concept has natural benefits.

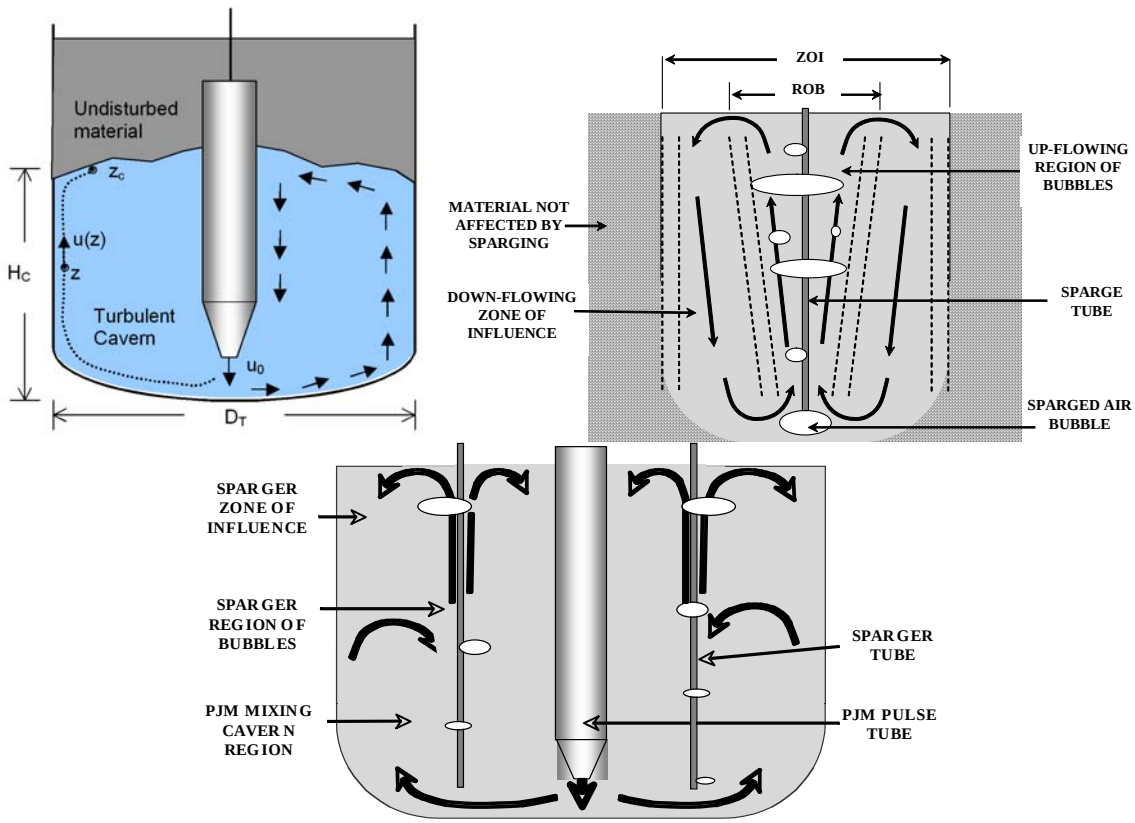

Figure 2.25. Illustration of PJM mixing cavern (top left), air sparge mixing (top right), hybrid PJM/sparger mixing concept (bottom)

Pulse Jet Mixing With Non-Cohesive Solids

Testing with non-cohesive solids at PNNL is ongoing. Results will be used for an overall assessment of PJM mixing system designs in the WTP. Initial tests have been performed at three scales. The testing is parametric in nature, with the solids properties (size and density), operational conditions, number of PJMs, nozzle size, and dish bottom shape all varied over a range of values. The primary measurements are the just suspended velocity (u_{js}) and the solids cloud height (H_{sc}). Additionally, vertical solids concentration profiles have been measured. The data has resulted in correlations for Ucs and Hcs in terms of the geometric, operational, and physical parameters. Additionally, an approximate correlation for the solids concentration at the bottom of the vessel has been obtained. Results indicate that for constant test conditions, U_{cs} scales with $\sim D^{0.28}$, while H_{cs}/D scales with $\sim D^{-1}$. The later result implies great care must be exercised when interpreting test results, as much higher solids loading near the bottom of the vessel will exist at the plant scale than at the observed test scale. The results of the non-cohesive testing at PNNL are provided in PNNL report number WTP-RPT-182 titled "Pulsating Jet Mixing Tests with Non-Cohesive Solids for Evaluating Adequacy of WTP Mixing Systems" which is in final draft to be published in November, 2008.

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NOTE: PDF versions of the above reports and many other relevant reports can be accessed at the following URL: http://www.pnl.gov/rpp-wtp/

2.5.3.2 Pulse Jet Mixers and Key Learnings

Pulse Jet Mixers are an innovative means of mixing without rotating mechanical mixers. They hold promise of long life and little maintenance. They were adopted for most mixing tanks in the new WTP design. They consist of a number of down pumping liquid jets driven by air. On further examination it was found that process operational and design knowledge was all based on qualitative experience and the material tested was poorly characterized and the results not well described. Design methods were based on a set of mechanical rules and were independent of the mixing task and material. In addition the few existing installations were not as large or as tall or had to deal with materials as difficult as those in WTP. A lot of money and effort was spent by WTP on tests by PNNL which first showed that the original design for non-Newtonian slurries was inadequate and had to be boosted in power and with the addition of air sparging. Questions arose on the design of several of the tank handling settling solids which seemed to have inadequate power and or number of mixers and would have trouble suspending the solids and achieving acceptable vertical distributions. Testing by PNNL confirm that these seem to be inadequate and significantly more power and a means for mixing the upper parts of the tank will be required resulting in expensive design revision and increased power and ventilation to achieve the project process goals.

- Pulse jet mixers are an innovative process technology that was not as fully developed as many believed and as often happens with innovative processes needed much more development work and redesign than expected. Such failures of innovation are common in the process industries with slurries.
- Often equipment manufacturers do not have extensive process application knowledge. This knowledge often gets lost over time due to loss of key technical people.
- PJMs are not designed to mix tall tanks by themselves.
- PJMs because they use high velocities must be checked for experimentally for erosion potential.
- Devices handling raw solids such as tank farm waste will be subject to a wide variety of feeds and cannot be designed based on average properties but rather on worst case conditions.
- Early review of key technology assumptions by technical people from the process industries would probably have uncovered the short comings of the PJMS. Such third party reviews are common in the process industries.

2.5.4 Computational Fluid Dynamics

The special session on computational fluid dynamics was moderated by Tom Michener. The first portion of this unplanned, Ad hoc session was dedicated to providing everyone an opportunity to discuss the CFD modeling being performed at their respective companies, in terms of the number of staff working as near full time modelers. Our intent was to get an order of magnitude feel for what is going on in CFD for the attendees at the workshop.

Table 2.8. Computational Fluid Dynamics Special Breakout Session Participants

Name	Affiliation
Art Etchells	AWE3 Enterprises
Randy Neuville	SRNL
Dominik Rhoads	Nexia Solutions Limited
Bob Leishear	SRNL
Steve Strand	Dow Chemical Company
Rick Bokrath	E.I. du Pont de Nemours & Co., Inc
Tom Michener	PNNL

The rest of time was spent discussing the role of CFD in supporting the slurry transport/mixing issues that DOE is facing while dealing with the transport/mixing processes involved as part of nuclear waste vitrification efforts. Private industry attendees working with non-nuclear slurries also provided their input on CFD and multi-phase modeling at their companies.

Estimates of Number Full Time Staff Performing CFD

SRNL has 1-2 applied CFD staff, Nexia Solutions has ~10 people, a mixture of applied and developers, Dow has also ~10 people running commercial codes with enhancements in some cases, Bechtel National Inc has 3-5 applied CFD people, DuPont has 3-4 applied CFD types and 1 developer, PNNL has ~20 staff performing CFD, of which ~6 are developers.

General Discussion

The discussion started with comments on the impact on the credibility of CFD when applied improperly; whether it is:

- Inappropriate grids
- Application of the software outside the bounds of the constitutive equation assumptions
- Presentation of predictions when the code has not been verified or validated for the specific type of application.

The opinion of the majority of the attendees was that multi-phase and non-Newtonian CFD capabilities are still weak at best. Examples of difficult problems that are not generally solved correctly are the slumping problem (50 cent rheometer) and accurately modeling particle distributions in mixers.

The non-Newtonian problems typically arise from a code problem in some of the popular software while the complex physics of the turbulent interaction on drag coefficients in multi-phase system is usually not adequately modeled. Thus multi-phase modeling needs extensive experimental validation.

Another example of CFD limitations is the free surface problem. Correctly predicting the vortex that forms during certain types of mixing conditions is still problematic.

There was a general consensus that CFD would be of the most value if modelers worked closer with the experimentalist, (process Engineer). The more the modeler knows about fluid flow in general the better the models. Validation against good and meaningful experimental data is a necessity.

There will be an enormous amount of data coming from the Hanford M1 (pipe line transport) project. This data will provide an opportunity to evaluate the multiphase modeling capabilities of existing CFD codes and provide a challenge for software currently in development, such as PNNL's lattice-boltzmann based multiphase CFD code. The initial data from M1 will provide data on critical flow velocities and the differing flow regimes for various particle concentration loadings. Eventually, more complex simulants and non-horizontal pipeline geometries will also be investigated.

In addition to the M1 project, BNI/PNNL has an effort, (M3) to look at mixing of multiphase mixtures in the different tank designs using Pulse Jet Mixers (PJMs). The M3 project is tasked with:

- Identifying tanks as having Newtonian or non-Newtonian wastes
- Identifying the suspected problem tanks (in regards to mixing)
- Defining the limiting rheologies
- Providing PJM operational guidance

While it is not the intention of the "M" series to provide data for the modelers, it is an opportunity for us as modelers to get access to data to perform model calibration and validation of existing and new approaches. Perhaps this data could become part of a standard suite of non-Newtonian data to validate CFD codes.

Conclusions

So what do we tell DOE?

1. There is a role for CFD
2. CFD needs to be done right (validated and correctly applied)
3. The understanding of multiphase flow turbulence & how to model it is limited. More needs to be done here.
4. CFD is maturing and is getting better
5. Bad CFD can be dangerously misleading.
6. Since "black" cells must work. CFD may be used to perform trend analyses to better understand the operational issues.

Pacific Northwest
NATIONAL LABORATORY

U.S. DEPARTMENT OF
ENERGY

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352
1-888-375-PNNL (7665)
www.pnl.gov

U.S. DEPARTMENT OF ENERGY

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Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop

Volume IV

GL Smith
AP Poloski
MW Rinker
RL Demmer
AW Etchells, III
BE Lewis Jr.
SL Marra

July 2009

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Pacific Northwest National Laboratory
Richland, Washington 99352

(1) Idaho National Laboratory, Idaho Falls, Idaho
(2) AWE3 Enterprises, Philadelphia, Pennsylvania
(3) Oak Ridge National Laboratory, Oak Ridge, Tennessee
(4) Savannah River National Laboratory, Aiken, South Carolina

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Appendix B

Sampling of Radioactive Wastes

Appendix B

B.1 Introduction

Obtaining samples of high-level waste created during the reprocessing of spent nuclear fuels presents unique challenges. Generally, high-level waste is stored in tanks with limited access to decrease the potential for radiation exposure to personnel. Samples must be obtained remotely because of the high radiation dose from the bulk material and the samples; samples require shielding for handling, transport, and storage. The quantity of sample that can be obtained and transported is limited due to the hazardous nature of the samples as well as their high radiation dose.

Many high-level wastes have been treated to remove strontium (Sr) and/or cesium (Cs) or have been neutralized to decrease corrosion of the tanks. These processes often result in precipitation, and produce multiphase wastes that are heterogeneous. Evaporation of waste with significant concentrations of dissolved salts has occurred in some tanks due to the high heat load associated with the high-level waste, resulting in the formation of a saltcake and/or crusts. Organic layers exist in some waste tanks, creating additional heterogeneity in the wastes.

Due to these extraordinary challenges, substantial effort in research and development has been expended to develop techniques that provide representative samples of the contents of the high-level waste tanks. A summary of the primary techniques used to obtain samples from high-level waste tanks at Department of Energy (DOE) sites is provided in Table 1. These techniques will be summarized in this Appendix. Guidance on selecting appropriate sampling devices for waste covered by the Resource Conservation and Recovery Act (RCRA) is also provided by the United States Environmental Protection Agency (EPA) (EPA 1999). Vapor sampling of the head-space is not included in this Appendix because it does not significantly affect slurry retrieval, pipeline transport, plugging, or mixing.

Table B.1. High-Level Waste Tank Sampling Methods

Technique	Material Type Solids	Liquids	Gas	Notes
Bottle on a String		X		Dip sample
Sample Cup	X			Manual system used at Savannah River Site to obtain salt-cake samples.
Auger	X			
Thief and Trier	X	X		Similar to core drilling. To obtain hard samples, the drill is rotated. For liquid and soft samples, the sample is taken in push mode.
Soft Sludge Sampler	X	X		Thin-walled pipe with a bottom closure.
SUMMA Canister			X	Stainless steel canister that has been evacuated to provide a vacuum that pulls the sample into the canister.
Sorbent Trap			X	OSHA sampler for obtaining aerosol samples.
Hydraulic Scoop	X			
Cylinder with retractable nose cone	X			
Cryogenic			X	Head-space vapors are drawn under vacuum through Teflon tubes to a cryogenic sampling apparatus.
Sample Thief (Bacon Bomb)		X		
Robotic Arm	X	X	X	Material type captured is dependent upon the end-effector.

B.2 Liquid-Sampling Techniques

The simplest of the liquid sampling techniques is dip sampling. At the Hanford Site, this sampling technique is often referred to as "bottle on a string." Only liquid or slurry samples can be taken by this method. Samples can be taken at various depths in the tank to determine whether there is vertical heterogeneity in the tank. If data on the stratification in the tank is not needed, waste in the tank should be sparged or mixed before taking the sample to increase the chance of obtaining a representative sample.

A dip sample is taken by lowering a stoppered and weighted bottle into the waste to the desired depth. After the bottle has reached the desired level, the stopper is pulled from the bottle and the liquid or slurry sample flows into the bottle. The bottle is then pulled from the tank. (WHC)

Liquid samples from radioactive-waste tanks have also been obtained using a vacuum-pump system. Samples were pulled by vacuum from the specified level in the tank through Teflon® tubing into a sample jar; if necessary, the sample jar could be shielded. A stainless-steel pipe nozzle is attached to the bottom of the Teflon® tubing to keep it vertical. A diagram of the vacuum-pump sampling system used at Oak Ridge National Laboratory (ORNL) is provided in Figure 1. (Sears 1990)

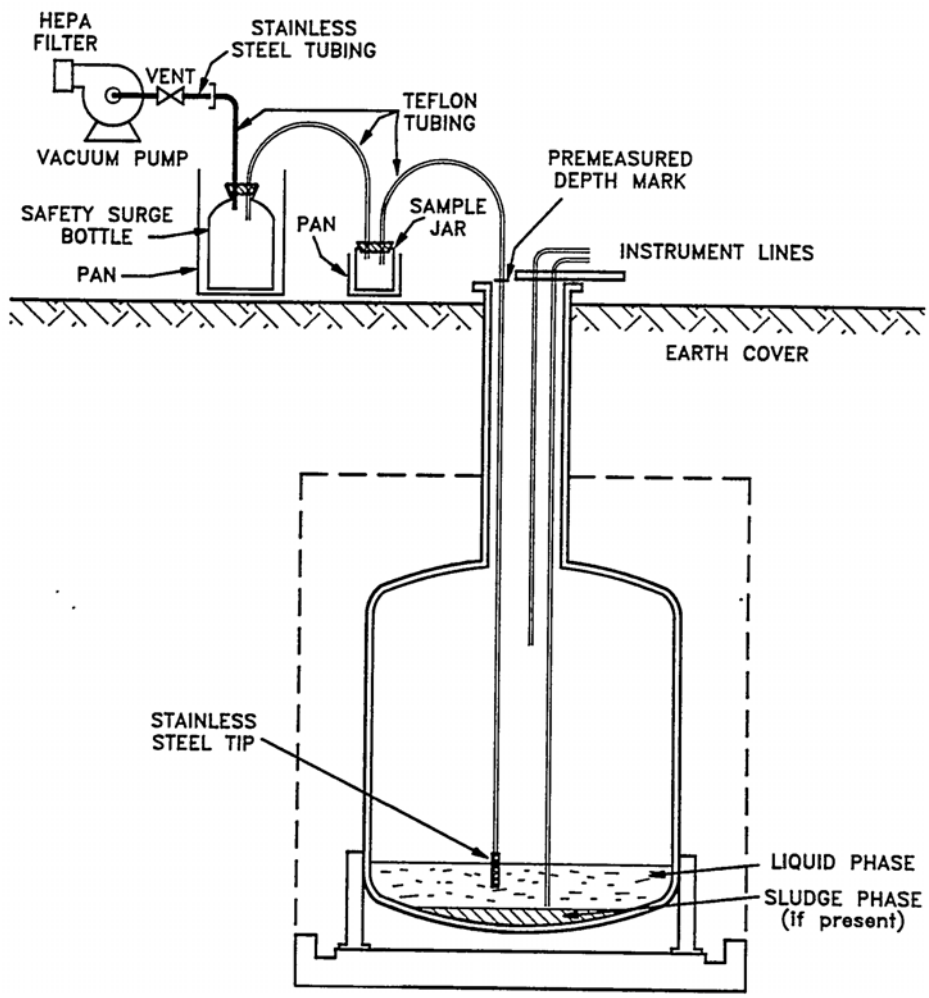

Figure B.1. Vacuum Pump Sampling System

Some high-level waste tanks, such as the Highly-Active Storage Tanks (HAST) tanks at Sellafield in the UK, had sampling systems installed in the tanks before the high-level waste was added. The HAST system uses a needle orifice as part of a reverse-flow diverter (RFD) to obtain samples. The needle orifices are easily plugged by particles; only liquid samples can be obtained by this system. The tanks also can agitate the contents to help obtain representative samples of the liquid phase. (Onishi 2005)

Liquid samples may also be captured by methods used primarily to obtain solid samples. These methods are described in more detail in the section for solids sampling.

B.3 Solids Sampling Techniques

Early sampling of the solids content of Hanford tank wastes was by the use of an auger. Auger samples were taken only from the surface of the waste and were limited to 6 inches. Some homogenization of the sample occurs while obtaining auger samples. These samples can only be taken directly beneath a penetration, or riser. (Beck 1992)

Auger samples are taken by encasing an auger in a shroud to contain the sample. The auger is rotated through the sample while the shroud remains stationary. Sample is collected along the flutes of the auger. Liquid is generally not contained in the auger unless it is associated with solids in the form of a sludge or highly viscous slurry.

Core drilling is the primary mechanism for obtaining samples from the Hanford waste tanks. A core-drilling truck with a shielded handler was specifically designed for this purpose. Two modes, push or rotary, can be used to obtain samples. Liquids, slurries, and soft sludges can be obtained in push mode; rotary-mode sampling must be used to obtain samples of harder sludges and salt-cake. Only minimal success has been achieved when sampling saltcake.

The Hanford sampler is based on a modified core-drilling design that is similar to the thief-and-trier-type samplers. Details of the core-drilling truck procedure are provided in *Waste Characterization Plan for Hanford Site Single-Shell Tanks* (Winters 1990 and Hill 1991). Liquid and solid samples are trapped in the sampler by a spring-actuated rotary valve (see Figure B.2). Two different sampler designs have been used, but both designs incorporated the spring-actuated rotary valve. The first design produced samples that were 19 inches long and 1 inch in diameter. The later design had a slightly larger diameter (1.25 inches). Core samples can be taken at varying depths to obtain samples that comprise the entire depth of the waste. A hydrostatic fluid is added via the drill string to keep the waste from slumping into the void created by the sample when the sampler is pulled from the tank. Normal paraffin hydrocarbons (NPH) were initially used as the hydrostatic fluid.

Figure B.2. Hanford Core Sampler

A sampler based on the same principle was used at ORNL to obtain samples of soft sludges from waste tanks at that site. Samples are collected by manually pushing a polyvinyl chloride (PVC) pipe with a detachable handle assembly into the sludge in the tank. A bottom closure that can be controlled from above by the operator is incorporated into the sampler (Figure B.3). This sampler is capable of capturing both liquid and soft sludge samples. A brief description of the operation of this sampler is provided in an ORNL technical document describing the sampling and analysis of radioactive waste tanks (Sears 1990).

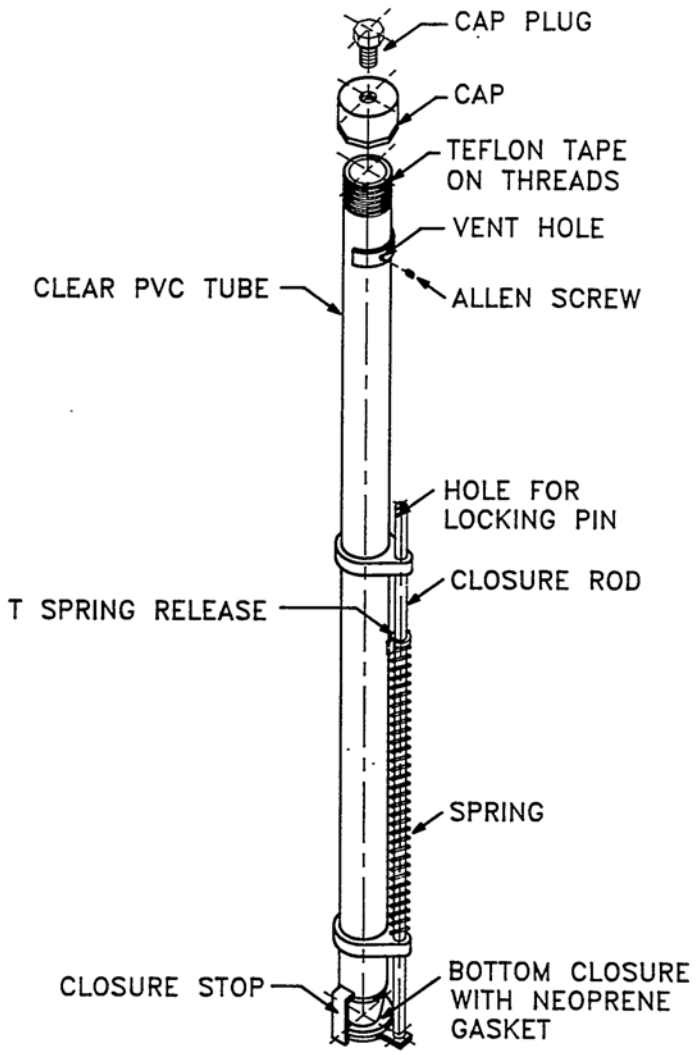

Figure B.3. ORNL Soft Sludge Sampler

The Savannah River Site developed a similar method for obtaining soft sludges. The sampler is a cylinder with a retractable nose cone at the bottom. Sections of pipe are added to the sampler to lower it to the desired depth in the tank. Penetration into the sludge is achieved by using the collective weight of the sampler and pipe sections. Once the desired depth is achieved, the nose cone is retracted into the cylinder, forming an annulus between the cone and cylinder. Gases and liquids pass through a vent at the top of the cylinder, allowing the sludge to be trapped in the cylinder. After the cylinder is closed, the sampler is raised out of the tank into a shielded cask. (Goheen 1995)

Savannah River Site staff have also developed a manual method of capturing salt-cake samples from waste tanks. This method incorporates a sample cup pinned to a handle that can be driven into the salt cake. The cup has a sharp edge to allow it to cut through the salt cake as the handle is pounded with a hammer. The bore of the cup has a ledge like a fishhook barb that captures the material once it is forced into the cup. The cup design is shown in Figure B.4 (Gray 1994).

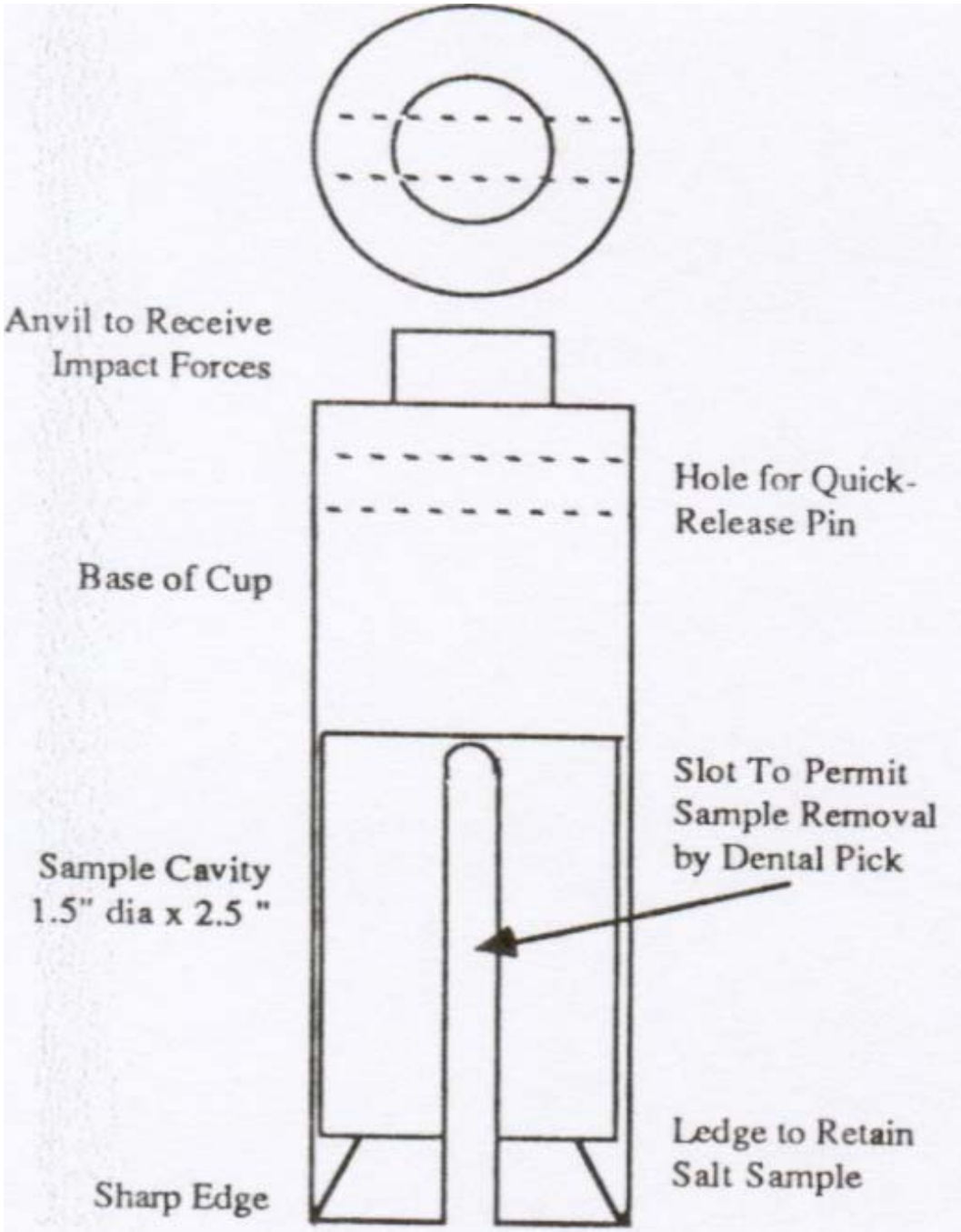

Figure B.4. Savannah River Site Salt Cake Sample Cup

Robotic arms have also been deployed in waste tanks to retrieve samples. Light-Duty Utility Arms (LDUAs) are mobile, multi-axis positioning systems that can access tank contents through the existing risers. These arms provide a flexible robotic deployment platform for many applications including sampling. Using the Extended Reach End-Effector (EREE), waste samples have been retrieved from Hanford tanks for laboratory analysis. The extended-reach arms allow samples to be taken throughout the tank, not just directly under risers. Samplers are detachable from the arm and can be designed to obtain samples of different volumes. Current samplers have a clamping force of 50 to 300 pounds and can capture both liquids and solids (Noonan 1998).

Several other systems for obtaining liquid and solid samples from radioactive waste tanks have been proposed but have not been tested extensively. These methods include hydraulic mining, hydraulic scoop, and bacon bomb samplers.

Hydraulic mining can be performed in several different ways to obtain different fractions of waste components. Slurries can be obtained by inserting a tube into the waste tank, generally through a riser. Water or other appropriate fluid is pumped down the tube at a flow rate and velocity high enough to suspend the non-soluble components of the waste. A portion of the solution is retrieved by pulling it up another passage in the sampling housing. Samples of the soluble components of the waste can be obtained by a similar procedure with lower flow rates and velocities such that the non-soluble fractions are not suspended. Non-soluble components can be sampled by placing a filter at the bottom of the sampling housing and allowing the water to pass through the filter and remain in the waste tank. The filter is pulled up to the top of the tank and taken to a hot cell to open the container and perform the desired analyses (Gray 1994).

Hydraulic scoops can be used to obtain liquids, sludges, and slurries. The scoop is opened and lowered into the tank to the desired level. Once the scoop has sunk to the desired level, the scoop is closed, capturing the sample. The scoop is then raised out of the tank.

Bacon bombs are commercial thief samplers used to obtain liquid samples from the bottom or at intermediate depths in storage tanks, tank cars, and drums. When the thief strikes the bottom of the tank, a plunger assembly opens to admit the sample. The plunger closes again when the bomb is withdrawn, forming a tight seal. Samples can be taken at any depth with the use of a secondary trip line that opens the plunger assembly.

B.4 Sampling-Plan Design

As discussed in the introduction of this appendix, sampling of high-level wastes from spent nuclear fuel reprocessing presents many extraordinary challenges, but some basic principles of sampling design will help obtain accurate results from the samples taken. These principles are not unique to sampling of high-level waste, and application of all these principles may not be possible in many sampling activities.

An essential part of planning a sampling program is to identify the goals of the program and the confidence level required for the desired results. Based on these two parameters, decisions can be made as to the number of samples retrieved, location of sampling, measurements to be performed, and the analyses to be made. Economic constraints may also drive some of these decisions.

Variability of a particular analyte throughout high-level waste tanks is often a significant consideration. Variations due to heterogeneity of the waste often exceed the deviation of the analytical methods. This variability may be due to the nature of the solids, stratification caused by treatment in the tank, changes in the waste streams as the tank was filled, and selective settling in the tank. Because of limited access to these high-level waste tanks, it is often difficult to obtain samples that provide an accurate picture of the range of variability of a particular analyte in the tank; therefore, the adequacy of the sampling population in representing the total population may be difficult to assess. Bias resulting from the method of sampling may also skew the results from the true population. Such biases may result from selectively capturing a particular phase, size, or density of the waste material. Even if the values for particular analytes are precisely determined from the samples obtained, the statistically true value may deviate from the scientifically true value because of failure in the sampling program.

Locations for sampling may be selected randomly, or deliberately chosen to represent the range of conditions observed in the field or unusual conditions thought to be of particular interest. Randomly selected locations are appropriate for overall assessments of site conditions, in wastes where variations are random (e.g., in liquid wastes), in wastes where enough samples are available to sufficiently characterize the range of values, or where outlier samples are considered either unlikely or unimportant. When these criteria are not met, deliberately chosen sample locations will better define the characteristics of the waste.

Thorough interpretation of the data obtained from these samples requires identification of the source of variation in the results, an assessment of the adequacy of the characterization of this variation, and an evaluation of the significance of the range of values obtained. Increased confidence in these analyses can be achieved by increasing the number of samples, using a multifaceted approach to examine the results, or using a backup system such as real time monitoring to verify the results. Other approaches that might be used along with laboratory results from samples include field observations, historical data, theory, laboratory and mathematical models, statistical analyses, and past experience. (Jensen 1988)

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Appendix C

Guidelines for Performing Measurements of Chemical, Physical, and Rheological Properties

Appendix C

Guidelines for Performing Measurements of Chemical, Physical, and Rheological Properties

C.1 Introduction

Review of past DOE projects shows that standardized chemical, physical, and rheological measurement-guidelines would be valuable for use across the DOE complex. The attached property-measurement guidelines were developed based on work at the Waste Treatment and Immobilization Project (WTP). They are a valuable starting point for a consensus property-measurement guidelines document. Use of these property-measurement guidelines is essential to assure standardized, comparable results from all actual-waste and simulant-based tests conducted across the DOE complex.

This document brings together chemical, physical, and rheological properties-measurement guidelines for the characterization of both actual and simulated wastes. This document is organized into four main sections:

- Introductory Text: These sections contain a table (see Table C.1) specifying the data that is to be obtained to properly characterize both actual and simulated wastes. All data are to be reported in hard copy with an accompanying electronic Excel® file (see Table C.16).
- Chemical-Properties Measurements: This section contains guidelines for obtaining the necessary chemical-composition data. The specific analytical procedures, minimum reportable quantities (MRQ), etc. will be agreed upon between the DOE project and the performer of the work via project-approved documentation, e.g., a test-specification and test-plan process. Only instructions for determining pH are given in this section.
- Physical-Properties Measurements: This section integrates the steps required to determine each physical property.
- Rheological Properties Measurements: This section provides a standardized approach to the determination of important rheological properties for slurries and melter feeds.

Acronyms

HLW – high-level waste

LAW – low-activity waste

M&TE – measurement and test equipment

MRQ – minimum reportable quantity

NIST – National Institute of Standards and Technology

PSD – particle-size distribution

RPM – revolutions per minute

SEM – scanning electron microscopy

TRU – transuranic

WTP – Hanford Tank Waste Treatment and Immobilization Plant

C.2 Applicability/Scope

The property-measurement protocols detailed in these guidelines are applicable for research and technology (R&T) testing within DOE projects. This is intended to be a 'living' document and will be updated as other DOE tasks determine what additional testing methods need to be added. After the additional specific-measurement procedures are developed, reviewed, and approved for use, this guideline document will be revised. Users are responsible for confirming that they are employing the latest version of this document.

C.2.1 Prerequisites

C.2.1.1 Characterization Data

This section defines suggested necessary-and-sufficient chemical, physical, and rheological properties to be measured for the characterization of both actual and simulated wastes to support DOE projects. Table C.1 provides an example of a matrix of the necessary-and-sufficient property measurements to be performed on tank waste (as received from Hanford tank farms), pretreated wastes, and melter feeds from the WTP Project. This test matrix applies to both actual and simulant wastes. Each DOE project should evaluate its own property-measurement needs and generate a similar table specific to its project. The goal of a table similar to Table 1 is to assure that each performer of the work/subcontractor/etc. supporting the project performs standardized analyses and reporting for each material so that data is comparable across all project functions.

A table similar to Table C.1, reviewed and approved by the project, will be used to determine which subset of property measurements will be conducted and reported as a default for each waste stream. Specific instructions within project-approved direction documents, e.g., test specifications, may be used to deviate from the specified property measurements delineated in the final project-approved Chemical, Physical and Rheological Properties Measurement Matrix.

Table C.1. Chemical, Physical, and Rheological Properties-Measurement Matrix for Tank Waste, Pretreated Wastes, and Melter Feeds (actual and simulant)

Property	Tank Waste	Low-Activity Waste (LAW) Pretreated Waste	High-Level Waste (HLW) Pretreated Waste	LAW Melter Feed	HLW Melter Feed
Chemical Composition	X	X	X	X	X
pH[a]	X	X	X	X	X
PSD	X		X	X	X
Particle configuration (size & shape)	X				
Heat Capacity	X				
Bulk Density	X		X	X	X
Supernatant-Liquid Density	X[b]	X	X	X	X
Vol % Settled Solids	X[b]	X[b]	X	X	X
Settling Rate			X	X	X
Centrifuged-Solids Density	X[b]	X[b]	X	X	X
Vol % Centrifuged Solids	X[b]	X[b]	X	X	X
Wt % Centrifuged Solids	X[b]	X[b]	X	X	X
Wt % Oven Dried Solids	X				
Wt % Total Dried Solids	X	X	X	X	X
Wt % Undissolved Solids	X[b]	X[b]	X	X	X
Shear-Stress-Versus–Shear-Rate, ambient and 40°C	X	X	X	X	X
Yield Strength	X[c]		X	X	X
Chemical Composition	X	X	X	X	X
Wt % total oxide	X	X	X	X	X

(a) Only aqueous materials with pH below 14 will be quantified and reported
(b) Only if solids present
(c) Only of tank-waste sludge

The following notes apply to analyses described in Table C.1:

1. A procedure for measuring heat capacity has not been included in this document at this time. A separate heat-capacity-measurement procedure will be added to the physical-properties measurement section once a measurement procedure has been developed.

2. Supernatant-liquid density provides fundamental physical information on the incoming waste; this information allows for the conversion between mass and volume of analyte concentrations and other process data, and is also valuable in modeling and understanding viscosity and settling data. Supernatant-liquid density is a good up-front test for simulant development. An incorrect simulant supernatant-liquid density is an early sign of simulant problems. Additionally, supernatant-liquid density is extremely easy to measure and therefore is included in the list of properties to be measured.

3. Settling-rate data helps assess the performance of mixing, pumping, and sampling functions that are critical to DOE projects and most waste-form qualification strategies. While not providing information on the heaviest material, the settling-rate measurement method does provide settling information valuable for process design and simulant validation. Since vol % settled solids data is still needed, the samples will be left for three days to settle under the force of gravity. Calculation of the settling rate simply requires occasional measurement of the settling-layer height. Collection of the settled-solids volume over this settling period can also be used to assess the accuracy of the settling test.
4. Centrifuged-solids information (centrifuged solids density, vol % centrifuged solids, and wt % centrifuged solids) aids in assessing the accuracy of actual and simulant properties. Centrifuged-solids information is of limited value because it is dependent on centrifugation rate and sample geometry. However, to determine the wt % undissolved solids, all samples are centrifuged at 1000 gravities for one hour. The liquids are then decanted to clean vials. The centrifuged solids and decanted liquids are then dried at 105°C. Since centrifugation is performed on all samples, calculation of the centrifuged-solids information is just a matter of recording the volume of solids following centrifugation (mass is already recorded). This information provides data on the packing efficiency of the centrifuged material that is useful in assessing the accuracy of the wt % undissolved-solids analysis. The resulting information can also be used to compare packing efficiencies between actual waste and simulant.
5. Wt % oxide data is needed for the calculation of effective glass yield.

C.2.2 Global Electronic Reporting

Electronic reporting in an Excel® format using specified units is required for all data generated for the project by this guideline. The objective of this formatted reporting is to facilitate data exchange between performers of the work/subcontractors/etc. and to enable other engineering groups to easily access the data. A global data table is provided in Table C.16 "Global Data Summary Electronic File" at the end of this document, where all data generated is brought together in one file in a standardized manner. By having all data assembled in this way, it is easy to compare data or determine whether data is missing.

C.2.3 Precision and Bias

Data are not yet assembled to evaluate either the intra-laboratory or inter-laboratory precision and bias of the property-measurement methods described in this guideline.

C.2.4 Identification of Samples

Sample identification should be done in a consistent logical fashion that allows the sample to be readily associated with its source.

C.2.5 General Equipment-Calibration Guidelines

Calibrations – Initially calibrate or indicate calibration status of all instruments used in implementing these guidelines. Verify calibration status during instrument use to indicate possible instrumental drift.

Calibration Schedule:

- *Temperature-Measurement Devices* shall be calibrated with standards traceable to National Institute of Standards and Technology (NIST).
- *Balances* are to be calibrated on an annual basis to 0.1% of their rated capacity and standardized before and after completion of all weighing using NIST standard masses.
- *pH meters* will be standardized before and after completion of groups of 10 samples or less using commercial buffer solutions that bracket the measured pH values. The pH meter shall be standardized at the same temperature as the system being measured. Expired buffer solutions shall not be used for standardization.
- *Water-Purification System*s shall be calibrated at least annually following the manufacturer's instructions.

C.3 Chemical-Properties Measurement

C.3.1 Chemical and Radiochemical Composition

The chemical and radiochemical (for actual waste only) composition of radioactive wastes and simulants must be reported to allow for accurate comparisons. Chemical-composition reporting will be consistent with Table C.16 "Global Data Summary Electronic File." If an analyte is not measured, it will be so noted; however, the analyte designation and row will not be deleted, to allow future ease of direct electronic comparison of the composition of the current sample with that of a different sample.

Specific analytes to be measured and the procedures for quantification of said analytes for individual scopes of work will be agreed upon between the specific DOE project task and the performers of the work/subcontractors/etc. via project approved documentation, e.g., test specifications and test plans. The Minimum Reportable Quantity, Minimum Detection Limit/Activity, Quality Control acceptance criteria, Laboratory Control Standards, and Matrix Spikes for given analytes of interest will be provided in project-approved documentation, e.g., test specifications. The detection limit for each analyte should be reported with the analytical results. Matrix-spike recoveries and laboratory control standards used for the agreed-upon analytes and analyses are to be reported. Additional analytes obtained on an opportunistic basis are also to be reported.

Note: Radioisotopes will not be measured in a non-radioactive simulant. Mercury (Hg) is measured in the initial waste received from tank farms. Mercury (Hg) should only be measured again, (e.g., after specific unit-operations testing), if found in sufficient quantity in the initial analysis to warrant further analysis.

C.3.2 Simulant Preparation and Chemical-Composition Analysis

Simulants should be produced in a manner consistent with the requirements in Appendix D of this report.

Determination and reporting of the simulant's chemical composition will rely on both the mass balance and sample analyses together as a cross check. A batching process description will be written that specifies the following:

1. The technical purity or grade of the initial chemical constituents; will require copies of each chemical's purity certifications and may require a confirmation of adsorbed water or waters of hydration;
2. Identify to the vendor/subcontractor the batching sequence and how/when to combine various sub-batches as necessary (Note: for typical contaminants such as chloride, these should be added after the amount already present from the other chemicals added is known);
3. In-process sampling and analyses at key simulant-preparation points, e.g., whether to analyze a nitrate solution before neutralizing and precipitating solids, or after a precipitation and washing sequence to verify that target values have been reached;
4. Review of completed batching sheet(s) by an independent individual;
5. Vendor/subcontractor to analyze and supply to the cognizant DOE project task the results of the analyses to verify the final batch composition for acceptance.

Following the simulant preparation, a confirmatory quantitative analysis is performed on the simulant to verify that all components were added and in the correct amount. This analysis is a final independent validation of the simulant composition. If the analysis indicates that an analyte component amount differs from the target amount by significantly more than the analytical uncertainty for that component, there is reason for concern that an error has occurred with the simulant preparation. Using both the mass balance (i.e., batching sheets, chemical addition and weighing confirmation, and calculation verification) and actual chemical-composition analysis together will greatly increase the probability of producing a simulant with an accurate chemical composition. Having both the mass balance and chemical-composition analysis will allow informed decisions on whether to rely on the calculated or measured analyte value or decide to re-analyze. For example, an adjustment would not have to be made to a simulant batch composition based on one analyte analytical result if the mass-balance composition and batching sheets corroborated the majority of the analysis. Disagreement between the measured analytical results and the mass balance or batching sheets due to errors in simulant preparation, however, could lead to a re-analysis and possible re-batching of the simulant. Potential errors in simulant preparation may include: 1) incorrect chemical quantities or incorrect chemicals being added; 2) use of chemicals with poor quality or high levels of impurities; 3) use of chemicals with elevated levels of waters-of-hydration from excessive storage; or 4) use of starting chemicals that were not reported.

The prepared simulant composition will be certified to a previously agreed-upon set of analyte values. Typically a graded range of analyte composition values are used for simulant-preparation work; the graded range will be provided to the vendor/subcontractor before simulant-preparation work begins. An example of a graded range of analyte composition values for preparation of a melter-feed simulant may be ± 5 wt% for major constituents (analytes greater than 0.5 wt% on an element basis) and ± 20 wt% for minor constituents (analytes less than 0.5 wt% on an element basis) known to not have an effect on the melter testing parameters to be studied.

Finally the chemical composition of the final simulant will be reported documenting the steps, decisions, etc. performed to confirm that the simulant meets the target composition as described above.

C.3.3 Melter-Feed Formulation and Mixing

This section provides two tables that must be included in any data package that reports test results on simulated and actual melter feed (HLW and LAW). A standardized approach to this reporting process is needed to assure that comparisons can be accurately made. Table C.2 provides an example from the WTP project used to document the quantity of glass-former chemicals added to pretreated wastes, as well as the sodium concentration of the pretreated LAW and oxides loading for HLW pretreated sludge. (Note: if the project uses glass frit, e.g., the Defense Waste Processing Facility at Savannah River, instead of glass-former chemicals, this table will be much shorter and simpler than the example provided.) Table C.3 is used to describe how and in what order glass-former chemicals/minerals were added to the pretreated waste to form the melter feed; the addition process can affect the properties of the resulting melter feed.

Table C.2. Glass-Former Chemicals/Minerals

Pretreated Waste Identification		
Pretreatment History (include: washing, leaching, chemical precipitation, mechanical-agitation time and intensity		
Sodium Concentration of LAW Pretreated Waste		Molar
Oxides Loading of HLW Pretreated Sludge		Total grams oxide/Liter

Source Chemical	Manufacturer	Oxide	Grade	Target wt %	Target Mass (g)	Actual Mass Added (g)
Kyanite	Kyanite Mining Corp	Al_2O_3				
Alumina A-2	Alcoa Alumina	Al_2O_3				
Boric Acid Technical	U.S. Borax	B_2O_3				
10M Borax	U.S. Borax	Na_2O/B_2O_3				
Soda Ash	Solvay Minerals, Inc.	Na_2CO_3				
Wollastonite	NYCO Minerals, Inc.	CaO				
Fe_2O_3 5001	Prince Mfg. Co.	Fe_2O_3				
Li_2CO_3	Chemetall-Foote	Li_2O				
Olivine	Unimin Corp	MgO				
SCS-75	U.S. Silica Company	SiO_2				
Rutile (Air floated)	Chemalloy Co., Inc.	TiO_2				
Kadox	Zinc Corp. of America	ZnO				
Zircon	Amer. Miner, Inc.	ZrO_2				
Sucrose	Amalgamated Sugar Co.	Sugar				

Table C.3. Mixing Operation Data Needed to Compare Mixing of Melter Feeds
(expand data fields as necessary to include requested information)

Activity/Property	Data or Explanation
Order of Chemical Additions	
Mixing Time	
Impeller Speed	
Impeller Diameter	
Tank Diameter	
Number of Baffles	
Size of Baffles	
Depth of Impeller	

Comments:

C.4 pH Measurement

C.4.1 Purpose / Scope

This section of the technical guideline describes a general method for pH measurement of solutions, slurries, sludges, and associated simulants. (Note: Measurement of pH for samples of actual tank waste should be consistent with EPA Method 9040B or similar methods outlined in approved project documentation, e.g., test specifications or test plans.) Other DOE project tasks may use these guidelines provided they involve materials of similar chemical and physical properties.

C.4.2 Applicability

This procedure is applicable to leachates, supernates, and sludge samples in the pH range of 0 to 12.5. For pH above about 12.5, a special electrode made for high-sodium and high-pH conditions is needed. The reliability with proper standardization shall be ±0.1 pH unit. Temperature affects pH measurement; therefore calibration and measurement shall be done at the same temperature.

C.4.3 Quality Control

C.4.3.1 Standardization

Because of the different capabilities of different pH meters in use, only minimum requirements can be specified. Manufacturer's instructions for calibration of the meters using standard buffers shall be followed. A minimum of two buffers shall be used and calibration checked with a third (different) buffer.

C.4.3.2 Control

Control is established by measuring, within ± 0.1 pH units, a buffer that is not used for calibration before beginning analysis, after every tenth sample, and at the end of the analysis run.

C.4.4 Equipment and Supplies

1. pH meter (electrometer)
2. indicating (glass) and reference electrode, or combination pH electrode
3. suitable beakers, 20-50 ml
4. Teflon™-coated stir bars
5. magnetic stirrers
6. reagents - pH buffer solutions at pH of 4,7,and 10. Commercially available certified buffers. Additional buffers are available for use if required.

C.4.5 Work Guidelines

C.4.5.1 Instrument setup

Note: *Glass electrodes are fragile and care must be taken not to scratch or break the electrodes. Electrodes shall be replaced when manufacturer-specified maintenance procedures do not return them to satisfactory operation. Electrodes shall be stored according to manufacturer's instructions when not in use. Results are to be recorded in laboratory record books, test instructions, or associated bench sheets. The data shall be transcribed into an EXCEL® electronic file and an electronic copy will be transferred to the project.*

Note: *To avoid contamination of buffer solutions, do not return used solutions to the container.*

1. Follow manufacturer's operating instructions for setup and calibration of pH meter using buffer solutions as specified. As a minimum, calibrate the pH meter using pH 4 and pH 10 buffers and perform verification check using pH 7 buffer. Only buffer solutions within manufacturer's suggested expiration date shall be used.
2. Immerse electrode(s) in buffer solution not used for calibration and, with stirring, allow reading to equilibrate. It shall read within ± 0.1 pH units of the buffer-solution stated value. If not, recalibrate.

C.4.5.2 Sample Analysis

Note: *Sample shall be at the same temperature as the buffer solutions (normally ambient temperature). Stirring shall not be so vigorous that air is entrained into the sample. The calibration and sample measurement temperature shall be recorded and reported.*

1. Place sufficient sample into a clean beaker such that the electrode tip and reference junction are immersed. Add stirring bar and stir continuously.
2. Allow sufficient time for reading to stabilize. Record this pH reading.
3. Remove electrode from sample and rinse it with water. Repeat measurement on the same sample to attain a duplicate result; then continue with remaining samples.
4. Repeat steps 1-3 on additional samples. Record all results in the laboratory record book, run log, or associated bench sheet. A copy is to be transferred to the project file.

C.5 Physical-Properties Measurements

C.5.1 Purpose/Scope

This guideline will be used to measure the physical properties listed below for solutions, slurries, and sludges. Not all the physical properties listed below may be measured for a given sample. Specific instructions within project-approved test specifications may be used to deviate from the specified property measurements delineated in this section accompanied with the technical rationale for said deviation.

1. Density of bulk slurry (g/mL)
2. density of settled solids (g/mL)
3. density of centrifuged solids (g/mL)
4. density of supernatant liquid (g/mL)
5. settling rate (vol%/min)
6. particle-size distribution
7. vol % centrifuged solids
8. vol % settled solids after 72 hours
9. wt% undissolved solids
10. wt % dissolved solids
11. wt % centrifuged solids
12. wt % total oxides
13. wt % oven-dried solids
14. wt % total dried solids

The test methods in this section may be used by other project tasks for characterization of radioactive and non-radioactive materials. For radioactive materials, implement the appropriate safe operating procedures for the work location and materials in question.

C.5.2 Applicability

The test methods delineated in this section are for the physical property characterization of radioactive and non-radioactive solutions, slurries, and sludge materials.

C.5.2.1 Nomenclature for Physical Properties Measurements

$\mathbf{M_B}$ – Total mass of bulk slurry after centrifugation (g)

$\mathbf{M_{CC}}$ – Mass of centrifuge cone (g)

$\mathbf{M_{CR}}$ – Mass of crucible (g)

$\mathbf{M_{CS}}$ – Mass of centrifuged solids (mL)

$\mathbf{M_{DCS}}$ – Mass of oven-dried centrifuged solids (g)

$\mathbf{M_{DCL}}$ – Mass of oven-dried centrifuged supernatant liquid (g)

$\mathbf{M_{FSC}}$ – Mass of oven-fired solids in crucible (g)

$\mathbf{M_{OSC}}$ – Mass of oven-dried solids in crucible (g)

$\mathbf{M_S}$ – Mass of decanted supernatant liquid after centrifugation (g)

$\mathbf{M_{SL}}$ – Mass of supernatant liquid after gravity settling (g)

$\mathbf{M_{SS}}$ – Mass of settled solids after gravity settling (g)

$\mathbf{M_{VL}}$ – Mass of decanted supernatant liquid after centrifugation being placed in vial (g)

$\mathbf{M_{WCS}}$ – Mass of wet sample in crucible (g)

$\mathbf{P_{MCS}}$ – Percent mass of centrifuged solids (%) in slurry

$\mathbf{P_{MDS}}$ – Percent mass of oven-dried solids sample in crucible (%)

$\mathbf{P_{MOX}}$ – Percent mass of oxides per gram (%) of slurry

$\mathbf{P_{MSS}}$ – Percent mass of settled solids (%)

$\mathbf{P_{MTS}}$ – Percent mass of total solids (%)

$\mathbf{P_{ODS}}$ – Percent mass of oven-dried solids in centrifuged solids (%)

$\mathbf{P_{MUS}}$ – Percent mass of undissolved solids (%) in slurry

$\mathbf{P_{VCS}}$ – Percent volume of centrifuged solids (%) in slurry

$\mathbf{P_{VSS}}$ – Percent volume of settled solids (%) in slurry

$\mathbf{V_B}$ – Total volume of bulk sample after centrifugation (mL)

$\mathbf{V_{CS}}$ – Volume of centrifuged solids (mL)

$\mathbf{V_S}$ – Volume of decanted supernatant liquid after centrifugation (mL)

$\mathbf{V_{SB}}$ – Total volume of bulk sample after gravity settling (mL)

$\mathbf{V_{SL}}$ – Volume of supernatant liquid after gravity settling (mL)

$\mathbf{V_{SS}}$ – Volume of settled solids after gravity settling (g)

ρ_B – Bulk density of slurry (g/mL)

ρ_{CS} – Density of centrifuged solids (g/mL)

ρ_S – Density of supernatant liquid (g/mL)

ρ_{SS} – Density of settled solids after gravity settling (g/mL)

C.5.2.2 Definitions

Density – The mass per unit volume.

Interstitial Solution – The solution lying between the suspended solid particles of a sludge sample.

Sludge – Wet solids having little or no standing liquid (mud-like).

Slurry – A mixture of solids and solution.

Supernatant Liquid – A liquid phase overlying material deposited by settling, precipitation, or centrifugation.

Solids Settling Rate – The rate at which solids in a homogenized sample settle. In this guideline, this is the change in the settled-solids height as a function of time.

Vol % Settled Solids – The percentage of the volume of the slurry sample that the settled solids occupy after settling for 72 hours under 1 gravity. These settled solids will contain interstitial solution.

Vol % Centrifuged Solids – The volume of the solids layer that separates from the bulk slurry after 1 hour of centrifugation at 1000 gravities divided by the total sample volume on a percentage basis. These centrifuged solids will contain interstitial solution.

Wt % Total Oxides – The percentage of the mass of the bulk sample that remains after converting all non-volatile elements to oxides. Some volatile elements, such as cesium, might be lost in this process.

Wt % Centrifuged Solids – The mass of the solids layer that separates from the bulk slurry after 1 hour of centrifugation at 1000 gravities divided by the total bulk slurry sample mass on a percentage basis. These centrifuged solids will contain interstitial solution.

Wt % Dissolved Solids – The mass of dissolved species in the supernatant liquid divided by the total mass of the supernatant liquid on a percentage basis.

Wt % Total Dried Solids – The percentage of the mass of the sample that remains after removing volatiles including free water by drying at 105 ± 5°C for 24 h.

Wt % Oven-Dried Solids – The percent mass of the centrifuged solids remaining after removing volatiles including free water by drying at 105 ± 5°C for 24 h.

Wt % Undissolved Solids – A calculated value reflecting the mass (on a percent basis) of solids remaining if all the supernatant liquid and interstitial solution could be removed from the bulk slurry.

C.5.2.3 Quality Control

Quality control has been integrated into work guidelines. The data is collected in duplicate or triplicate as indicated in the work instruction. Statistical analysis is performed on the final data. Acceptance-tolerance criteria are dictated by DOE project staff for the particular matrix being analyzed.

C.5.3 Work Guidelines – (Physical Properties)

Note: *The following weight measurements (for example, centrifuge cones, 20 ml vials, etc.) shall include the container cap unless specifically stated otherwise. Record all data in Table C.4. Record all masses to the nearest milligram and all temperatures to the nearest 1 °C.*

1. Using the physical properties data sheet, fill in appropriate information about the fluid and record the physical-property data collected. The corresponding work-instruction steps are referenced on this worksheet for ease in recording. In addition, complete the Measurement and Test Equipment (M&TE) information. (See Table C.4 – Physical-Properties Data Sheet)
2. Mix the sample to combine any separated liquid and solids phases.
3. Pre-weigh 3 clean volume-graduated centrifuge cones and record the masses on the data sheet. These cones (and lids) must be rated for at least 105°C and be cylindrical from the 10-ml to the 4-ml graduations (Kimble 45200-10 or equivalent).
4. Measure the distance from the 10-ml graduation to the 4-ml graduation on each of the cones. Record this data below as well as the volume mark just above where the cone departs from a cylindrical shape.
5. Transfer subsamples of approximately 5 to 10 ml into each of the 3 centrifuge cones and reweigh. Record the mass on the physical-properties data sheet.
6. Mobilize the settled solids by either shaking the cones, using a vortex mixer, or other comparable means. Record the time and date this process was initiated.
7. Record settling information (volume of the total sample and the volume of settled solids) at the specified intervals in the attached data table. If multiple solids layers are visible, include this data at the same intervals on a separate sheet of paper and attach to the data sheet.
8. Centrifuge the cones at ~1000 gravities for one hour. Record the volume of the total sample and volume of centrifuged solids on the physical-properties data sheet.
9. Decant as much as possible of the centrifuged supernatant liquid to a pre-weighed graduated cylinder and record the mass and volume of the supernatant liquid on the physical properties data sheet.

10. Weigh the solids left in the centrifuge cone and record the mass on the physical properties data sheet.
11. Transfer the decanted supernatant liquid to pre-weighed 20-ml vials that have lids rated to 105°C. Weigh the loaded vials. Record on the physical-properties data sheet.
12. Air dry the solids and liquids overnight to minimize possible splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.
13. Remove lids and transfer the solids and liquids to an oven at 105°C for at least 24 hours.
14. Remove the dried solids and liquids from the oven and cap the vials and cones. Allow them to cool for ~10 minutes and weigh. Record on the physical-properties data sheet.
15. Remove lids and transfer the solids and liquids to an oven at 105°C for at least 24 hours.
16. Repeat steps 14 and 15 until a stable mass is reached (e.g., change in mass is less than 0.1 % in 24 hours). Record the masses on the physical-properties data sheet. Use the last row to calculate the final mass of dry material. Perform the calculations that are noted in the calculation section of this test instruction.

Table C.4. Physical-Properties Data Sheet Format

Balance 1:	Calibration ID _____		
	Calibration Expiration _____	Location	_____
Balance 2: (if needed)	Calibration ID _____		
	Calibration Expiration _____	Location	_____
Thermocouple:	Calibration ID _____		
	Calibration Expiration _____	Location	_____
Digital Thermometer:	Calibration ID _____		
	Calibration Expiration _____	Location	_____

Institution	Analyst	Sample	Preparation Date

Table C.4. (contd)

Physical-Properties Data-Collection Sheet Sample ID: _____

Step #	Description	Units
3	Centrifuge cone tare	g
4a	Distance from 10-ml to 4-ml mark	cm
4b	Volume mark at taper pt.	cm
5a	V_B = Total wt after transfer	g
	M_B = Step 5a – Step 3	g
5b	Total initial volume	ml
7a	V_{SB} = 72-hr total vol.	ml
7b	V_{SS} = 72-hr total settled solids	ml
8a	V_B = total volume after centrifuging	ml
8b	V_{CS} =solids volume mark after centrifugation	ml
9a	Tare wt of graduated cylinder	g
9b	Liquid-plus-gGraduate wt.	g
	M_S =Step 9a – Step 9b	g
9c	V_S = liquid volume in graduate	ml
10	Total wet solids wt. + cone wt.	g
	M_{CS} =Step 10-Step 3	g
11a	Tare wt of 20-ml vial	g
11b	Liquid-plus-20-ml-vial wt.	g
	M_{VL} =Step 11b – Step 11a	g
16a	Final dry wt. of cone	g
	M_{DCS} =Step 16a-Step 3	g
16b	Final dry Wt. of 20-mL vial	g
	M_{DCL} =Step 16b-Step 11a	g

Table C.4. (contd)

Step 17) Record the height of the total volume and solids volume layers on the following table. Measurements beyond 6 hours may be varied to accommodate work shifts, but at least 2 measurements are to be taken each day approximately 7 hours apart.

Settling Schedule			Cone A		Cone B		Cone C	
Interval	Date	Time	Total	Solids	Total	Solids	Total	Solids
5 minutes								
10 minutes								
15 minutes								
20 minutes								
30 minutes								
40 minutes								
50 minutes								
1 hour								
2 hours								
3 hours								
4 hours								
5 hours								
6 hours								
24 hours								
32 hours								
48 hours								
56 hours								
72 hours final settled (V_{SS})								

Table C.4. (contd)

Time	Date	Cone A	Vial A	Cone B	Vial B	Cone C	Vial C
Tare, g							
Final sample mass, g		M_{DCS}:	M_{DCL}:	M_{DCS}:	M_{DCL}:	M_{DCS}:	M_{DCL}:
Comments:							

C.5.4 Calculations (Physical Properties)

Note: *Calculations described below should be recorded in Table C.5.*

1. Bulk Density of Slurry

   The total mass (M_B) and volume (V_B) of bulk slurry following centrifugation were recorded in Steps 5 and 8 of Section C.4.4. These values are to be used for the density calculation. Volume data from earlier in the guideline could be biased low due to entrained gas as well as an inability to clearly measure the total sample volume due to material smeared on the sides of the centrifuge tubes. The density of the bulk slurry is calculated as $\rho_B = M_B/V_B$.

2. Vol % Settled Solids

   The volume of the settled solids (V_{SS}) was recorded after 72 hours under Step 7 of Section C.4.4. The total volume after centrifuging (V_B) was recorded in Step 8 of Section C.4.4. Using this data, the vol % settled solids is then calculated as $P_{VSS} = V_{SS}/V_B \times 100\%$.

3. Centrifuged-Solids Density

The mass (M_{CS}) was recorded in Step 10 of Section C.4.4 and the volume (V_{CS}) of the centrifuged solids was recorded in Step 8 of Section C.4.4. The density is calculated as $\rho_{CS} = M_{CS}/V_{CS}$.

4. Vol % Centrifuged Solids

The volume of centrifuged solids (V_{CS}) and total slurry volume (V_B) were recorded under Step 8 of Section C.4.4. The vol % centrifuged solids is calculated as $P_{VCS} = V_{CS}/V_B \times 100\%$.

5. Wt % Centrifuged Solids

The mass of centrifuged solids (M_{CS}) was measured in Step 10 and the total mass (M_B) was measured in Step 8 of Section C.4.4. Using this data, the wt % centrifuged solids is calculated as $P_{MCS} = M_{CS}/M_B \times 100\%$.

6. Supernatant-Liquid Density

The mass (M_S) of and volume (V_S) of a decanted supernatant liquid subsample was recorded in Step 9 of Section C.4.4. Using this data, the supernatant liquid density is calculated as $\rho_S = M_S/V_S$.

7. Density of Settled Solids

The mass of the settled solids can include a significant fraction of interstitial solution, but does not include the standing supernatant liquid that separates during settling. The density of the settled solids is determined by first calculating the mass of the settled supernatant liquid (M_{SL}) in the centrifuge cone using the supernatant-liquid density (ρ_S) calculated in the previous step and multiplying it by the volume of the separated liquid layer (V_{SL}) after 72 hours from step 17 of Section C.5.3 ($M_{SL} = \rho_S \times V_{SL}$). Then subtract M_{SL} from the mass of the bulk slurry (M_B) from Step 8 of Section C.3.8 to get the mass of the settled solids ($M_{SS} = M_B - M_{SL}$). The density of the settled solids is now calculated ($\rho_{SS} = M_{SS}/V_{SS}$).

8. Wt % Settled Solids

Using the mass of settled solids (M_{SS}) calculated in the previous step, the wt % settled solids can be calculated as $P_{MSS} = M_{SS}/M_B \times 100$ %.

9. Wt % Total Dried Solids

The centrifuged solids and supernatants were dried separately at 105°C for 24 hours in Steps 13 through 16 of Section C.4.4. The masses of the dried, centrifuged supernatant liquid (M_{DCL}) and dried, centrifuged solids (M_{DCS}) were then measured under Step 16 of Section C.4.4. Assuming that all mass lost during the drying process is water and not another volatile component, the weight percent total solids in the bulk slurry was calculated ($P_{MTS} = \{[(M_{DCL} \times M_S)/(M_{VL} \times M_B)]+[M_{DCS}/M_B]\} \times 100$ %). Waters of hydration or volatile organics can lead to low bias in M_{DCS}.

10. Wt % Oven-Dried Solids

This is the mass of dried, centrifuged solids (M_{DCS}) measured in Step 16 of Section C.4.4 divided by the mass of centrifuged solids (M_{CS}) that was measured in Step 10.

$$P_{ODS} = M_{DCS} / M_{CS} \times 100\%$$

11. Wt % Undissolved Solids

An additional calculation is performed to determine the wt % solids in the samples excluding all interstitial liquid. We have referred to this as "wt % undissolved solids". This wt % undissolved can be thought of as the solids left if all the supernatant liquid could be drained from the bulk slurry.

The following equation is used:

$$Wt\ \%\ undissolved\ solids = \left(1 - \frac{1 - \frac{M_{DCS}}{M_{CS}}}{1 - \frac{M_{DCL}}{M_{VL}}} \right) \times \frac{M_{CS}}{M_B} \times 100\ \%$$

This calculation assumes 1) that the supernatant liquid and the interstitial liquid have the same composition, and 2) that all mass loss during the drying of the centrifuged solids is water loss from interstitial liquid. Record results of calculation in Table C.5 (Physical Properties Calculation Sheet) and transfer appropriate data to Table C.8.

Table C.5. Physical-Properties Calculation-Sheet Format

Calc. Step	Description	Units	Subsample A	Subsample B	Subsample C	Average	Std. dev.
1	Bulk Density $\rho_B = M_B/V_B$	g/ml					
2	Vol % Settled Solids $P_{VSS} = 100\ \% \times V_{SS}/V_B$	%					
3	Density of Centrifuged Solids $\rho_{CS} = M_{CS}/V_{CS}$	g/ml					
4	Vol % Centrifuged Solids $P_{VCS} = 100\ \% \times V_{CS}/V_B$	%					
5	Wt. % Centrifuged Solids $P_{MCS} = 100\ \% \times M_{CS}/M_B$	%					
6	Supernatant Liquid Density $\rho_S = M_S/V_S$	g/ml					
7	Density of Settled Solids $\rho_{SS} = 100\ \% \times M_{SS}/V_{SS}$	g/ml					
8	Wt. % Settled Solids $P_{MSS} = 100\ \% \times M_{SS}/M_B$	%					
9	Wt. % Total Solids $P_{MTS} = \{[(M_{DCL} \times M_S)/(M_{VL} \times M_B)] + [M_{DCS}/M_B]\} \times 100\ \%$	%					
10	Wt % Oven-Dried Solids $P_{ODS} = M_{DCS} / M_{CS} \times 100\%$	%					
11	Wt. % Undissolved Solids $P_{MUS} = \left(1 - \frac{1 - \frac{M_{DCS}}{M_{CS}}}{1 - \frac{M_{DCL}}{M_{VL}}} \right) \times \frac{M_{CS}}{M_B} \times 100\%$	%					

Analyst: Date:

C.5.5 Work Guidelines (Wt % Total Oxides)

Note: *Record all data including Measurement & Test Equipment information in Table C.6.*

1. Mix the bulk-slurry-source sample to combine any separated liquid and solid phases.
2. Record the tare mass (±0.001 g) of three pre-fired crucibles rated for at least 1200°C on the Wt % Oxides Data Sheet (Table C.6).
3. Transfer sub-samples of approximately 3 or more grams to each crucible. Record the mass (±0.001 g) on the Wt % Oxides Data Sheet (Table C.6).
4. Air-dry the material in the crucibles overnight to minimize splattering during the next drying step. Consult the cognizant scientist on the use of a heat lamp or other drying technique to speed up this preliminary drying.
5. Transfer the solids crucibles to an oven at 105°C for 24 hours. Record the time and date on the Wt % Oxides Data Sheet (Table C.6).
6. Remove the crucibles from the drying oven. Allow to cool ~1 hour and weigh. Record the time, date and weight on attached Wt % Oxides Data Sheet (Table C.6).
7. Return the crucibles to an oven at 105°C for at least 24 hours. Record the time and date again on the Wt % Oxides Data Sheet (Table C.6).
8. Remove the crucibles from the drying oven. Allow to cool ~1 hour and weigh. Record the time, date and weight on attached Wt % Oxides Data Sheet (Table C.6).
9. As directed by the cognizant scientist, repeat heating and reweighing steps until the samples reach a stable mass. Record all data on attached Wt % Oxides Data Sheet (Table C.6).
10. Place the crucibles in the furnace.
11. Heat the furnace and hold for 30 minutes between 1000 and 1050°C.

Caution: *If sample is cooled too fast in Step 12, the crucible could shatter. Take appropriate precautions to mitigate hazards to personnel and equipment. This may need to be addressed in facility-specific documentation.*

12. Allow sample to cool to 150°C. This can be accomplished by turning off the furnace, lowering the furnace temperature settings, or removing the sample from the furnace. Proceed to next step after sample has reached this temperature.
13. Using tongs, transfer the crucibles to a desiccator or an appropriate covered, stainless-steel tray containing desiccant and cool to ambient temperature (~1 hour).
14. Weigh the crucibles. Record all data on attached Wt % Oxides Data Sheet (Table C.6). Perform the pertinent calculations as required in the calculation section below.

Table C.6. Wt % Oxides Data Sheet Format

Balance 1:	Calibration ID _____		
	Calibration Expiration _____	Location	_____

Balance 2: (if needed)	Calibration ID _____		
	Calibration Expiration _____	Location	_____

Thermocouple:	Calibration ID _____		
	Calibration Expiration _____	Location	_____

Digital Thermometer:	Calibration ID _____		
	Calibration Expiration _____	Location	_____

Institution	Analyst	Sample	Preparation Date

Wt % Oxides — Data Sheet Format Sample ID: _____

Step	Description	Units	Crucible A	Crucible C	Crucible C
2	Pre-fired crucible tare	g			
3	Crucible + sample wet material	g			
	Net wt. sample wet material M_{WCS} = Step 19 – Step 18	g			
6	Stable dry wt. @ 105°C.	g			
	Stable dry sample mass @ 105°C. M_{OSC} = Step 25 – Step 18	g			
14	Total Wt. after 1000-1050°C.	g			
	Net Wt. after 1000-1050°C. M_{FSC} = Step 30 – Step 18	g			

Table C.6. (contd)

Step 6) Record drying data below until a stable mass is reached after drying at 105°C (weight measured after cooling at ambient temperature for one hour).

Time	Date	Crucible A	Crucible B	Crucible C
Stable mass at 105°C, g				

Step 14) Record calcined weight data after heating for 60 minutes at 1000-1050°C. (Note: weight is measured after cooling sample to 150°C, placing sample into a desiccator, and allowing it to cool to ambient temperature.)

Time	Date	Crucible A	Crucible B	Crucible C

C.5.6 Calculations – (Wt % Total Oxides)

Note: *Calculations described below should be recorded in Table C.7.*

1. Wt % Dried Sample Fraction after 105°C

The percent mass of total solids in the sample (P_{MDS}) can be calculated as follows:

$$P_{MDS} = 100\ \% \times M_{OSC}/M_{WCS}$$

where M_{OSC} = mass of the oven dried (105°C) sample (g)
M_{WCS} = mass of the wet sample (g)

2. Wt % Total Oxides

The percent mass of oxides in the sample (P_{MOX}) can be calculated as follows:

$$P_{MOX} = M_{FSC}/M_{WCS} \times 100\ \%$$

where M_{FSC} = mass of the oven fired (~1000°C) sample (g)
M_{WCS} = mass of the wet sample (g)

Transfer appropriate data to Table C.8.

Table C.7. Wt % Oxides Calculation-Sheet Format

Wt% Oxides — Calculation Sheet Sample ID: _____

Calc. Step	Description	Units	Crucible A	Crucible B	Crucible C
11	Wt% Dried Sample fraction after 105°C $P_{MDS} = 100\ \% \times M_{OSC}/M_{WCS}$	%			
12	Wt % Oxides after 1050°C $P_{MOX} = 100\ \% \times M_{FSC}/M_{WCS}$	%			

Prepare: _____ Date: _____

Review: _____ Date: _____

_____ _____

Table C.8. Physical-Property Data Summary

Physical Property	Values (units)
Sodium concentration of LAW waste or pretreated waste (Molarity)	
Oxides loading of HLW sludge or pretreated sludge (total grams oxide/Liter)	
pH (aged 1day, 1week, 1mo)	
Solid phases present	
Density – bulk slurry (g/ml) (aged 1day, 1week, 1mo)	
Density – settled solids (g/ml)	
Density – centrifuged solids (g/ml)	
Density - supernatant liquid (g/ml)	
Vol. % settled solids after 72 hours (aged 1day, 1week, 1mo)	
Vol. % centrifuged solids	
Wt % total dried solids	
Wt % centrifuged solids	
Wt % oven-dried solids	
Wt % undissolved solids	
Wt % dissolved solids	

C.6 Rheological-Properties Measurements

C.6.1 Purpose/Scope

This section of the guideline describes the process for measuring rheological properties of solutions, slurries, sludges, and associated simulants. Samples of actual tank-waste received and previously composited, homogenized, and sub-sampled under this project undergo further process-testing and characterization.

The test methods in this section may be used by other DOE project tasks provided they involve materials of similar chemical and physical properties. Specific instructions within project-approved test specifications may be used to deviate from the specified property measurements delineated in this section accompanied with the technical rationale for said deviation.

C.6.1.1 Applicability

This guideline will be used to measure the rheological properties listed below for solutions, slurries, and sludges. Not all the rheological properties listed below may be measured for a particular sample.

1. shear strength
2. shear stress versus shear rate
3. yield stress

This guideline will be used for characterization of radioactive and non-radioactive materials.

C.6.1.2 Nomenclature for Shear-Strength Measurements

B – The steady-state torque in a shear-strength test (N•cm)

D – The diameter of the shear vane (cm)

$\mathbf{D_T}$ – The diameter of the shear-strength sample cup (cm)

H – The height of the shear vane (cm)

N – Rotational rate of shear vane (rpm)

$\mathbf{R_t}$ – The maximum torque in a shear-strength test (N•cm)

$\mathbf{Z_1}$ – Height of sample above the top of the immersed shear vane (cm)

$\mathbf{Z_2}$ – Height of sample below the top of the immersed shear vane (cm)

$\boldsymbol{\tau_o}$ – Shear strength (Pa)

C.6.1.3 Nomenclature for Shear-Stress-versus-Shear-Rate Measurements

b – Herschel-Bulkely power-law exponent

k – Herschel-Bulkely consistency coefficient ($Pa \bullet s^{-b}$)

n – Power-law exponent

m – Consistency coefficient ($Pa \bullet s^{-n}$)

τ – Shear Stress (Pa)

τ_O^B – Bingham yield stress (Pa)

τ_O^H – Herschel-Bulkley yield stress (Pa)

η_p – Plastic viscosity ($Pa \bullet s^{-1}$)

$\dot{\gamma}$ – Shear rate (s^{-1})

C.6.1.4 Definitions

Apparent Viscosity – The measured shear stress divided by the measured shear rate.

Interstitial Solution – The solution contained within the sludge sample.

Newtonian Fluid – A fluid whose apparent viscosity is independent of shear rate.

Non-Newtonian Fluid – A fluid whose apparent viscosity varies with shear rate.

Rheogram or Flow Curve – A plot of shear stress versus shear rate.

Sludge – Wet solids having little or no standing liquid (mud-like).

Slurry – A mixture of solids and solution.

Solution – A liquid phase possibly containing dissolved material.

Yield Stress – The minimum stress required to initiate fluid movement as determined by a flow curve using a rheological model.

Shear Strength – The minimum stress required to initiate fluid movement as determined by the vane method.

C.6.2 Quality Control

Quality control has been integrated into work guidelines.

C.6.3 Calibration Check

1. Calibration checks are to be performed on viscometers used under these protocols at least once every 30 days that data is generated.
2. Calibration checks are to be performed using certified NIST-traceable viscosity standards.

3. The viscosity standard and rheological program used for the calibration check should be appropriate for the torque and rotation rate used during the analysis. Since the calibration check assesses torque and rotational sensors within the viscometer, a specific viscosity standard is not appropriate for all instruments and applications. Consult the cognizant scientist on the appropriate standard, sensor, and test conditions.
4. The calibration check must be within ± 10% of the certified viscosity standard value. If the check is outside this range, consult the instrument manual for acceptable errors and recalibrate as necessary.
5. Include which viscosity standard was used and calibration data in the final report.

C.6.4 Simulant Information

For simulants tested under these protocols, include the simulant formulation as well as the date the simulant was prepared in the final report.

C.6.5 Shear-Strength Work Guideline

Yield stress and shear strength are measures of the pressure required to transition a material to fluid behavior. Under this protocol, the shear strength will be characterized using the vane method developed by Dzuy and Boger (1985). The general vane geometry and minimum specifications are presented in Figure C.1. Additional guidance and reference are provided by Heath (1986), and Barnes and Nguyen (2001). When applied to samples under vitrification tasks, this yield-stress measurement is to be performed on two duplicate samples.

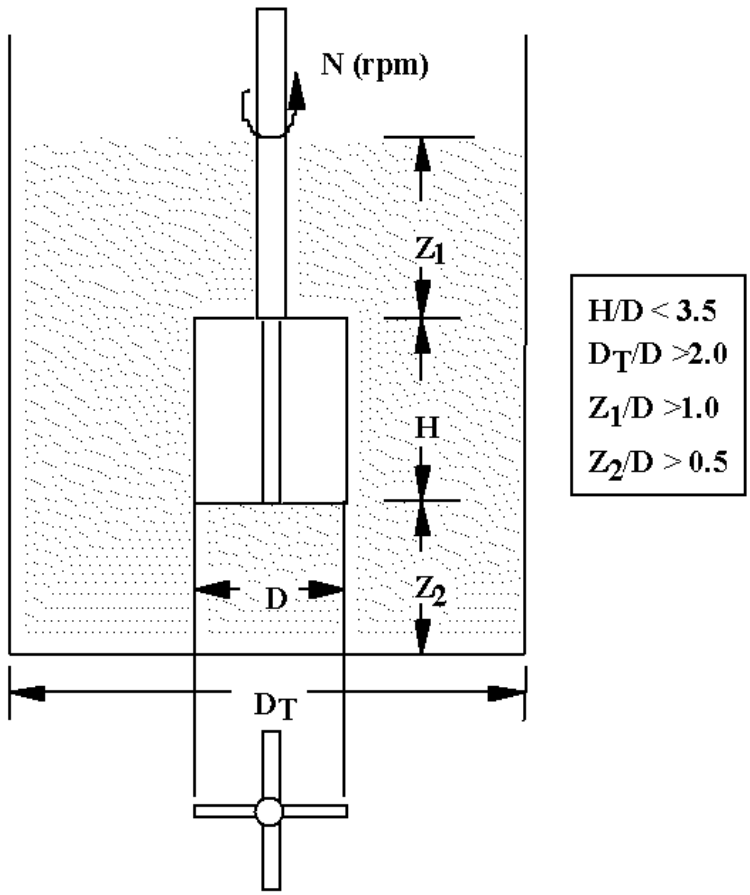
1. Allow the sample to sit undisturbed for at least 48 hours before testing. A lid should be placed on the sample container to minimize evaporative losses. The sample should remain at the analysis temperature during the period (i.e., if shear strength is to be measured at 40°C, then the sample should sit at 40°C for at least 48 hours prior to testing).
2. Install the shear vane on the viscometer. Vane dimensions of D = 16 cm and H = 1.6 cm are preferred.
3. Put the sample on a lab jack or stage and position under the shear vane.
4. Zero the viscometer.

Caution: *When raising the sample, take care to avoid sideways movement of the sample. This could deform the sample and result in a low value.*

1. Carefully raise the sample until the shear vane is immersed to a depth such that $Z_1/H=1$. It is recommended to pre-mark the shaft since it is difficult to judge depth otherwise.
2. Verify that the torque is changing at this time, since at this time it is normal for the viscometer to read a nonzero torque. Do not re-zero.

Note: *The preferred rate for the Haake M5 head is 0.3 rpm, although values of up to 0.6 rpm are acceptable based on alternative viscometer capabilities. The [0] torque reading should quickly climb at a roughly linearly rate, level out reaching a maximum torque (R_t), and then decline to a steady-state value (B).*

1. Set the viscometer program to operate at a fixed rotational rate (N) for a period of at least 2 minutes. The preferred rotational rate is 0.3 rpm. A rotational rate of 0.6 rpm may also be used.
2. Initiate the analysis as soon as possible.
3. Record H, D, D_T, Z_1, Z_2, N, B, and R_t. Results are to be recorded in a laboratory record book, test instructions, or associated bench sheet. A copy is to be transferred to the project file.
4. Calculate the shear strength τ_o using the following equations.
5. Transfer appropriate data to Table C.9. In the data report, include H, D, D_T, Z_1, Z_2, N, B, R_t, and τ_o. Also include a plot of torque versus time and printouts of the raw torque and time data in tabular form in an Excel® spreadsheet. Use the format provided in Table C.9.



Where R_t is the maximum torque in N•cm

D is the diameter of the vane in cm
H is the height of the vane in cm

Figure C.1. Vane Geometry Measurement Dimensions (Dzuy and Boger 1985)

Table C.9. Example Excel® Spreadsheet Reporting Format for Shear Strength

	A	B	C	D
1	Date			

	A	B	C	D
2	Substance			
3	File Name			
4	Operator			
5	Sensor			
6	Measurement System			
	Temperature (°C)			
7	M Factor should be set to 1 (R-series Pa/N•m) (M-series: Pa/100 % scale)			
8	A Factor should be set to 1 ([1/sec]/[radians/sec])			
9	H (cm)			
10	D (cm)			
11	D_T (cm)			
12	Z_1(cm)			
13	Z_2(cm)			
14	N (rpm)			
15	Torque at 100 % Scale (N•m) (R-series: N/A) (M5: 0.049 N•m)			
16	Time (sec)	Torque (N•cm) R-series: Torque = (Shear Stress)/(100 × M Factor) M-series: Torque = (Torque at 100 % Scale × Shear Stress)/ (100 × M Factor)	Shear Rate (1/s) 0.3 rpm = 0.3 × (2π/60) radians/sec = 3.142^{-3} radians/sec 0.6 rpm= 6.283^{-3} radians/sec	Shear Stress (Pa)
17				
18				
19				
20				
21				
22				
23				

Note: Data is reported in cells with heavy outlines.

C.6.6 Shear-Stress-versus-Shear-Rate Work Guideline

All samples are to be characterized for shear-stress versus shear-rate using the flow-curve parameters detailed in this section. Flow-curve testing parameters and data will be recorded in Table C.10.

1. General parameters:
   a. Sensor
      i. Haake M5 – The MV1 is preferred for all samples with high solids content (e.g., HLW Sludge, HLW and LAW Melter Feed), while the NV1 is preferred for solutions with little or no solids (e.g., LAW Waste).
      ii. Haake RS- The Z41 is preferred for all samples with high solids content (e.g., HLW Sludge, HLW and LAW Melter Feed), while a double-gap sensor comparable to the NV1 is preferred for solutions with little or no solids (e.g., LAW Waste).
   b. The shear rate is to be linearly increased from 0 to 1000 s^{-1} (or the maximum shear rate of the selected sensor) at a rate of 200 s^{-1} per minute. The shear rate is then held at 1000 s^{-1} (or the maximum) for one minute. The shear rate is then linearly decreased to 0 at 200 s^{-1} per minute.
   c. Temperature - all analyses are to be conducted at 25 and 40°C. Test temperatures of 25 and 40°C were selected based on process requirements and are consistent with subcontractor capabilities. The test temperature of 25°C is representative of ambient conditions supporting simulant development and provides a lower bound for potential plant conditions. The temperature of 40°C is representative of the upper end of the anticipated plant conditions.
   d. Reanalysis - Analyses are to be conducted twice on the same sample. If the results for the second analysis are lower than the first, then a third analysis is to be conducted with the same sample. These replicates will be used to assess the sample's sensitivity to shear history.
   e. Duplicates - at least two aliquots of each sample are to be characterized by this flow-curve protocol (i.e., if 3 analyses are required under Step d, then a total of six analyses will be performed).
2. Confirm that an acceptable calibration check has been performed in the past 30 days as defined in the general instructions.
3. Select and install the appropriate sensor as specified in Step 1.a.i.
4. Set the viscometer temperature to the test temperature and allow sufficient time for the system to equilibrate thermally.
5. If the sample has separated because of settling, invert the sample jar, or use a spatula to gently mix the sample.

Note: *It is important that the sample be at temperature before initiating the analysis in the following step. For samples stored at room temperature (~23°C), or sensors requiring large sample volumes (like the MV1) it may be necessary to preheat the samples at least 1 hour before the analysis. For samples stored in hot cells (~35°C) or small sample-volume sensors (like the NV1), preheating is not critical.*

6. Transfer a sub-sample to the viscometer and initiate the analysis as soon as possible.
7. Save the analyses using any vendor format as well as comma-separated x-y variable format.

8. Examine the rheogram for Taylor vortices. Taylor vortices are the result of a secondary flow that occurs as the inner cylinder of the concentric-cylinder instrument rotates. Taylor vortices result from analyzing a material at too high a shear rate. All data collected at shear rates above the onset of Taylor vortices is invalid. Taylor vortices are anticipated when the following two equations are satisfied [Chang 1996]:

$$R_e > 41.3[R_2/(R_2-R_1)]^{1/2}$$

$$Re = \dot{\gamma}\,(R_2-R_1)^2\,\rho\,/\mu$$

where R_1 = Radius of Inner cylinder (0.02004m for MVI)
R_2 = Radius of Outer cylinder (0.02100m for MVI)
$\dot{\gamma}$ = Shear Rate (s^{-1})
ρ = density (g/ml)
μ = Apparent viscosity (Pa•s)

9. Report any Taylor vortices observed and discard this part of the dataset prior to modeling in the next step.

10. Model the increasing shear-rate curves (excluding portions affected by Taylor vortices) using the following models:

    a. Ostwald (or power-law) model

$$\tau = m\dot{\gamma}^n$$

where m = Consistency Coefficient
n = Power Law Exponent
$\dot{\gamma}$ = Shear Rate

If n<1, then the material is referred to as pseudoplastic (shear-thinning). If n>1 that material is referred to as dilatant (shear-thickening). Since dilatant flow behavior is rare, dilatant behavior is an indication of possible Taylor vortices or other measurement errors.

    b. Bingham-Plastic model

$$\tau = \tau_O^B + \eta_P\dot{\gamma}$$

where τ_O^B = Bingham Yield Stress
η_p = Plastic Viscosity
$\dot{\gamma}$ = Shear Rate

c. Herschel-Bulkley model

$$\tau = \tau_O^H + k\dot{\gamma}^b$$

where τ_O^H = Yield Stress
K = Herschel-Bulkely Consistency Coefficient
B = Herschel-Bulkely Power Law Exponent
$\dot{\gamma}$ = Shear Rate

11. Include the Ostwald, Bingham, and Herschel-Bulkley fit parameters along with the associated r^2 values in the report. Place a constraint on the Herschel-Bulkley yield stress to be equal to or greater than zero; a negative yield stress is not realistic. Transfer appropriate data to Table C.11.

12. In the data report, include printouts of the raw shear-stress-versus-shear-rate data in tabular form in an Excel® spreadsheet. Use the following format:

Table C.10. Example Excel® Spreadsheet Reporting Format for Flow-Curve Testing Parameters and Data

	A	B	C	D
1	Date			
2	Substance			
3	File Name			
4	Operator			
5	Sensor			
6	Measurement System			
7	Temperature (°C)			
8	M Factor			
9	A Factor			
10	Shear Rate (1/s)	Shear Stress (Pa)	Viscosity (cP)	Time (min)
11				
12				
13				
14				
15				
16				
17				

Note: Data is reported in the cells with heavy outlines.

Table C.11. Rheological Property Data Summary

Model/Model Parameter	Parameter Value
Shear Strength (by Vane Method)	
τ_O – Shear Strength (Pa)	
Ostwald (or Power Law)	
m – the consistency coefficient (cP)	
n – the power law exponent	
R^2 – correlation coefficient	
Bingham Plastic	
τ_O^B – the Bingham yield stress (Pa)	
η_p – the plastic viscosity (cP)	
R^2 – linear correlation coefficient	
Herschel-Bulkley	
τ_O^H - the yield stress (cP)	
k – the Herschel-Bulkely consistency coefficient (cP)	
b – the Herschel-Bulkely power law exponent	
R^2 – correlation coefficient	

C.7 Particle-Size Distribution (PSD) Measurement Method

C.7.1 Overview

The goal of a consensus PSD-measurement method description is to produce comparable, accurate PSD results regardless of the performer-of-work/subcontractor/etc.. This can be accomplished through the use of standardized sample preparation, sample analysis, and data reporting consistent with ISO 13320-1(Particle-size analysis – Laser-diffraction methods – Part 1: General Principles). The proposed method is designed to be general enough in nature that the procedure could be applied to any of the laser-diffraction instruments currently used by most DOE subcontractors.

C.7.1.1 Instrument Location

In order to provide accurate, repeatable measurements, ISO 13320-1 recommends placing the laser-diffraction instrument in a location away from electrical noise, mechanical vibration, temperature gradients, and direct sunlight. The area should be well-ventilated and the laboratory bench should be sturdy to avoid misalignments of the optical system.

C.7.1.2 Dispersion Liquid

Selection of the dispersion (or suspending) liquid can significantly affect the results of a particle-size measurement. ISO 13320-1 recommends a liquid with a refractive index that is known at the laser wavelength of the instrument to be used (see Table C.12).

Table C.12. Examples of Laser Wavelengths Used at DOE Laboratories

Instrument	Laser Wavelength (nm)	Radiological Samples	Non-Radiological Samples
Microtrac S3000	780	none	SRNL
Microtrac X100	780	PNNL, SRNL	PNNL
Microtrac UPA150	632.8	PNNL	PNNL

The dispersion liquid should also meet the following criteria:

a. Be transparent at the laser wavelength (see Table C.12);

b. Be compatible with materials used in the instrument (O-rings, tubing, etc.);

c. Not dissolve or alter the size of the particulate material;

d. Be essentially free from air bubbles or other particles;

e. Favor easy and stable dispersion of particulate material;

f. Have a refractive index which significantly differs from the particulate material. (See Annex D of ISO 13320-1 for a list of refractive-index values for various solids and liquids);

g. Have a viscosity that will allow recirculation;

h. Meets safety and health requirements.

Because the suspending fluid may alter the sample PSD through, among various methods, dissolution of sample particles (see requirement c above), the chemistry of the dispersion liquid must closely match that of the actual waste liquid. Use of actual tank-waste supernate is often not feasible because the system requires ~200-300 ml of suspending fluid. An aqueous-phase tank supernate simulant should be used as a dispersion liquid. The recipe for the simulant material should be based on existing data from the source-tank interstitial liquid. This recipe should be supplied to the project performer-of-work/subcontractor/etc. a minimum of two weeks before particle-size analysis. This two-week period should allow enough time for the project performer-of-work/subcontractor/etc. to procure the necessary chemicals and manufacture a quantity of simulant sufficient for several (e.g., three) particle-size analysis runs (approximately 2 liters). The system should be filled and drained with the dispersion liquid prior to beginning a series of actual data measurements; this is done to minimize cross-contamination and dilution of the suspending medium.

If the tank supernate-simulant material possesses properties that are incompatible with the instrument (see requirement b), other formulations may be recommended by the project performer-of-work/subcontractor/etc., or organic liquids may be chosen. However, the use of organic liquids should be avoided as it may alter the PSD (see requirement c) through crystallization of dissolved material and formation of micelles. Glycerol solutions for particle-size measurements of actual Hanford tank waste slurries have been used successfully (Shaver 1993).

C.7.1.3 Sample Inspection

ISO 13320-1 recommends that the sample material be inspected either visually or with the aid of a microscope. The purpose of this step is to estimate the particle size and shape prior to analyses and determine whether the particles are adequately dispersed. Note that this is not currently in the scope of most WTP tasks; however, if scanning-electron-microscopy (SEM) images are part of the scope of work on the project sample, these SEM images can be used for particle-size and -shape estimation.

C.7.1.4 Sample Preparation

ISO 13320-1 recommends that the sample should initially be a well-mixed paste. The consistency of the paste should be high enough to avoid segregation errors. This paste could be formed by allowing the sample to settle for 48 hours prior to analysis. The free-standing supernate liquid should be removed from the sample via decant or pipette. Care should be taken not to remove any fine material from the top of the settled sludge layer. A large-bore pipette should be used to thoroughly mix the settled sludge layer. If the sample is too thick to effectively pipette the paste, standing supernate from the previous separation should be added drop-wise until an adequate consistency is obtained. ISO 13320-1 recommends a consistency similar to honey or toothpaste. If the paste becomes too fluid by mistake and particle settling becomes an issue, it shall not be used, and a new preparation should be initiated. This new preparation can be obtained by recombining the separated supernatant liquid and paste, mixing, and letting the sample settle for 48 hours.

C.7.1.5 Sample Dispersion

ISO 13320-1 recommends that stirring and ultrasonication be used to facilitate proper dispersion of the particles in the dispersion liquid. For the Microtrac X100 and S3000 laser-diffraction instruments, dispersion can occur due to the shear induced by circulating the particles through the flow loop. If available, inline ultrasonication of the test suspension should be used in accordance with the measurement procedure described in Section C.7.2.

For the Microtrac UPA150 particle-size distribution analyzer, no flow or ultrasonication options are available. For this instrument, the sample should be mixed in the sample cell with a pipette prior to analysis. Ultrasonication with a separate ultrasonication horn may also be performed on the sample prior to loading into the UPA150 sample cell.

C.7.1.6 Sample Concentration

For instruments with manual sample-concentration settings, ISO 13320-1 discusses the need to keep the particle concentration in the dispersion above a minimum level, which for many instruments corresponds to about 5% obscuration, in order to produce an acceptable signal-to-noise ratio in the detector. Likewise it should be below a maximum level, which for many instruments will correspond to about 35% obscuration for particles larger than about 20 μm, in order to avoid multiple scattering (where light is scattered subsequently at more than one particle). For particles smaller than about 20 μm, the obscuration value should be kept below about 15% for the same reason.

For the Microtrac instruments with automatic sample-concentration settings, the necessary particle concentration will be determined by the instrument software during the sample-loading phase of the measurement procedure described in item 7.

C.7.2 Measurement Procedure

ISO 13320-1 states that the following measurement steps are required in order to produce a particle-size measurement. This procedure should be followed during the calibration-check phase (see Section C.C.7.4) and sample-measurement phase.

C.7.2.1 Blank Measurement

A blank measurement is performed on a particle-free dispersion liquid. The detector data are saved in order to subtract them later from the raw sample data to obtain net sample results.

C.7.2.2 Measurement of Scattering Pattern of Dispersed Sample

Generally, a measuring time allowing for a large number of detector sweeps at short time intervals is used, typically 2 seconds or 1,000 sweeps.

Selection of an Appropriate Optical Model

Most often either the Fraunhofer or the Mie theory is used. When using the Mie theory, the refractive indices of the particulate and medium, or their ratio, should be brought into the instrument to allow calculation of the model matrix. To obtain traceable results, it is essential that the refractive-index values used are reported.

Conversion of the Scatter Pattern into a PSD

This deconvolution step is the inverse of the calculation of a scattering pattern for a given PSD. Several mathematical procedures have been developed by different instrument manufacturers and integrated into the instrument software package.

For project samples, the instrument's operating manual should be used to select the appropriate settings. The discussion that follows is a summary of the PNNL-recommended instrument settings for WTP samples using the Microtrac X100, S3000 and UPA instruments:

C.7.2.3 Instrument Flow Rate

The Microtrac X100 and S3000 instruments circulate a suspending fluid through the laser-scattering diffraction-detection cell. The flow rate and density of the suspending fluid have an impact on the concentration of particles of a given size in the detector cell, due to particle settling within the instrument. Therefore, Microtrac has established guidelines on the flow-rate settings based on these parameters (see Figure C.2). In the case of the approved WTP glass formers, rutile has the fastest settling rate with a density of 4.25 g/ml and a maximum particle-size diameter of approximately 105 μm. Therefore, the graph in Figure C.2 indicates that a nominal flow setting for a WTP sample should be approximately

80 ml/sec. Due to bubble formation, flow rates above 80 ml/sec typically cause high-background flags during the blank-measurement phase. Therefore, 80 ml/sec should be considered the maximum flow rate for the Microtrac X100 and S3000 instruments.

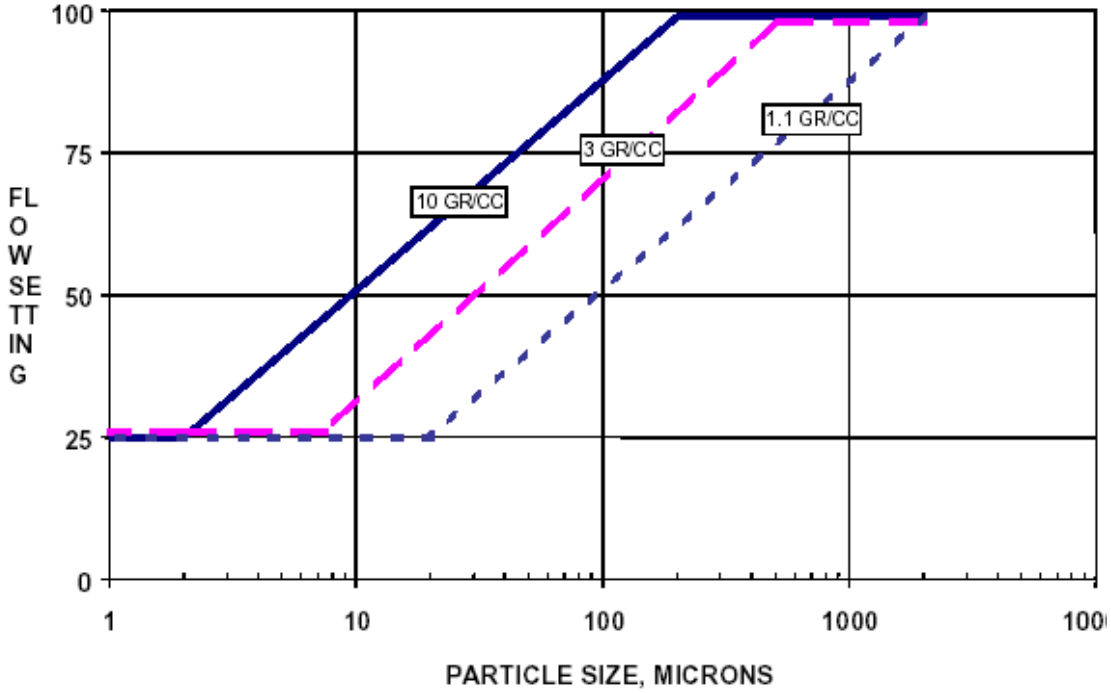


Figure C.2. Recommended Flow Rate (mL/sec) as a Function of Particle Density and Particle Size

Typical WTP Project results for melter-feed samples indicate that peaks are observed in the PSDs at about 3-5 and 20-30 µm. The required flow rate should increase with particle size and density. A particle size of 30 µm should provide a large enough basis for this to provide an adequate circulation of the expected larger particles. This would correspond to a minimum recommended flow rate of 50 ml/sec, based on the diagram provided by Microtrac (see Figure C.2), as a trade-off between optimum circulation and bubble prevention for an average particle density of ~3.0 g/ml.

C.7.2.4 Inline Ultrasonication

The Microtrac X100 and S3000 instruments can sonicate the fluid during flow to disperse the samples. The effect on particle size of agglomerate ultrasonication can be seen in Figure C.3. Most of the dispersion occurs within the first 90 seconds. Microtrac warns that ultrasonication longer than a 90 sec time period could lead to particle fracture and inaccurate results. Additionally, bubble formation can occur after 30 seconds of ultrasonication. Therefore, the ultrasonication will be applied in 30 second intervals not to exceed 90 seconds total ultrasonication time. After an ultrasonication interval, the material should recirculate for 30 seconds before another ultrasonication interval or beginning a measurement. The maximum recommended power setting for the Microtrac X100 is 40W.

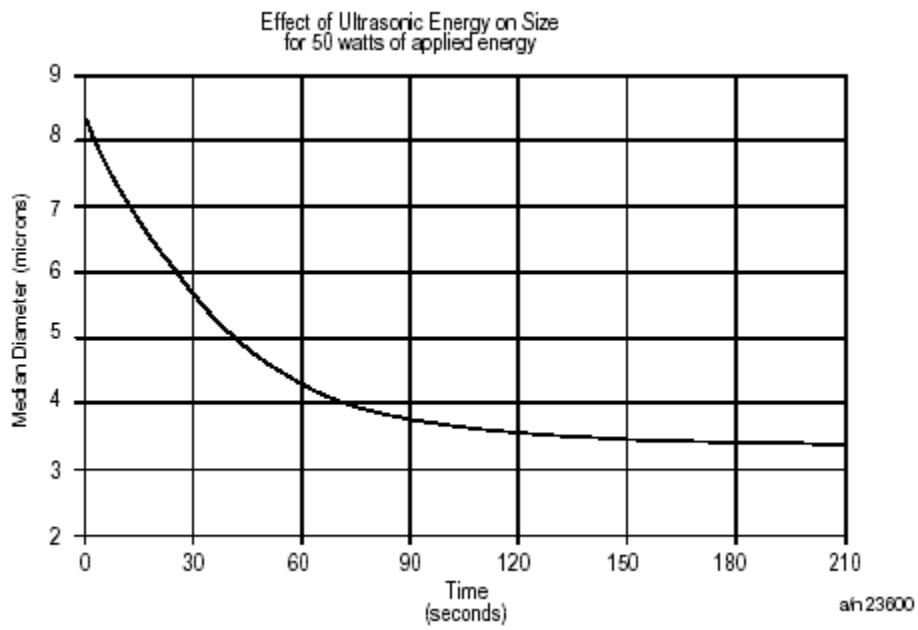


Figure C.3. Effect of Ultrasonication on Agglomerate Particle Size Based on 50 W of Applied Power

To retain comparability to instruments without ultrasonication capabilities, unsonicated data should also be obtained. Therefore, two measurements with either the Microtrac X100 or S3000 shall be obtained under the following set of conditions:

1. no ultrasonication;
2. ultrasonicated (two 30-second ultrasonication periods at 40 W power for the Microtrac X100 or S3000).

C.7.2.5 Measuring Time

The run time of the instrument should be consistent between runs and should be the same for all project performers-of-work/subcontractors/etc. No guidance for run time could be found in the Microtrac X100 instrument manual. However, the following statement was found in another Microtrac instrument operating manual (Microtrac II; the precursor to the Microtrac X100) that indicates that a 30-second minimum run time is valid:

> *In general, the longer the sampling time, the more precise the results. With a little experience, you should easily be able to select the shortest time that gives the degree of precision you need for your particular purpose. (Try 30 seconds, which is fairly typical, to start). Note that random error is approximately proportional to* $\sqrt{1/t}$ *, where t is the sampling time.*

A 60-second run time has been used successfully in previous measurements at PNNL. A justification for using a different value-measurement time should be clearly stated in any generated report.

For the UPA instrument, no ultrasonication or flow options are available. Therefore, the sample is placed in the instrument and the measurements are performed on the as-received, stationary material. However, Microtrac recommends that the measurement period be determined in accord with Table C.13.

Table C.13. UPA Specific Run Time

Particle Size Range (nanometers)	Minimum Run Time (seconds)
Below 60	60
60 to 300	180
300 to 900	360
Above 900	600

C.7.2.6 Refractive-Index Selection

Since the refractive indices of the sample and simulants are unknown, a typical value of 1.54 for the particle refractive index (which corresponds to quartz sand) and 1.33 for the fluid refractive index (which corresponds to water) should be used unless true values are known. Refractive indices of other materials can be found in Annex D of ISO 13320-1. For example, polystyrene, a common particle-size standard material, has a refractive index of approximately 1.59.

The following is a justification for the selection of the refractive indices recommended above. According to NIST (Jillavenkatesa et al. 2001), two major theories are used in laser-diffraction measurement analysis. One is the Mie theory and the other is the Fraunhofer approximation. When using Mie theory, refractive index is a significant parameter for particles smaller than approximately 2 μm. The Fraunhofer approximation does not require refractive index and is inaccurate for particles under 2 μm in size. Most modern instruments, including the Microtrac instruments used by the WTP subcontractors, possess enough computational power that Mie theory is implemented. Since most of the engineering design for DOE projects will be based on particle sizes above 2 μm, refractive indices of the solution and particles should not be considered a significant impact to the PSD results for this application. Consequently, the approximations discussed above will have a minimal effect on the particle-size results. However, pretreatment operations may need to consider refractive index in performing PSD measurements, because much of the entrained solids may include an appreciable fraction of particles smaller than 2 μm.

The Microtrac instrument setting for particle opacity should be set to “Absorb.” This corresponds to the imaginary portion of the complex refractive index for the particles. While a numerical value cannot be specified for the Microtrac X100 and S3000 instruments, the “Absorb” setting indicates a large value for the imaginary portion of the complex refractive index.

C.7.2.7 Histogram Progression

It is recommended that the maximum number of histogram bins be selected to provide a distribution of the highest resolution for a particular instrument.

If possible, a distribution should be chosen that is consistent with the Microtrac-based histogram. The Microtrac particle-size results are saved in the form of a histogram with varying bin sizes. Consistent bin-border values between instruments would lead to comparable results. The upper range of each bin is determined by a root-8 geometric sequence (see equation below):

$$\hat{d}_{i+1} = \left(\hat{d}_i\right) \cdot 2^{-\frac{1}{m}} \tag{1}$$

where $\hat{d}_1 = 704.0\mu\text{m}$ for the X100

$\hat{d}_1 = 1428.0\mu\text{m}$ for the S3000

$m = 8$ for the X100 and S3000

$\forall i = 1...100$ for the X100 and S3000

$\hat{d}_1 = 6.541\mu\text{m}$ for the UPA

$m = 8$ for the UPA

$\forall i = 1...87$ for the UPA

	X100	S3000	UPA
$\hat{d}_1$, um	704.0 μm	1428.0 μm	6.541 μm
m	8	8	8
$\forall i$	1…100	1…100	1…87

The lower range for each bin is determined as follows:

$$\breve{d}_i = \hat{d}_{i+1} \tag{2}$$

where $\breve{d}_{100} = \frac{\hat{d}_{100}}{\sqrt[8]{2}} = 0.122\ \mu\text{m}$ for the X100; $0.243\ \mu\text{m}$ for the S3000

$\breve{d}_{88} = \frac{\hat{d}_{88}}{\sqrt[8]{2}} = 0.0032\ \mu\text{m}$ for the UPA

The Microtrac histogram information above shows that these instruments possess varying measurement ranges. It is recommended that measurements cover the complete range of particle sizes allowed by instrument capability. When the measurement ranges overlap (through the use of the same geometric sequence), the individual bin sizes are identical, allowing for relatively easy comparability in these common regions. When comparing distributions from different instruments, only data from these common histogram bins should be used. Data from bins outside of these common areas should be discarded and the distribution renormalized to 100%.

Other instrument parameters

For inter-laboratory consistency, it is recommended that the particle-shape setting should be set to "irregular". The auto-align function should also be turned on so that the laser is realigned before each set of runs.

C.7.3 Repeatability

ISO 13320-1 states that for samples with a PSD coefficient-of-variation[1] less than about 50% in the mid-range of the instrument's capabilities, five measurements should be taken. The coefficient of variation of the median values (D_{50}) should be less than 3%. The coefficient of variation of values on the sides of the distribution (D_{10} and D_{90}) should be less than 5%. Below 10 μm, ISO 13320-1 recommends that these values be doubled.

For some waste samples, particles smaller than 10 μm are expected. In addition, particles in this range are near the small end of the diameter range of instrument capabilities. For these reasons, five measurements each on different samples from the same batch are recommended. The coefficient of variation of the median values (D_{50}) and those at the sides of the distribution (D_{10} and D_{90}) should be calculated. The target value for the coefficient of variation of the median values (D_{50}) is less than 10%. The target value for the coefficient of variation of values on the sides of the distribution (D_{10} and D_{90}) is less than 15%. Regardless of whether the target values are met, the actual values measured should be reported in the final report and the end-user of the data should evaluate its acceptability.

Because new particles are constantly being passed through the detector cell in these instruments, a "batch" of sample material should be considered as the material loaded into the instrument to achieve correct sample loading. This also limits the quantity of radioactive material required for analysis to a reasonable level for work in a fume hood. Consequently, five sets of measurements should be performed on this single batch. The average distribution of these five runs should be reported as a final result.

C.7.4 Accuracy

ISO 13320-1 states that calibration of laser-diffraction instruments is not required, but correct operation of the instrument should be confirmed through calibration validation. Calibration is validated through the use of certified or standard reference materials. Certified standards with a known distribution having a range of spherical particles over one decade in size are preferred. The real and imaginary part of the complex refractive index must be precisely specified for the material if the Mie theory is to be applied.

ISO 13320-1 recommends that the calibration be considered valid if the mean value of the median values (D_{50}) of three measurements deviates less than 3% from the certified range of values of the certified material. This range of values for the certified material is the mean value together with its standard deviation. The mean value for the sides of the distribution (D_{10} and D_{90}) should be less than 5% from the certified values together with their standard deviation.

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[1] Relative measure (%) for precision; standard deviation divided by the mean value for the population then multiplied by 100%

NIST-traceable polystyrene standards from Duke Scientific Corporation have been used within DOE projects. These standards should be considered monodisperse (i.e., the PSD of these standards consists of a single mode with an extremely small standard deviation) and a certified mean value is specified; sometimes a standard deviation is also specified. However, D_{10} and D_{90} values are not supplied. Consequently, these standards do not comply with ISO 13320-1 recommendations. However, most DOE projects require that all standards be traceable to a nationally recognized organization. Until a replacement standard material traceable to a nationally recognized organization has been identified that also conforms to the ISO 13320-1 recommendations outlined above, the Duke Scientific standards are recommended for use in DOE projects.

Because standards from Duke Scientific are monodisperse, the certified particle size distribution will only fit into a few measurement-histogram bins. Often these bins are much wider than 3% from the lower bin range to the upper bin range. This results in low accuracy in the certified particle mean value, leading to inaccuracies greater than the ISO 13320-1 recommendation of 3%. Based on previous experience in using these monodisperse Duke Scientific standards, it is recommended that the mean value of the median values (D_{50}) of three measurements should deviate less than a target value of 10% from the certified value together with its standard deviation. Regardless of whether the target values are met, the actual values measured should be reported in the final report and the end-user of the data should evaluate its acceptability.

Because new particles are constantly passed through the detector cell in these instruments, a "batch" of sample material should be considered the material loaded into the instrument to achieve correct sample loading. Consequently, three sets of measurements should be performed on this single batch. The average distribution of these three runs should be reported as a final result. Calibration validation efforts should be performed in accord with the manufacturer's recommendations, and they should be considered valid for a minimum of 30 days, or longer if so directed by the manufacturer.

C.7.5 Error Sources

If the measurement repeatability or accuracy comes into question during a measurement, the instrument operating manual should be consulted. Note that ISO 13320-1 contains a list of common measurement errors that could potentially solve such problems.

C.7.6 Data Reporting

ISO 13320-1 recommends that the following information be recorded along with the measurement data; additional information may be required depending on the applicable quality-assurance project plan:

a. Sample
   - sample identification
   - sampling procedure, i.e., sampling method and sample-splitting procedure
   - date of analysis

b. Dispersion
   - Description of dispersion liquid
   - Ultrasonication frequency (energy), duration and pause before starting measurement
   - Pump speed

c. Laser Diffraction Measurement
   - Instrument type and number
   - Software version
   - Actual size range used for the measurement
   - Date of last alignment
   - Last validation with a traceable standard to a nationally recognizable organization
     1. Vendor
     2. Material
     3. Lot
     4. Date of analysis
     5. Certified result (D_{10}, D_{50}, D_{90}) together with their standard deviations
     6. Mean of three measurements of D_{10}, D_{50}, D_{90}
     7. Percent deviation of the measured-mean results (D_{10}, D_{50}, D_{90}) and the certified results together with their standard deviation
   - Date and time of sample measurement
   - Optical concentration/obscuration
   - Type of light-scattering model applied (Mie theory for Microtrac Instruments)
   - Real and imaginary parts of complex refractive index (if Mie theory is used)
   - Other proprietary instrument settings that are significant for reproducibility of results

d. Analyst identification (retained in project file, but not included in report)
   - Name and place of laboratory
   - Operator's name

The reported measurement data should at a minimum consist of the following:

1. Volume-distribution histogram (average from five measurements)
2. Volume-distribution data in tabular form (average from five measurements)
3. Cumulative volume-distribution histogram (average from five measurements)
4. Cumulative volume-distribution data in tabular form including: D_{10}, D_{20}, D_{30}, D_{40}, D_{50}, D_{60}, D_{70}, D_{80}, D_{90}, D_{95} (average from five measurements)

5. Coefficient of variation of the median values (D_{50}) and values from the sides of the distribution (D_{10} and D_{90}) from the five measurements

Number and area distributions are calculated from the measured volume distribution by assuming spherical particles. As such, these distributions are of limited value and should only be reported if specifically requested.

C.7.7 Summary of technical recommendations

A summary of the recommended conditions discussed above is presented in Tables C.14 & C.15. For instruments with in-line ultrasonication capability, measurements should be made before and after ultrasonication. The instrument operating manual and ISO 13320-1 should be followed when questions arise regarding a particular operating condition. See Table C.16, Sheet #6 for reporting requirements. The UPA instrument can be used for PSD determination of nanometer-sized particles.

Table C.14. Recommended Microtrac X100 & Microtrac S3000 Nominal Operating Conditions

Condition	Setting
Flowrate	80 mL/sec
Ultrasonication Power	40 W (if equipped)
Ultrasonication Time	30 seconds (if equipped)
Number of Ultrasonication Periods	2 (if equipped)
Pause before starting measurement	30 seconds
Histogram Progression	Geometric Root-8
Upper Histogram Range	X100: 704.0 µm S3000: 1408.0 µm
Lower Histogram Range	X100: 0. 122 µm S3000: 0.243 µm
Run Time	60 seconds
Number of Runs	5 / average reported
Particle Transparency	“Absorb” if unknown
Particle Refractive Index	“1.54” if unknown
Particle Shape	“Irregular” if unknown
Fluid Refractive Index	1.33 if unknown

Table C.15. Recommended Microtrac UPA Nominal Operating Conditions

Condition	Setting
Histogram Progression	Geometric Root-8
Upper Histogram Range	6.541 μm
Lower Histogram Range	0.0032 μm
Run Time	10 minutes
Number of Runs	5
Particle Transparency	"Absorb" if unknown
Particle Refractive Index	1.54
Particle Shape	"Irregular" if unknown
Fluid Refractive Index	1.33

C.8 Records

Documentation generated by this guideline shall be submitted to the DOE project document control for logging, issuance, distribution, and records retention to meet project records management requirements.

C.9 References

Barnes HA and QD Nguyen. 2001. "Rotating Vane Rheometry-a Review", *Journal of Non-Newtonian Fluid Mechanics* 98 (1):1-14

Chang CY. 1996."Laboratory Procedure for the Rheological Characterization of Slurry Suspensions", PNNL-10985, Pacific Northwest National Laboratory, Richland Washington.

Dzuy NQ and DV Boger. 1985. "Direct Yield Stress Measurement with the Vane Method." *Journal of Rheology* 29(3) 335-347.

Health WO "Procedure for Measuring Sludge Shear Strength Using Shear Vanes."1986. WHO-86-1, Pacific Northwest Laboratory, Richland, Washington.

International Standard; ISO 13320-1; "Particle size analysis – Laser diffraction methods – Part 1: General Principles"

Jillavenkatesa A., S.J. Dapkunas, L.H. Lum; 2001; Particle Size Characterization; NIST recommended practice guide; Special Publication 960-1

Shaver RL. 1993. "PNL 325 Laboratories Single-Shell Tank-Waste Characterization, Tank B-201 Cores 26 and 27 Data Package". WHC-SD-WM-DP-037 and Addendum 1A Rev. 0, Westinghouse Hanford Company, Richland, WA.

US EPA. 1995 rev. 2. "pH Electrometric Measurement", Method 9040B in *Test Methods for Evaluating Solid Waste*. EPA SW-846, Environmental Protection Agency, Washington, D.C.

Table C.16. Example Global Data Summary Electronic File
Excel File – Sheet #1 – Waste Composition

Sample Identification:

Sample History (include: washing, leaching, chemical precipitation, mechanical agitation of any kind (time and intensity)):

Sodium concentration of LAW Waste or Pretreated Waste: _____ Molar
Oxides Loading of HLW Sludge or Pretreated Sludge: _____ Total grams oxide/Liter
pH of the Waste: _____ pH

Analyte:	mg/L (LAW) or mg/kg (HLW)
Cations mg/L (LAW) or mg/kg (HLW)	
Ag	
Al	
As	
Cations mg/L (LAW) or mg/kg (HLW)	
B	
Ba	
Be	
Bi	
Ca	
Cd	
Ce	
Co	
Cr	
Cs	
Cu	
Dy	
Eu	
Fe	
Hg	
K	
La	
Li	
Mg	
Mn	
Mo	
Na	

Table C.16. (contd)
Excel File – Sheet #1 – Waste Composition

Analyte:	mg/L (LAW) or mg/kg (HLW)
Cations mg/L (LAW) or mg/kg (HLW)	
Nd	
Ni	
P	
Pb	
Pd	
Pr	
Pt	
Rb	
Rh	
Ru	
S	
Sb	
Se	
Si	
Sn	
Sr	
Ta	
Te	
Th	
Ti	
Tl	
U	
V	
W	
Y	
Zn	
Zr	
Carbon Analyses mg/L (LAW) or mg/kg (HLW)	
TIC	
TOC	

Table C.16. (contd)
Excel File – Sheet #1 – Waste Composition

Anions mg/L (LAW) or mg/kg (HLW)
F
Cl
Br
NO_2
NO_3
PO_4
SO_4
CN
NH_3
Free OH
Total OH
Radioisotopes mCi/L (LAW) or mCi/kg (HLW)
H-3
C-14
Cr-51
Fe-59
Ni-59
Co-60
Ni-63
Se-79
Y-88
Sr-90
Sr-90/Y-90
Nb-94/95
Tc-99
Ru-103
Ru-106
Sn-113
Sb-125
Sn-126
Sb\Sn-126
I-127
I-129

Table C.16. (contd)
Excel File – Sheet #1 – Waste Composition

Radioisotopes mCi/L (LAW) or mCi/kg (HLW)
C-133
Cs-134
Cs-135
Cs-137
Ce-144
Sm-151
Eu-152
Eu-154
Eu-155
Pa-231
U-233
U-234
U-235
U-236
U-238
Np-237
Pu-236
Pu-238
Pu-239
Pu-240
Pu-239/240
Pu-241
Pu-242
Pu-241/Am-241
Am-241
Am-241, Am-243
Am-242
Am-243
Cm-242
Cm-243
Cm-244
Cm-243/244

Table C.16. (contd)
Excel File – Sheet #1 – Waste Composition

Radioisotopes mCi/L (LAW) or mCi/kg (HLW)
Sum of alpha (TRU) = Σ (Pu-238, Pu-239, Pu-240, Am-241)
Total alpha
Total beta
Total gamma
Organic Analytes mg/L (LAW) or mg/kg (HLW)
Oxalate
Citrate
Formate
Gluconate
Glycolate
EDTA (ethylenediaminetetraacetic acid)
HEDTA (N-(2-hydroxyethyl)ethylenediaminetriacetic acid)
D2EHPA (bis-(2-ethylhexyl)phosphate
NTA (nitrilotriacetic acid)
IDA (iminodiacetic acid)
Succinic Acid
ED3A (ethylenediaminetriacetic acid)
Analytes Obtained on an Opportunistic Basis

Table C.16. (contd)
Excel File – Sheet #2 – LAW or HLW Melter Feed Preparation Description

Pretreated Waste or HLW Pretreated Sludge Identification:

Pretreatment History (include: washing, leaching, chemical precipitation, mechanical agitation of any kind (time and intensity)):

Sodium Concentration of LAW Pretreated Waste — Na Molarity:

Oxides Loading of HLW Pretreated Sludge — Total Grams Oxide per Liter:

Source Chemical	Manufacturer	Oxide	Target Mass (g)	Actual Mass Added (g)
Kyanite	Kyanite Mining Corp	Al_2O_3		
Alumina A-2	Alcoa Alumina	Al_2O_3		
Boric Acid Technical	U.S. Borax	B_2O_3		
10M Borax	U.S. Borax	Na_2O/B_2O_3		
Soda Ash	Solvay Minerals	Na_2CO_3		
Wollastonite	NYCO	CaO		
Fe_2O_3 5001	Prince Mfg. Co.	Fe_2O_3		
Li_2CO_3	Chemettal-Foote	Li_2O		
Olivine	Unimin Corp	MgO		
SCS-75	U.S. Silica	SiO_2		
Rutile (Air floated)	Chemalloy Co.	TiO_2		
Kadox	Zinc Corp Amer.	ZnO		.
Zircon	Amer. Miner. Inc.	ZrO_2		
Sucrose	Amalgamated Sugar Co.	Sugar		

Table C.16. (contd)
Excel File – Sheet #2 – LAW or HLW Melter Feed Preparation Description

Mixing Operation Data Needed to Compare Mixing of the Melter Feed	
Melter Feed ID:	
Processing Scale (lab/bench, pilot, or full):	
Activity/Property	Data or Explanation
Order of Chemical Additions	
Mixing Time	
Impeller Speed	
Impeller Diameter	
Tank Diameter	
Number of Baffles	
Size of Baffles	
Depth of Impeller	
Comments:	

Table C.16. (contd)
Excel File – Sheet #3 – Physical Property Data

Physical Property Data Summary for Sample	
Physical Property	Values (units)
Sodium concentration of LAW waste or pretreated waste (Molar)	
Oxides loading of HLW sludge or pretreated sludge (total grams oxide/Liter)	
pH (aging 1 day, 1 week, 1 mo)	
Solid phases present	
Density – Bulk slurry (g/mL) (aging 1 day, 1 week, 1 mo)	
Density – settled solids (g/mL)	
Density – centrifuged solids (g/mL)	
Density - supernatant liquid (g/mL)	
Vol. % settled solids after 72 hours (aging 1 day, 1 week, 1 mo)	
Vol. % centrifuged solids	
Wt % total dried solids	
Wt % centrifuged solids	
Wt % oven dried solids	
Wt % undissolved solids	
Wt % dissolved solids	

Table C.16. (contd)
Excel File – Sheet #4 – Rheological Property Data

Flow Curve Summary Report				
	A	B	C	D
1	Date			
2	Sample ID:			
3	File Name			
4	Operator			
5	Sensor			
6	Measurement System			
7	Temperature (°C)			
8	M Factor			
9	A Factor			
10	Shear Rate (1/s)	Shear Stress (Pa)	Viscosity (cP)	Time (min)
11				
12				
13				
14				
15				
16				
17	(use as many lines as needed)			

Note: Data is reported in the cells with a heavy outline.

Table C.16. (contd)
Excel File – Sheet #5 – Rheological Property Model Parameter Data

Model/Model Parameter	Parameter Value
Shear Strength (by Vane Method)	
τ_O - Shear Strength (Pa)	
Ostwald (or Power Law)	
m – the consistency coefficient (cP)	
n – the power law exponent	
R^2 – correlation coefficient	
Bingham Plastic	
τ_O^B - the Bingham yield stress (Pa)	
η_p – the plastic viscosity (cP)	
R^2 – linear correlation coefficient	
Herschel-Bulkley	
τ_O^H - the yield stress (cP)	
k - the Herschel-Bulkely consistency coefficient (cP)	
b - the Herschel-Bulkely power law exponent	
R^2 – correlation coefficient	

Table C.16. (contd)
Excel File – Sheet #6 – Particle Size Distribution Data

	A	B	C	D
1	D_{10}		D_{10} coefficient of variation	
2	$D2_0$			
3	D_{30}			
4	D_{40}			
5	D_{50}		D_{50} coefficient of variation	
6	D_{60}			
7	D_{70}			
8	D_{80}			
9	D_{90}		D_{90} coefficient of variation	
10	D_{95}			Time (min)
11	Upper Histogram Bin Particle Size (micrometers)	Volume Distribution Histogram Data	Cumulative Volume Distribution Histogram Data	
12				
13				
14				
15				
16				
17	(use as many lines as needed)			

Appendix D

Guideline for Simulant Development, Approval, Validation, and Documentation

Appendix D

Guideline for Simulant Development, Approval, Validation, and Documentation

D.1 Objective

This document defines the process for simulant development, approval, validation, and documentation for research and technology needs within the Department of Energy (DOE) complex.

D.2 Scope

This guideline provides the instructions for research and technology (R&T) project personnel to develop, approve, validate, and document simulants for use within their projects. This guideline applies to new and revised simulant-development activities and their associated project documents such as test specifications, test plans, test exceptions, simulant-preparation procedures, and test reports. Following the direction contained in this document will facilitate a consistent methodology for development, preparation, and validation of simulants for use within DOE-complex projects.

This guideline directs how R&T project personnel will:

1. Define simulant use;
2. Define simulant composition or range of compositions;
3. Define simulant-design requirements;
4. Review and approve simulant-development activities;
5. Verify and validate simulant meets design requirements;
6. Verify that simulant is consistent with flowsheet predictions; and
7. Document simulant-development activities and preparation procedures.

The process for simulant development, approval, validation, and documentation that will be followed is shown schematically in Figure D.1.

D.3 Responsibilities and Definitions

Responsibilities:

R&T Cognizant Engineer is responsible for:

- Producing a Test Specification or similar document that includes a simulant scope statement defining simulant use and necessary quality assurance/quality control (QA/QC) level for development, simulant target composition(s), and simulant design requirements

- Working with the Simulant Coordination Team members representing the appropriate project functions, e.g., characterization, pretreatment, vitrification, and waste-form qualification, to define the best available waste-stream source-term data
- Working with the process-engineering organization to define the best available analytical composition data for potential flowsheet process and recycle streams
- Working with appropriate project staff, e.g., R&T, process operations, process engineering, production engineering, etc., to define simulant compositions formulated for specific unit operations or bounding simulants that may not mimic a specific waste stream
- Developing simulant-design requirements with technically cognizant personnel (process operations, process engineering, production engineering, etc.) within all affected unit operations, equipment, etc
- Insuring that each technical discipline affected by the simulant work reviews all pertinent paperwork, such as test specification(s), test plan(s), test exception(s), test report(s), etc.

The R&T Simulant Coordinator is responsible for (Note: for larger projects, a single point of contact, a Simulant Coordinator, is recommended):

- Insuring that the development and use of simulants is coordinated, consistent, and defensible across the project
- Being the principal point of contact for all simulant questions (both internal and external)
- Insuring that ongoing radioactive-waste testing provides valid characterization data for simulant definition
- Insuring that any contract requirements for comparing results of tests on actual waste with results of tests on simulant are met
- Reviewing all new work with simulants and any actual-waste testing that collects data that may be used to define simulant characteristics
- Facilitating resolution, along with Simulant Coordination Team Members, of all issues that involve the "performance" of a simulant.

Simulant Coordination Team Members are responsible for:

- Review of simulant documents within their own technical areas (characterization, pretreatment, vitrification, waste form qualification, commissioning, etc.)
- Facilitating integration within their technical areas, with other project technical areas, and with other project departments (process operations, process engineering, mechanical systems, commissioning, etc.)
- Acting as points of contact (POC) for simulant questions within their technical areas.
- R&T actual-waste-characterization Group Team Member will provide the latest waste-stream characterization source-term data.

Appropriate project managers are responsible for:

- Review and approval of research and testing to insure that contractual and technical requirements are met.
- Release of all Administrative Hold points, e.g., release of simulant-preparation procedure and release of simulant for use in testing.

Assigned simulant-development personnel or subcontractor (if used) are responsible for:

- Preparation of test plans, etc. to implement all approved project test specifications or other guiding documents
- Developing standalone simulant-preparation procedure(s) following all simulant design criteria
- Verifying and validating that produced simulant(s) meet all final design and testing requirements
- Documenting all simulant development, verification and validation, and approval in a final test report.

Definitions:

Validate Simulant – confirm that process behavior adequately mimics actual waste.

Verify Simulant – confirm that specified properties are met.

D.4 Simulant Development, Approval, Validation, and Documentation

D.4.1 Background

This guideline provides consistent methodology for development, preparation, validation, approval, and documentation of simulants for use within DOE projects. This methodology is applicable to all project staff and subcontractors whose work involves simulants; use of this methodology will establish a consistent approach to all aspects of simulant work. Methodology for validation of simulants used on DOE projects, an important step, is described. Standardized chemical-, physical-, and rheological-measurement methods are necessary for work performed by DOE subcontractors so consensus property-measurement guidelines were developed. Use of these property measurements is essential to ensure standardized, comparable results from all actual and simulant-based tests conducted across DOE projects. These property measurements are contained in Appendix C, Guidelines for Performing Measurements of Chemical, Physical, and Rheological Properties. In addition, all work with simulants should be compared to results from similar testing with actual waste; following the simulant-documentation criteria contained herein meets this requirement.

D.4.2 Simulant-Development Scope Statement

All simulant development and testing activities should be identified in project documentation to facilitate coordination. All new scope should be reviewed in accordance with the appropriate project procedures, etc. and approved.

D.4.2.1 Simulant Use Definition

The R&T cognizant engineer will determine what the simulant is to be used for, e.g., only for precipitation, for filtration, ion exchange, evaporation, or all pretreatment unit operations. Once the simulant use is defined, the usage will dictate whether a single unit operation or multiple unit operations will be affected and what potential recycle streams may be important. Through simulant-use definition, the characteristics of the simulant (chemical, physical, or rheological or a combination thereof) required for development will be determined. This effort should be coordinated with process engineering and operations project personnel.

The R&T cognizant engineer will also determine which quality assurance requirements are to be met in accordance with the projects quality assurance program, e.g., a QA program that implements Nuclear Quality Assurance, NQA-1 (1989) and applicable portions of NQA-2a, Part 2.7 (1990) or QARD DOE Order 0333P (2002) QA requirements, or simulant-development activities that support regulatory and environmental compliance-related aspects of a waste-vitrification program may need to be performed in accordance with project quality-assurance requirements for generating environmental regulatory data. [Note: use of simulants for project testing that is only scoping in nature may not need to comply with specific QA requirements, e.g., NQA-1 or QARD requirements, but this classification will need to be discussed, documented, and project concurrence gained during preparation and review of the test specification.]

D.4.2.2 Simulant-Composition Definition

Approaches to simulant-composition development will vary depending on the type of simulant required for testing. Simulant compositions may be based on actual radioactive-waste sample-characterization data, or may be formulated for specific unit operations, or for bounding or testing the limits of a process or specific piece of equipment.

Compositions for simulants based upon actual waste samples will be defined using project characterization data as the starting point (see Figure D.2). The Simulant Coordination Team member representing the project's Characterization group will supply the best available tank source-term analytical data along with a comparison against comparable inventory data, historical process information, or feed vectors developed by the project. This comparison will highlight analytical outlier values that will need to be addressed for an analyte. The best available actual-waste source-term analytical data will reference the project analytical characterization data used and will be transmitted via written documentation to the R&T cognizant engineer developing the simulant.

For simulant compositions that will mimic project flowsheet streams later in the process (after the best available waste source-term analytical information on the incoming waste stream is provided), the cognizant engineer may need to schedule and use process flowsheet runs to assure that the correct additional stream compositions (recycle streams from other flowsheet unit operations, e.g., filtration, washing) are addressed and incorporated. The compositional waste-stream source-term data used as inputs to the model will be the ones provided by the Characterization member of the Simulants Coordination Team. Any other planned operations that could affect flowsheet compositions being simulated (e.g., adjustment of actual-waste-composition data to reflect future waste-feed delivery activities to arrive at the 'best forecast composition' need to be documented in the Test Specification. The Characterization member of the Simulant Coordination Team will facilitate obtaining and

incorporating the future waste-stream operations information. The assumptions and inputs to the process flowsheet used will need to be described and discussed (e.g., offgas components [SO_4, Cl^-, F^-] partitioning, wash/leach factors), and will need to be checked against project reports and documented in the Test Specification. The feed vectors, flowsheet scenarios, run requests, and the like should be developed with the assistance of the project's Process Operations group. By this process, the best forecast simulant composition would be traceable to actual project waste-characterization data. (Note: the project should compare analytical data from actual waste to waste-stream-modeling results to validate modeling results. If errors in modeling are observed the models should be adjusted appropriately).

For simulant compositions formulated for specific unit operations, the composition may be targeted to only the chemical, physical, and rheological properties that are known to affect specific key operating/processing parameters.

For a simulant intended to bound the limits of a process or specific piece of equipment, a range of compositions should be developed to define these operational limits, e.g., purely physical simulants to determine the rheological bounds between which a specific vessel is able to meet a required process condition. For this approach, multiple simulants may be required to test numerous parameters. A bounding simulant may consist of an existing simulant spiked with specific compounds to test process performance (e.g., added organics to test destruction across the entire melter system) or a purely physical simulant to test the acceptable physical and rheological process limits of a system.

D.4.3 Simulant Design Requirements

The cognizant engineer will determine the "necessary & sufficient" simulant properties to measure for each affected unit operation, waste or recycle stream; these should be the same for both actual and simulant waste where the simulant is based upon actual-waste characterization data. D.7 of this Appendix provides an example of chemical, physical, and rheological properties measurement matrices for several Waste Treatment and Immobilization Plant (WTP) waste streams that would be considered in developing simulant-design requirements for this project. (Note: It is recommended that a similar chemical, physical, and rheological property-measurement matrix be developed for your specific project). The cognizant engineer will consult with engineers and scientists (both within the project and appropriate subcontractor staff) who are technically knowledgeable with all affected unit operations to determine how close each measured property must be to the target (e.g., in the example given, a Sr/TRU precipitation and filtration simulant, carbonate, will have to be within 10% of simulant target-composition value) for the important analytes, physical properties, etc. These will then become the acceptance criteria for the simulant eventually prepared, to verify the simulant-preparation procedure.

Each project technical area (e.g., waste characterization, pretreatment, vitrification technology & waste-form qualification) should complete a review comparing actual and simulant waste samples and streams within their cognizant area for each of their relevant unit operations (e.g., from Section D.7 from this appendix example, for HLW vitrification, the focus would be on the blended HLW feed, i.e., HLW pretreated sludge, Sr/TRU precipitate & entrained solids, and LAW Cs & Tc ion-exchange eluants received from pretreatment into the concentrate receipt vessel; the HLW melter feed in the Melter-Feed Preparation Vessel and the Melter Feed Vessel; and the Submerged Bed Scrubber (SBS) solutions. These simulant-to-actual-waste comparison reviews should include, at a minimum:

- baseline flowsheet testing results and any additional testing around the baseline flowsheet processes;

- a data review to facilitate the development of a list of properties necessary-and-sufficient to measure on both simulant and actual waste and the properties needed to accurately produce simulant wastes;
- a determination, using both actual and simulant waste data, of a bounding envelope for each of the necessary-and-sufficient properties; and
- a comparison of simulant testing results with actual-waste test results.

Known actual-waste baseline flowsheet process information that is associated with the major unit operations will allow for a process check of eventual "commissioning simulants" as they move through the entire project flowsheet either in an integrated pilot plant or the actual plant.

Whether or not specific project technical-area reviews are completed and available to assist cognizant engineers in developing simulant design requirements, the following key properties will need to be discussed and documented in the implementing document, e.g., a test specification:

- Key processing properties. These will consist of the properties that are measured during testing of a piece of equipment or unit operation. Examples include filtrate flux rate, decontamination factors, fouling, scaling, pressure drop, sample homogeneity, etc. (Note: The cognizant engineer will need to take into consideration plant process upset conditions in testing requirements).
- Key chemical properties. The chemical properties of the simulant necessary to ensure a valid simulant are prepared.
- Key physical properties. The key physical properties of the simulant will be listed. Examples include density, heat capacity, thermal conductivity, heat of vaporization, particle-size distribution, settling rate, wt% settled and centrifuged solids, vol% settled and centrifuged solids, wt% total dried solids, wt% total oxide, etc.
- Key rheological properties. The key rheological properties of the simulant will be listed. May include yield stress (vane) and shear-stress-versus-shear-rate viscosity measurements.
- Design-basis range. Key design assumptions employed at the particular point in the plant will also be listed. For example, key design parameters for pumps, agitators, piping, vessels, etc. that would affect the simulant development need to be documented.
- If simulant melter feeds are to be developed, the cognizant engineer will ensure that the glass-former chemicals (GFCs), used for testing, are project-approved.

The key simulant properties and acceptance criteria will be documented in the implementing document, e.g., a test specification, preferably in table format. An example for a WTP LAW Melter Feed is provided in Section D.8 of this Appendix. (Note: the cognizant engineer should develop the requirements for use, composition, and design of the simulant in conjunction with project process-operations staff and other appropriate project personnel to minimize review comments and the review cycle. In addition, the cognizant engineer should also remind process-operations staff to confirm that the work scope is consistent with the plant flowsheet).

Standardized chemical, physical, and rheological property measurements for work performed should be used (see Appendix C, Guidelines for Performing Measurements of Chemical, Physical, and Rheological Properties). Use of these property measurements is essential to ensure standardized, comparable results between all actual-waste and simulant-based tests conducted across the DOE project.

D.4.4 Review and Approval of Simulant Use, Composition, and Design Requirements

The information developed from Section D.4.2, Simulant Development Scope Statement, and D.4.3, Simulant Design Requirements, will be included in the project implementing document, e.g., a test specification. A team for review of this document will consist of individuals who are technically cognizant within all affected unit operations. The review should include appropriate subcontractor personnel as necessary.

The person assigned to perform of the work shall prepare a draft test plan that implements the approved test specification. The test plan will indicate what methodologies will be employed to verify and validate simulant-property data produced during preparation and testing activities. The test plan will indicate that a separate standalone Simulant Preparation Procedure will be included in or with the final test report. The draft test plan will be reviewed for compliance with the test specification and by each technical discipline affected by the simulant work; review comments will be resolved by the performer of the work, and the final test plan is to be approved by the appropriate project management.

D.4.5 Develop Simulant Preparation Procedure

Once the test plan has been approved, the performer of the work will execute the simulant-development activities in accordance with the test plan to produce a standalone Simulant Preparation Procedure. The performer of the work will make sure all simulant design criteria are followed to develop the simulant-preparation procedure, for example:

- Specified ionic forms of waste components to be used.
- Charge balancing to be completed appropriately.
- Appropriate substitutes to be used for radioactive species, as required.
- Matching of pertinent physical properties of solids (e.g., phase, morphology, size, and crystalline vs. non-crystalline).
- Sequence of addition of chemical compounds, water, etc. to avoid unwanted chemical reactions.
- Actual processing parameters of the simulant important in developing a final simulant (e.g., washing, leaching, shearing of HLW solids or generation and sampling of a submerged-bed-scrubber simulant) are stipulated.

The cognizant engineer may want to add an Administrative Hold point to the test specification for "Review and Approval of Simulant Preparation Procedure" so that technically cognizant personnel of all affected unit operations can review and approve the simulant-preparation procedure, for risk-reduction purposes, before testing begins. Documentation may be via a letter, e-mail message, etc.

D.4.6 Verify Simulant Meets Design Requirements

The performer of the work will document that the simulant has been verified. This may be accomplished by a written statement to the cognizant engineer and appropriate project management. The correspondence should request release of the simulant for use in testing as necessary.

The simulant-verification activities will include and document:

- Simulant generated using an approved simulant-preparation procedure
- Simulant necessary-and-sufficient properties were measured and compared to acceptance criteria
- All necessary-and-sufficient properties are within acceptance criteria specifications. (Note: If in the initial testing of the simulant, not all of the necessary-and-sufficient properties are within the acceptance criteria specified in the Test Specification, the performer of the work will work iteratively with cognizant project personnel to choose a path forward, which may include a change of acceptance criteria). All changes to testing are to be documented and controlled by modified test specification, modified test plan, or test exceptions consistent with project procedures.

D.4.6.1 Simulant Chemical Composition Verification

Determination and reporting of the chemical composition of the simulant will rely on both the mass-balance and sample analyses together as a cross check. A batching process/sheet should be written that specifies the following:

- The technical purity or grade of the beginning chemical constituents. This will require copies of each chemical's purity certifications and may require a confirmation of adsorbed water or waters of hydration;
- The batching sequence and how and when to combine various sub-batches as necessary (Note: for typical contaminants such as chloride, these ingredients should be added after the amount already present from the other chemicals added is known);
- In-process sampling and analyses at key simulant-preparation points, as necessary (e.g., analyze a nitrate solution before neutralizing and precipitating solids, or after a precipitation and washing sequence to verify the target values have been reached);
- Review of completed batching sheet(s) by an independent, qualified individual; and
- Results of the simulant analyses to verify the final batch composition for acceptance. The vendor or performer of the work will supply the confirmatory analysis results to the project in verification documentation.

Following preparation of the simulant, a simple confirmatory quantitative analysis is performed on the simulant to verify that all components and their amounts were added correctly. This analysis is a final independent validation of the simulant composition. If the analysis indicates that the amount of an analyte component differs from its target amount by significantly more than the analytical uncertainty for that component, there is reason for concern that an error has occurred with the simulant preparation. Using both the mass balance (i.e., batching sheets, chemical addition and weighing confirmation, and calculation verification) and actual chemical composition analysis will increase the probability of producing a simulant with an accurately known chemical composition. This will allow for informed decision making on whether to rely on the calculated or measured analyte value or to re-analyze. For example, an adjustment would not have to be made to a simulant-batch composition based upon one analyte analytical result if the mass-balance composition and batching sheets corroborated the majority of the analysis. Disagreement between the measured analytical results and the mass balance or batching

sheets due to errors in simulant preparation, however, could lead to a re-analysis and possible re-batching of the simulant. Potential errors in simulant preparation may include:

- incorrect chemical quantities or incorrect chemicals being added;
- use of chemicals with poor quality or high levels of impurities;
- use of chemicals with elevated levels of waters-of-hydration from excessive storage; or
- use of starting chemicals that were not reported.

The prepared simulant composition will be certified to the previously agreed-upon set of analyte values. Typically, a graded range of analyte composition values is used for simulant preparation work; the graded range will be provided to the performer of the work in the test specification before simulant-preparation work begins. An example of a graded range of analyte composition values for preparation of a melter-feed simulant may be ± 5 wt% for major constituents (analytes greater than 0.5 wt% on an element basis) and ± 20 wt% for minor constituents (analytes less than 0.5 wt% on an element basis) known to not have an effect on the melter testing parameters to be studied.

The chemical composition of the final simulant will be reported through documentation of the steps and decisions performed to confirm the target composition of the simulant as described above.

If testing was completed under a simulant development-only test specification, then the performer of the work will proceed to documentation in a test report (see following section). If testing was completed under a combined simulant-development and simulant-testing test specification, the performer of the work will request project approval of simulant verification and permission to use the simulant in further testing.

The cognizant engineer should add an Administrative Hold point to the test specification for "Release of Simulant for Use in Testing" so that technically cognizant personnel of all affected unit operations can review and approve the simulant for risk-reduction purposes before testing begins. Documentation may be via a letter, e-mail message, etc.

D.4.7 Simulant Development, Validation, and Preparation Activities Documentation

Upon completion of testing, the performer of the work shall prepare and submit to the project for review and comment a draft test report consistent with project requirements. (Note: Radioactive sample properties should be used to validate simulants and the results from simulant testing. Process scale-up should be demonstrated with actual waste and appropriate simulants. This is consistent with the definition for "Validate Simulant" provided in Section D.3 and should be the basis for simulant-validation activities within the project).

The draft test report shall address the following simulant-development activities in addition to any other testing performed using the approved simulant:

- Simulant Designation

- Simulant waste-stream composition / Unit operation usage / Requirements
  - Characterization data determination
  - Flowsheet operations for which simulant was developed
  - Simulant design requirements and acceptance/success criteria
- Actual step-wise simulant preparation procedure included as an appendix specifying:
  - Chemicals used (for consistency)
  - Chemical addition order
  - Precautions
  - All other important considerations necessary for correct preparation by independent users, such as, precipitation, filtration, temperature control, scaling issues, and simulant shelf-life.
- Key characteristics, limitations, etc. of simulant
- Which verification-and-validation testing procedure was followed and its results, e.g.,:
  - Chemical composition
  - Specified ionic forms of waste components used
  - Charge-balancing completed appropriately
  - Appropriate substitutes used for radioactive species, as required
  - Matching of pertinent physical properties of solids, e.g., phase, morphology, size, and crystalline vs. non-crystalline, etc.,
  - Necessary-and-sufficient properties measured and acceptance criteria met
  - Baseline flowsheet design-basis criteria met
  - A=Any other acceptance criteria met
  - All other important considerations required for validation
- For all testing completed using simulants, compare the results to any similar testing with actual waste
  - Summarize the tests performed, the data collected and compare to expected plant conditions, as applicable
- Provide all agreed-upon raw data in the report appendices
- Include reference for development organization, contact name, etc. in test report

The draft test report will be reviewed for compliance with the test specification and test plan by technically cognizant project staff, by each technical discipline affected by the simulant work, and the same reviewers that reviewed the test plan. The review comments will be resolved by the performer of the work and the final test report is to be approved by the appropriate project management.

Note: Simulant development, approval, validation, and documentation activities (described in Sections D.4.2 through D.4.7) have been summarized as a check list in Section D.9 of this appendix to

allow the cognizant engineer and reviewers a means to determine whether all appropriate areas have been addressed in the associated project documentation.

D.5 Records

Documentation generated by this Guideline, e.g., Test Specifications, Test Plans, Test Exceptions, and Test Reports, shall be submitted to the project's document control program for logging, issuance, distribution, and records retention to meet project records-management requirements.

D.6 References

Appendix B, Sampling of Radioactive Wastes

Appendix C, Guidelines for Performing Measurements of Chemical, Physical, and Rheological Properties

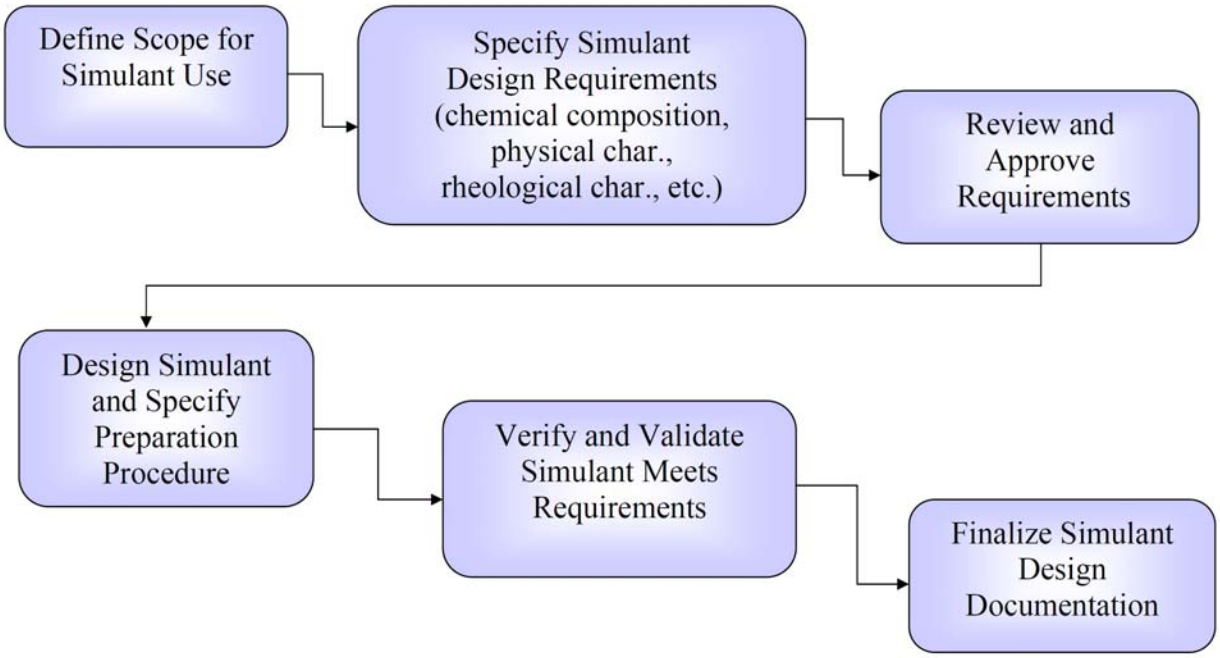

Figure D.1. Simulant Development, Approval, Validation, and Documentation Flowsheet

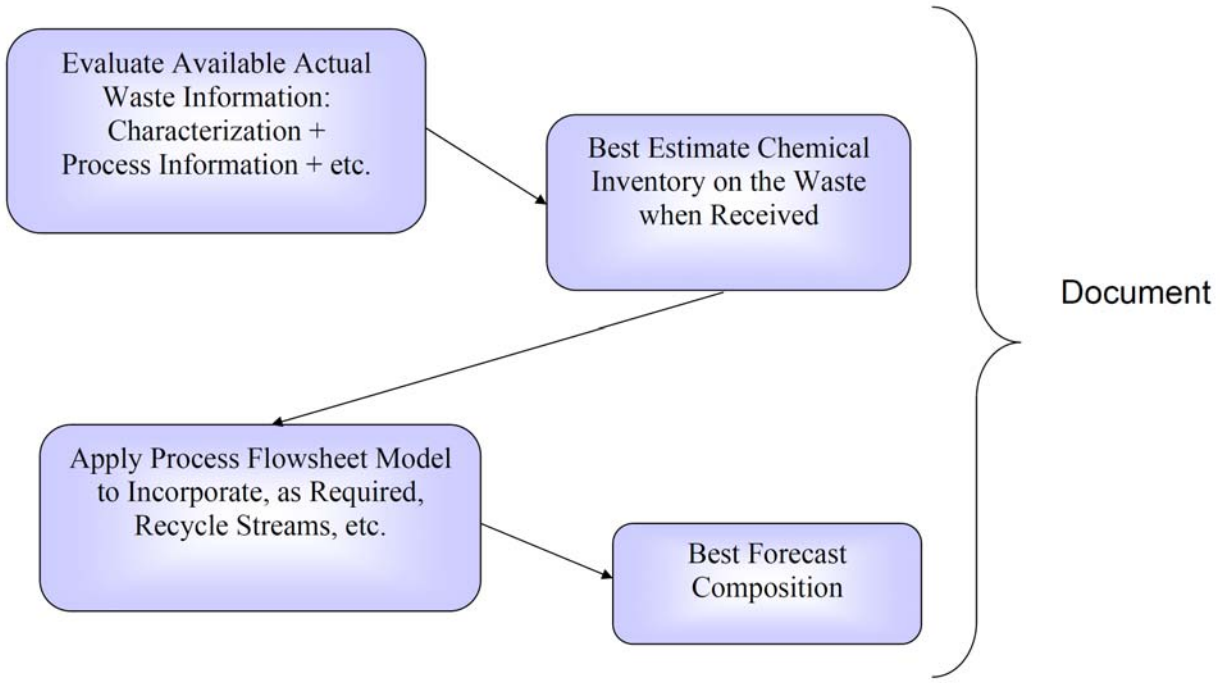

Figure D.2. Flowsheet for Simulant Composition Determinations Based Upon Actual Waste Sample Characterization Data

D.7 Appendix 1: Waste Streams Chemical, Physical, and Rheological Necessary and Sufficient Properties Measurement Matrix

Property	Tank Waste	Sr/TRU Precipitate	Ultrafiltration Feed IX	Feed	Cs &Tc IX Effluents	Cs & Tc IX Eluants
Chemical Composition	X	X	X	X	X	X
pH X		X	X	X	X	X
PSD	X	X	X			
Particle (size & shape)	X	X	X			
Heat Capacity	X					X
Thermal Conductivity						X
Bulk Density	X	X	X			X
Supernatant Liquid Density	X X		X	X	X	X
Vol % Settled Solids	X	X	X			
Settling Rate		X	X			
Centrifuged Solids Density	X	X				
Vol % Centrifuged Solids	X X					
Wt % Centrifuged Solids	X	X				
Wt % Oven Dried Solids	X X					
Wt % Total Dried Solids	X	X				
Wt % Undissolved Solids	X X		X		X	X
Shear Stress Versus Shear Rate ambient and 40°C	X	X	X		X	X
Yield Strength	X	X				
Wt % total oxide	X	X				X

Property	Treated LAW Evaporate	LAW Pretreated Waste	HLW Pretreated Waste	LAW Melter Feed	HLW Melter Feed
Chemical Composition	X	X	X	X	X
pH	X X		X	X X	
PSD			X	X	X
Particle (size & shape)					
Heat Capacity	X				
Thermal Conductivity	X				
Bulk Density	X		X	X	X
Supernatant Liquid Density	X X		X	X X	
Vol % Settled Solids		X	X	X	X
Settling Rate	X		X	X	X
Centrifuged Solids Density		X	X	X	X
Vol % Centrifuged Solids	X		X	X	X
Wt % Centrifuged Solids	X	X	X	X	X
Wt % Oven Dried Solids	X				
Wt % Total Dried Solids	X	X	X	X	X
Wt % Undissolved Solids	X X		X	X X	
Shear Stress Versus Shear Rate ambient and 40°C	X	X	X	X	X
Yield Strength			X	X	X
Wt % total oxide	X	X	X	X	X

D.8 Appendix 2: Example: Property-Acceptance Criteria for Low Activity Waste Melter Feed

Example Only of Necessary and Sufficient Properties and Acceptance Criteria for Validation of LAW Melter Feeds:

Chemical composition: ± 5 wt% for major constituents (analytes >0.5 wt% on an element/compound basis) and ± 20 wt% for minor constituents (analytes <0.5 wt% on an element/compound basis) known to not have an affect on melter testing parameters to be studied.

Property Accepta	nce Criteria
pH	± 0.5 unit
Particle size distribution (D_{95}) ±	20%
Particle Density	± 0.2 g/mL
Density - Bulk slurry	± 5%
Density - centrifuged solids	± 10%
Density - supernatant	± 5%
Vol. % settled solids	± 20%
Vol. % centrifuged solids	± 10%
Wt% total dried solids	± 5%
Wt% centrifuged solids	± 5%
Wt% undissolved solids	± 5%
Wt % total oxides	± 5%
Settling Rate	± 20%
Flow Curve (maximum apparent viscosity at low shear rates (≈25s^{-1})	± 200%
Yield Stress (settled solids at 40ºC)	± 50%

D.9 Appendix 3: DOE-Project Simulant Development, Approval, Validation, and Documentation Check List

Development of Test Specification:

Scope Statement:

- ☐ Simulant use defined:
  - ☐ Single unit operation
  - ☐ Multiple unit operations
  - ☐ Recycle stream
  - ☐ Commissioning
  - ☐ Type of simulant determined: ☐ chemical ☐ physical ☐ rheological
  - ☐ QA/QC level determined: ☐ NQA-1 ☐ QARD ☐ N/A (must explain why)

- ☐ Simulant composition defined:
  - ☐ Simulant based on actual waste characterization data:
    - ☐ Characterization data recommendation from project waste characterization group
      - ☐ Compositional changes (recent and planned future) incorporated as necessary
    - ☐ Compared pertinent waste characterization data and discrepancies rectified and documented
    - ☐ Process flowsheet model(s) used to assess process & recycle stream composition affects
      - ☐ Assumptions and inputs to process flowsheet model(s) used have been reviewed and approved, e.g. HLW wash/leach factors, offgas partition coefficients, etc.
      - ☐ Internal process waste stream/recycle data assessed, reviewed and approved
    - ☐ As necessary, project approved glass former chemicals are specified
  - ☐ Simulant designed to test unit operation(s)
  - ☐ Simulant designed to bound or test the limits of the process or equipment.

Simulant Design Requirements:

- ☐ "Necessary & sufficient" properties of simulant (should be the same for both actual and simulant waste where simulant is based upon actual waste characterization data) for each affected unit operation defined
- ☐ Acceptance criteria, based upon actual waste testing data where available, for each "necessary & sufficient" property specified

Review and Approve Simulant Use, Composition, & Design Requirements:

- ☐ Technically cognizant personnel within all affected unit operations review and approve
  - ☐ Characterization Group
  - ☐ Pretreatment Group
  - ☐ Vitrification & Waste Form Qualification Group
  - ☐ Simulant Coordination
  - ☐ Process Technology Group
  - ☐ Process Engineering Group
  - ☐ Mechanical Systems
  - ☐ Commissioning Group
  - ☐ Environmental, Safety, and Health (ES&H)
  - ☐ Quality Assurance

Release of Simulant Preparation Procedure from Administrative Hold:

Develop Simulant Preparation Procedure:

- ☐ Simulant design criteria were followed to develop simulant preparation procedure, e.g.
  - ☐ Specified ionic forms of waste components were used
  - ☐ Charge balancing was completed appropriately
  - ☐ Appropriate substitute species were used for radioactive ions, as required
  - ☐ Pertinent physical properties of solids, e.g. phase, morphology, size, and crystalline vs. non-cr ystalline, etc. matched
  - ☐ Sequence of chemical compounds, water, etc. are sufficient to avoid unwanted chemical reactions
  - ☐ Actual processing of the simulant (e.g. washing/leaching/shearing of HLW solids or generation and sampling of a submerged bed scrubber simulant) important in developing a final simulant are stipulated

Potential Hold Point for Review and Approval of Simulant Preparation Procedure:

- ☐ Technically cognizant personnel within all affected unit operations review and approve
  - ☐ Characteri zation Group
  - ☐ Pretreat ment Group
  - ☐ Vitrification & Waste Form Qualification Group
  - ☐ Sim ulant Coordination
  - ☐ Process Technology Group
  - ☐ Process Engineering Group
  - ☐ Mechanical Systems
  - ☐ Co mmissioning Group
  - ☐ Environmental, Safety, and Health (ES&H)
  - ☐ Quality Assurance
  - ☐ Subcontracto r Personnel
  - ☐ Other _____
- ☐ Simulant preparation procedure and review are documented via a letter, E-mail message, etc. based

Verification and Validation of Simulant:

Verify & Validate Simulant Meets Design Requirements:

- ☐ Simulant generated using an approved simulant preparation procedure
- ☐ Simulant "necessary & sufficient" properties are measured and compared to acceptance criteria
  - ☐ All "necessary & sufficient" properties are within acceptance criteria
    - ☐ If work completed under a simulant development only test specification, proceed to docum entation
    - ☐ If work completed under a combined simulant development & simulant testing test specification, request project approval of simulant validation and permission to use in further testing
  - ☐ Not all "necessary & sufficient" properties are within acceptance criteria; iterate with cognizant project personnel to come to closure on path forward or change of acceptance criteria

    Test changes documented and controlled by:
    - ☐ Modified test specification
    - ☐ Modified test plan
    - ☐ Test Exceptions

    [Note: once revised "necessary & sufficient" properties are within acceptance criteria see above direction.]

Release of Simulant for Use in Testing:

Hold Point for Review and Approval of Simulant Use in Testing:

- ☐ Technically cognizant personnel within all affected unit operations review and approve
  - ☐ Characteri zation Group
  - ☐ Pretreat ment Group
  - ☐ Vitrification & Waste Form Qualification Group
  - ☐ Sim ulant Coordination
  - ☐ Process Technology Group
  - ☐ Process Engineering Group
  - ☐ Mechanical Systems
  - ☐ Co mmissioning Group
  - ☐ Environmental, Safety, and Health (ES&H)
  - ☐ Quality Assurance
  - ☐ Subcontracto r Personnel
  - ☐ Other _____
- ☐ Simulant use for testing review is documented via a letter, E-mail message, etc. based upon instruction provided in the original project documentation, e.g. test specification or test

Simulant Documentation:

Document Simulant Development Activities and Preparation Procedure:

- ☐ Completed simulant Preparation Procedure documentation; will include:
  - ☐ Sim ulant Designation
  - ☐ Simulant waste stream composition / Unit operation usage / Requirements
    - ☐ Characterization data determination
    - ☐ Flowsheet operations for which simulant was developed
  - ☐ Actual simulant preparation procedure:
    - ☐ chemicals to use
    - ☐ chemical addition order
    - ☐ precautions
    - ☐ all other important 'aspects' needed for correct preparation by independent users
  - ☐ Key characteristics, limitations, etc. of simulant
  - ☐ Validation procedure followed, i.e. document the steps delineated above, e.g.:
    - ☐ chem ical composition
    - ☐ Specified ionic forms of waste components were used
    - ☐ Charge balancing was completed appropriately
    - ☐ Appropriate substitute species were used for radioactive ions
    - ☐ Pertinent physical properties of solids, e.g. phase, morphology, size, and crystalline vs. non-cr ystalline, etc. matched
    - ☐ "necessary & sufficient" properties measured
    - ☐ baseline flowsheet design basis (does the simulant fall within DB?)
    - ☐ acceptance criteria met
    - ☐ all other important 'aspects' needed for validation
  - ☐ Technical/Peer review comments, issues, etc. addressed
    - ☐ all other pertinent comments

Appendix E

Slurry Rheology

Appendix E

Slurry Rheology

E.1 A Primer on Slurry Rheology

Rheology is the study of the flow and deformation of materials. When a force (stress) is placed on an object, the object deforms or strains. Many relationships have been found relating stress to strain for various fluids. The flow behavior of a fluid can generally be explained by considering a fluid placed between two plates separated by a distance x (Figure E.1). The lower plate is held stationary while a force, F, is applied to the upper plate, of area A, that results in the plate moving at velocity, v. If the plate moves a length ΔL, the strain, γ, on the fluid can be defined by Equation (E.1).

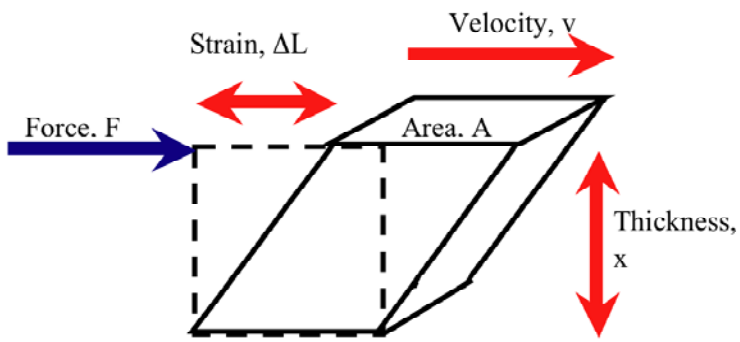

Figure E.1. Diagram of Fluid Flow Between Stationary and Moving Plates

$$\gamma = \frac{\Delta L}{\mathrm{x}} \quad \text{(E.1)}$$

The rate of change of strain (also called shear rate), $\dot{\gamma}$, can be defined by Equation (E.2). Because the shear rate is defined as the ratio of a velocity to a length, the units of the variable are the inverse of time, typically s^{-1}.

$$\dot{\gamma} = \frac{d\gamma}{dt} = \frac{d}{dt}\left(\frac{\Delta L}{\mathrm{x}}\right) = \frac{\mathrm{v}}{\mathrm{x}} \quad \text{(E.2)}$$

Typical shear rates for food-processing applications can be seen in Table E.1. Depending on the application, shear rates in the range of 10^{-6} to 10^{7} s^{-1} are possible. Human concept of a fluid is typically based on a shear rate of approximately 60 s^{-1}.

Table E.1. Typical Shear Rates in Food-Processing Applications (Steffe 1996)

Situation	Shear Rate Range (1/s) Typ	ical Applications
Sedimentation of particles in a suspending liquid	10^{-6} to 10^{-3}	Medicines, paints, spices in salad dressing
Leveling due to surface tension	10^{-2} to 10^{-1}	Frosting, Paints, printing inks
Draining under gravity	10^{-1} to 10^{1}	Vats, small food containers
Extrusion 10	^{0} to 10^{3}	Snack and pet foods, toothpaste, cereals, pasta, polymers
Calendering	10^{1} to 10^{2}	Dough sheeting
Pouring from a bottle	10^{1} to 10^{2}	Foods, cosmetics, toiletries
Chewing and swallowing	10^{1} to 10^{2}	Foods
Dip coating	10^{1} to 10^{2} Pain	ts, confectionery
Mixing and stirring	10^{1} to 10^{3}	Food processing
Pipe flow	10^{0} to 10^{3}	Food processing, blood flow
Rubbing	10^{2} to 10^{4}	Topical application of creams and lotions
Brushing 10	^{3} to 10^{4}	Brush painting, lipstick, nail polish
Spraying	10^{3} to 10^{5}	Spray drying, spray painting, fuel atomization
High-speed coating	10^{4} to 10^{6} Pape	r
Lubrication	10^{3} to 10^{7}	Bearings, gasoline engines

The shear stress applied to the fluid can be found with Equation E.3. Because the shear stress is defined as the ratio of a force to an area, the units of the variable are pressure, typically expressed in Pascals, abbreviated PA, which are Newtons /m^2.

$$\tau = \frac{F}{A} \quad \text{(E.3)}$$

The apparent viscosity of the fluid is defined as the ratio of the shear stress to shear rate (see Equation E.4). Often the shear stress and viscosity vary as a function of shear rate. Since the viscosity is defined as the ratio of shear stress to shear rate, the units of the variable are Pa·s. Typically, viscosity is reported in units of centipoise (cP); 1 cP = 1 mPa·s, or 1000 cP = 1 Pa·s. Water has a viscosity of 1 cP.

$$\eta(\dot{\gamma}) = \frac{\tau(\dot{\gamma})}{\dot{\gamma}} \quad \text{(E.4)}$$

For Newtonian fluids, the apparent viscosity is independent of shear rate (Equation E.5). Examples of the viscosity of common Newtonian materials can be seen in Table E.2.

$$\tau = \eta\dot{\gamma} \quad \text{(E.5)}$$

where τ is the shear stress, η is the Newtonian viscosity, and $\dot{\gamma}$ is the shear rate.

Table E.2. Viscosities of Several Common Newtonian Fluids (Steffe, 1996)

Material	Viscosity at 20ºC (cP)
Acetone	0.32
Water 1.	0
Ethanol	1.2
Mercury 1	.6
Ethylene Glycol	20
Corn Oil	71
Glycerin	1,500

Fluids that do not behave as Newtonian fluids are referred to as non-Newtonian fluids. Rheograms, or plots of shear stress versus shear rate, are typically used to characterize non-Newtonian fluids. Examples of typical rheograms can be seen in Figure E.2.

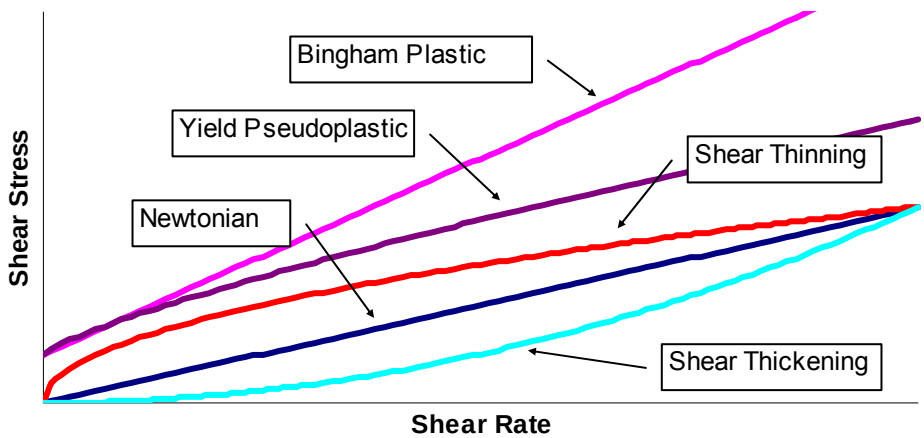

Figure E.2. Rheograms of Various Fluid Types

Shear-thinning and shear-thickening fluids can be modeled by the Ostwald equation (Equation E.6). If n<1, then the material is referred to as pseudoplastic (shear thinning). If n>1, then the material is referred to as dilatant (shear thickening). These fluids exhibit decreasing or increasing apparent viscosities as the shear rate increases, depending on whether the fluid is shear thinning or shear thickening, respectively. Since shear-thickening flow behavior is rare, shear-thickening behavior is often an indication of possible secondary flow patterns or other measurement errors.

$$\tau = m\dot{\gamma}^n \quad \text{(E.6)}$$

where m is the power law consistency coefficient, n is the power law exponent, and $\dot{\gamma}$ is the shear rate.

When a rheogram possesses a non-zero y-intercept, the fluid is said to possess a yield stress. A yield stress is a shear-stress threshold that defines the boundary between solid-like behavior and fluid-like behavior. The fluid will not begin to flow until the yield-stress threshold is exceeded. A rheogram for a

Bingham plastic does not pass through the origin; for Bingham plastic materials, once enough force has been applied to exceed the yield stress, the material approaches Newtonian behavior at high shear rates (Equation E.7).

$$\tau = \tau_B + \mu_B \dot{\gamma} \quad \text{(E.7)}$$

where τ_B is the Bingham yield stress, μ_B is the plastic viscosity, and $\dot{\gamma}$ is the shear rate.

Fluids that exhibit a non-linear rheogram with a yield stress can be modeled by the three-parameter Herschel-Bulkley equation (Equation E.8). Again, shear-thickening behavior is uncommon, and typically the Hershel-Bulkley power-law exponent is less than unity.

$$\tau = \tau_H + k\dot{\gamma}^b \quad \text{(E.8)}$$

where τ_H = yield stress
k = Herschel-Bulkley consistency coefficient
b = Hershel-Bulkley power law exponent
$\dot{\gamma}$ = shear rate.

If one desires a rheology model that is nonlinear but has a yield stress and infinite shear viscosity, the Casson fluid model can be used. This rheology model is shown as Equation (E.9).

$$\tau^{\frac{1}{2}} = \tau_C^{\frac{1}{2}} + \left(\mu_C \dot{\gamma}\right)^{\frac{1}{2}} \quad \text{(E.9)}$$

where τ_C is the yield-stress fit to the Casson fluid model (Pa), and μ_C is the Casson infinite shear viscosity (Pa•s).

An example of these rheological properties can be considered with a scenario of pipeline-flow through a 3-inch-ID smooth pipe transporting fluid at 90 gallons per minute. This equates to an average pipeline velocity of 4.1 ft/sec. The fluid is a Bingham plastic with a Bingham yield stress, τ_B, of 30 Pa, a Bingham consistency or plastic viscosity, μ_B, of 30 cP, and a slurry density of 1.2 kg/L. In this case, the fluid flow will be in the laminar regime with the velocity and apparent viscosity profiles shown in Figure E.3. The flow profile reflects a "plug flow" regime where the center core of the fluid moves at constant velocity. This is because the shear stress in this region does not exceed the yield stress of the fluid, which acts as a solid material with an infinite apparent viscosity. At a radius of approximately 1.1 inches, the shear stress in the pipe exceeds the yield stress of the fluid, and the fluid transitions from behaving as a solid to behaving as a "shear-thinning" liquid. The apparent viscosity in the sheared region near the pipe wall (1.1- to 1.5-inch radius) drops from an infinite value to approximately 100 cP at the pipe wall. The pressure drop for flow under these conditions is calculated at 9 psig/100 ft of straight horizontal pipe.

The case of a Newtonian fluid with the same pressure drop is then considered. At 90 gpm, a Newtonian viscosity of 300 cP is required for a 9-psi/100-ft pressure drop. The flow profiles for this system are shown in Figure E.4. The flow profile shows a parabolic velocity profile that is characteristic of Newtonian, laminar pipe flows. The apparent viscosity in this case is constant at 300 cP throughout the pipe radius.

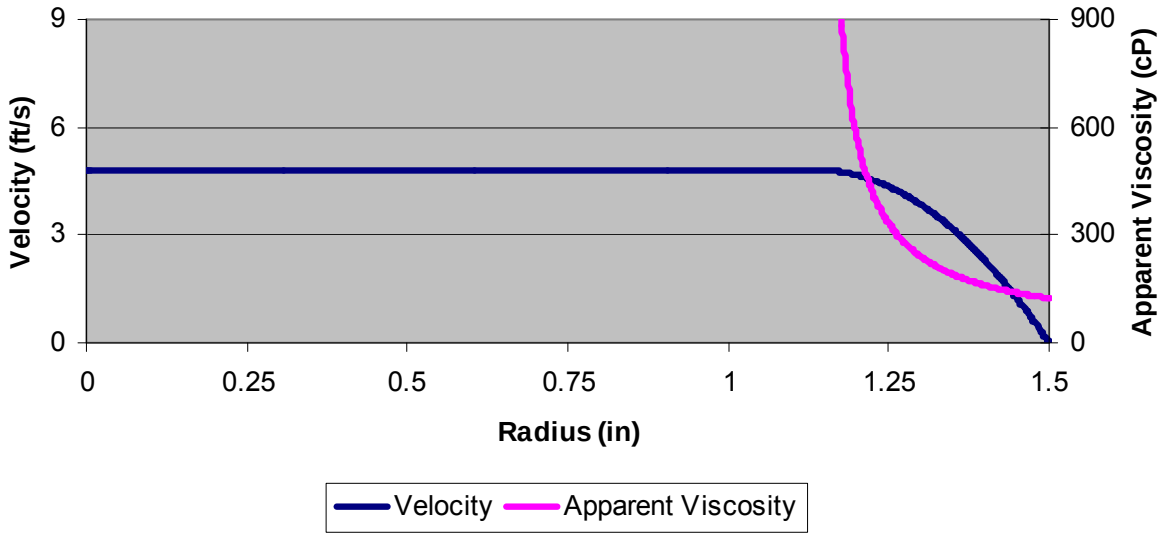

Figure E.3. Example Flow Profiles for a Bingham Plastic Fluid (30 cP consistency, 30 Pa yield stress) in a 3-inch-ID Smooth Pipe at 90 gpm

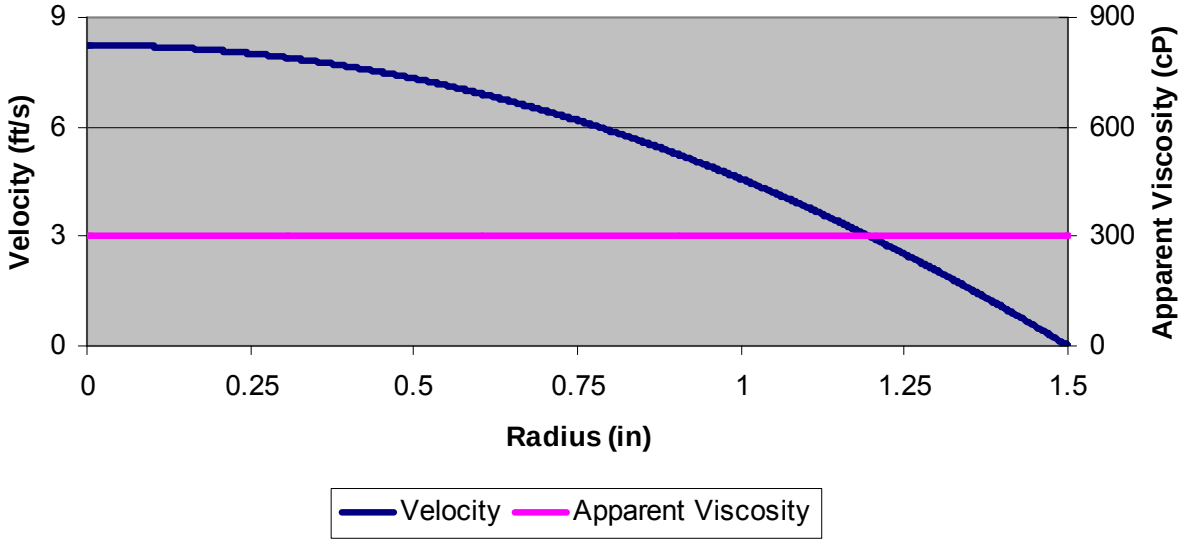

Figure E.4. Example Flow Profiles for a Newtonian Fluid (30 cP viscosity) in a 3-inch-ID Smooth Pipe at 90 gpm

E.2 Chemical and Physical Basis for Rheology

From 2003 to 2006, a DOE Environmental Management Science Project investigated the impacts of colloidal and particle properties on the yield stress and rheology of fluids. The joint University of Washington and PNNL effort produced several papers, including one entitled "Relating Clay Yield Stress to Colloidal Parameters" (Laxton and Berg 2006).

In this paper, Laxton and Berg (2006) explain that Larson (1998, p. 351) has derived a mechanistic relationship for the yield stress of a complex fluid consisting of interacting spherical particles. The approach taken is to model the interactions between the particles as interaction potentials, Φ. At the minima of the interaction potential, the net interparticle force, F, is zero. When the system is at equilibrium, the particles will be in a configuration corresponding to one of these minimum-interaction-potential states. Each equilibrium state has a separation distance between the particles, D_0, where the interparticle forces are at zero and the interaction potential is at a minimum.

If a shear field is placed on the sample at equilibrium, the separation distance will change and the forces between the particles will become non-zero. When the shear is removed, these particles will tend back to the equilibrium conditions where the net interparticle forces are zero.

If a greater shear is placed upon the sample, the separation distance will increase, and the net interaction forces between the particles will go through a maximum as the distance increases. At this point, the sample is said to "yield" and begin to flow. Note that this property arises from a purely cohesive interaction. For granular systems, these "yield" interactions can be frictional in origin; this discussion is limited to colloidal systems where frictional interactions are negligible.

The bulk yield stress is then proportional to the interparticle forces at that separation distance times the number of particle-to-particle contacts per unit area being sheared. Laxton and Berg (2006) explain that the number of particle contacts scales with ϕ^2/r^2, where ϕ is the volume fraction of solids in the sample, and r is the particle radius.

$$\tau_y \propto \frac{\phi^2}{r^2} F\left(D_y\right) \quad \text{(E.10)}$$

The net interactive force between the particles is equal to the derivative of the interaction potential. Laxton and Berg (2006) state that this is roughly equal to $F\left(D_y\right) \approx -\frac{\Phi\left(D_0\right)}{D_0}$.

$$\tau_y \propto -\frac{\phi^2}{r^2} \frac{\Phi\left(D_0\right)}{D_0} \quad \text{(E.11)}$$

According to the Derjaquin, Landau, Verwey, and Overbeek (DVLO) theory, the interaction potential is the sum of the van der Waals and electrostatic potentials.

$$\Phi = \Phi_{vdW} + \Phi_e \quad \text{(E.12)}$$

In the case of monodisperse spherical particles with small relative separation distances, the van der Waals potential can be estimated as shown below:

$$\Phi_{vdW} \approx \frac{rA_H}{12D} \quad \text{(E.13)}$$

where A_H is the Hamaker constant.

Assuming constant surface charge density on the particles, the electrostatic potential can be estimated as follows:

$$\Phi_e \approx 2\pi\varepsilon\varepsilon_0 r\psi_s^2 \ln\left[\frac{1}{1-\exp(-\kappa D)}\right] \quad \text{(E.14)}$$

where ε = dielectric constant of the medium
ε_0 = permittivity of a vacuum
ψ_s = surface charge potential
κ = inverse Debye length.

Combining these equations and replacing the surface potential with the zeta potential, ζ, produces the following relation:

$$\tau_y \propto \frac{\phi^2}{r}\left\{\frac{A_H}{12D_0^2} - \frac{2\pi\varepsilon\varepsilon_0}{D_0}\zeta^2 \ln\left[\frac{1}{1-\exp(-\kappa D_0)}\right]\right\} \quad \text{(E.15)}$$

The inverse Debye length can be approximated as

$$\kappa = \sqrt{\frac{2N_a e^2 I}{\varepsilon\varepsilon_0 kT}} \quad \text{(E.16)}$$

where N_a = Avogadro's Number
e = elementary charge
I = ionic strength of the electrolyte
k = Boltzmann's constant
T = temperature.

As explained by Laxton and Berg (2006), an estimate of the Hamaker constant can be obtained from Lifshitz theory for two identical solid phases (1) across a medium (2) using the following equation:

$$A_H = \frac{3}{4}kT\left(\frac{\varepsilon_1 - \varepsilon_2}{\varepsilon_1 + \varepsilon_2}\right)^2 + \frac{3hv_e}{16\sqrt{2}}\frac{\left(n_1^2 - n_2^2\right)^2}{\left(n_1^2 + n_2^2\right)^{3/2}} \quad \text{(E.17)}$$

where h is the Planck constant, n is the refractive index, and v_e is the frequency of electron cloud oscillations (typically 3 to 5×10^{15} s^{-1}).

The particle separation at minimum interaction potential typically has a value of 0.1 to 0.3 nm.

Zhou et al. (2001) normalized and simplified the above equation and obtained the following relation.

$$\frac{\tau_y}{\tau_{y\max}} = 1 - \frac{24\pi\varepsilon\varepsilon_0\zeta^2 D_0^2}{A_H\left(1 + e^{\kappa D_0}\right)} \quad \text{(E.18)}$$

where A_H = Hamaker constant of the slurry

D_0 = minimum separation distance between the particles in a flocculated state

ε_r = dielectric constant

ε_0 = permittivity of a vacuum

κ = inverse Debye length.

Gao et al. (2003) make the following statement about this equation:

> A feature of the above equation is to correlate the normalized shear yield stress with surface chemistry independently of the structural properties of particles. Thus, all normalized data of yield stress for the suspensions with a variety of solids concentrations should collapse onto a single master curve in a plot against the square of zeta potential or pH.

The equations presented above represent ideal situations where the DLVO theory can be directly applied. These types of situations are rare in practice, but the effect of several physical and chemical variables can be predicted. A table summarizing the expected trends is shown below. Note that chemical properties such as pH and dissolved salt content have a dramatic effect on the yield stress of a suspension.

If one takes a stable suspension and begins to alter the pH or dissolved salt content, the suspension will begin as a stable suspension with a low yield stress. As the zeta potential approaches the isoelectric point (IEP), the suspension will begin to flocculate or form a solid structure that increases yield stress. Further shifts in pH or salt content will stabilize the suspension as the suspension progresses away from the IEP. However, even further changes in pH or salt content result in the inverse Debye length being the dominant factor rather than the zeta potential, and the suspension will again flocculate and increase the yield stress. The dynamics of this system are discussed in Table E.3.

E.3 Rheology of Real Systems

Ferraris et al. (2001, pp. 215–241) provide an equation that describes how the Bingham-plastic consistency parameter varies with solids concentration:

$$\mu_B = me^{n\phi} \quad \text{(E.19)}$$

where μ_B is the Bingham consistency in cP, ϕ is the solids volume fraction, and *m* and *n* are the fitting parameters.

Table E.3. Physicochemical Factors that Affect Slurry Rheology

Physicochemical Factors	Colloidal Variables Affected Fun	ctionality	Result
pH and Dissolved Salt Content	Zeta potential	Moderate pH values tend to push Zeta potential to zero at IEP.	Measured yield stress approaches maximum at IEP.
Dissolved Salt Content	Inverse Debye length	Increases with increasing ionic strength.	Measured yield stress approaches maximum under high ionic strength conditions.
Undissolved Solids Content	Solid-particle volume fraction	Volume fraction increases with undissolved solids content.	Maximum yield stress increases with increasing undissolved solids content.
Particle Size	Particle radius	Increases with particle size.	Maximum yield stress decreases with increasing particle size.
Solid and liquid chemical morphology and speciation	Hamaker constant and equilibrium particle separation distance	Varies with chemical particle morphology and system chemical composition.	Varies with chemical particle morphology and system chemical composition.

Zhou et al. (2001) provide an equation that describes how the Bingham yield stress varies with solids concentration (Equation 4.11). This equation is of similar form to the shear-strength equation.

$$\tau_B = p\phi^q \quad \text{(E.20)}$$

where τ_B is the Bingham yield stress, ϕ is the solids volume fraction, and *p* and *q* are the fitting parameters.

The parameters *m*, *n*, *p*, and *q* are a function of solids and liquid composition, pH, temperature, the state/configuration of particle aggregation, and particle size. These equations are typically valid only when the chemistry of the slurry is held constant while varying the solids loading. This can be achieved by diluting the slurry with decanted or separated interstitial liquid that is at equilibrium with the slurry solids. The yield-stress equation based on DLVO theory predicts a dependence of an inverse relationship between particle size and yield stress. However, many experiments have demonstrated that yield stress is

proportional to the inverse square of particle size. On this topic, Zhou et al. (2001) state, "Although the perplexing question of why the additional inverse size dependence observed is open for discussion, it is not likely to come from the force dependence (which is well predicted by traditional DLVO equations)."

The fluid rheology of waste slurries intended for treatment at WTP is dependent on many complex parameters that may change dramatically during physical and chemical processing. For instance, as they exist in the tanks, the initial waste tank solids are suspended or slurried in a saturated salt solution. Retrieval and transport of these slurries to waste pretreatment operations is facilitated with water dilution. The dilution will lower the pH, the dissolved salt content, and the solids volume fraction due to dissolution of salt crystals, particle size, and solid/liquid chemistry. Extreme batch-to-batch variability due to the different processes that generated waste being retrieved from a particular waste tank also adds to the complexity.

The retrieved high-level waste (HLW) slurry feed will then be processed through a leaching operation where the solid aluminum and chromium phases will be removed with the remaining solids concentrated to a target of 20 wt% undissolved solids. The HLW slurry interstitial liquid will be replaced with "inhibited water," which is a 0.01-M sodium hydroxide solution, to form a pretreated HLW sludge stream. Again, changes to pH, dissolved salt content, solids volume fraction, particle size, and solid/liquid chemistry will occur. The permeate from this leaching operation, also called pretreated low-activity waste (LAW), will change because dissolved aluminum and chromium species will be added as well as water needed for unit operation.

Lastly, glass-former chemicals (GFCs) will be added to the pretreated HLW sludge and LAW streams. The GFC composition, which contains a mixture of soluble and insoluble species, will vary from batch to batch. Again, changes to pH, dissolved-salt content, solids volume fraction, particle size, and solid/liquid chemistry will occur.

E.4 Recommendations

Due to the level of complexity discussed above, a set of findings and recommendations has been successful in obtaining rheological models to be used for DOE-EM cleanup applications. These recommendations have been compiled in a rheology- and physical-properties-characterization guidelines document that is attached as Appendix D in this document. The guidelines document was used as a consensus method for obtaining data in the same manner between different laboratories such as Pacific Northwest National Laboratory, Savannah River National Laboratory, and the Vitreous State Laboratory in Washington, DC. The guideline documents discuss rheometry with concentric-cylinder systems. Measurements with cone/plate and plate/plate geometries were prone to errors due to particle jamming in the narrow gap and drying of the slurry around the edge of the cone or plate. The concentric-cylinder geometry avoids these problems as most of the measurement-surface area is submerged in a wider-gap system. Particle-settling problems still exist with the concentric-cylinder geometry. However, the off-bottom distance can be increased to eliminate the sensor rotating in a bed of sediment.

Many of the slurries characterized in the Hanford, SRS, and INL tank farms exhibit time dependent thixotropic rheological properties. For this reason, the guide recommends a ramp in shear rate from 0 to 1000 1/s over a five minute period. The 1,000 1/s shear rate is then held for one minute and a rampdown from 1000 to 0 1/s is performed. Often a hysteresis loop is observed. The procedure is repeated several

times until the rheological structure is broken down and the hysteresis loop is repeatable. Then a series of rheological model fits are performed with Newtonian, power-law, Bingham plastic, and Herschel Bulkley models obtained for the increasing and decreeasing shear-rate ramps. This process should be repeated for each sample that represents a change in solids concentration or chemistry.

Of these model fits, the Newtonian and Bingham-plastic models are preferred for design purposes. For slurries, the Bingham-plastic fit offers a conservative yield-stress value under zero shear. As shear rate is taken to infinity, a constant non-zero value for viscosity is extrapolated. In addition, the Bingham-plastic model only uses two-parameters and a relatively large number of solved applications using this model are discussed in the literature. The Casson model offers both of these features with the advantage of having a degree of curvature on the flow curve. However, the Casson model has a much smaller number of solved applications available in the open literature.

For dilute slurries or supernatant, the Newtonian rheological model is often used. Taylor vortices are often observed when using the techniques discussed in the rheology guidelines with low-yield-stress materials. The guidelines provide a mathematical check to determine when these secondary flow patterns are to be expected.

The guidelines also provide guidance on how to perform shear-strength measurements with a shear vane. Wall effects can be significant when performing these measurements and criteria for distance between the vane and the container surfaces are provided. Lastly, the guidelines discuss how to perform moisture-content measurements in order to obtain solids concentrations in high molarity salt slurries.

E.5 References

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