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Crystalline Silicotitanate Gate Review Analysis

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J.L. Swanson
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November 1997

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

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Richland, Washington 99352

Summary

Crystalline silicotitanate (CST) is an ion-exchange method for removing radioactive cesium from tank waste to allow the separation of the waste into high- and low-level fractions. The CST, originally developed by Sandia National Laboratories personnel in association with Union Oil Products Corporation, has both a high affinity and selectivity for sorbing cesium-137 from highly alkaline or acidic solutions. For several years now, the U.S. Department of Energy has funded work to investigate applying CST to large-scale removal of cesium-137 from radioactive tank wastes. In January 1997, an expert panel sponsored by the Tanks Focus Area met to review the current state of the technology and to determine whether it was ready for routine use. The review also sought to identify any technical issues that must be resolved or additional CST development that must occur before full implementation by end-users.

The CST Gate Review Group concluded that sufficient work has been done to close developmental work on CST and turn the remaining site-specific tasks over to the users. This report documents the review group's findings, issues, concerns, and recommendations as well as responses from the Tanks Focus Area expert staff to specific pretreatment and immobilization issues.

Contents

Acronyms

CST	crystalline silicotitanate
DOE	U.S. Department of Energy
FY	fiscal year
HLW	high-level waste
NCAW	neutralized current acid waste (waste type at Hanford)
PCT	Product Consistency Test
TCLP	Toxicity Characteristic Leaching Procedure
TFA	Tanks Focus Area
TRU	transuranic (elements)
UOP	Union Oil Products Corporation

1.0 Review Analysis

The U.S. Department of Energy (DOE) is responsible for remediating radioactive waste in 273 underground tanks at the Hanford Site (Washington), Idaho National Engineering and Environmental Laboratory (Idaho), Oak Ridge Reservation (Tennessee), and Savannah River Site (South Carolina). The approximately 94,000,000 gallons of waste in these tanks needs to be characterized, retrieved, pretreated, and immobilized to reduce the risks to the public and the environment. One of the challenges of this task is the waste's composition. Over more than half a century, as a result of creating and refining nuclear materials, a vast array of chemicals and radionuclides have been intimately mixed together to form the supernatant saltcake and sludge in the tank waste. With appropriate technologies, the waste can be separated into low- and high-level waste. If the low- and high-level components can be separated, great cost savings can be realized.

One method for separating the low- and high-level fractions in supernatant and dissolved saltcake is to remove the radioactive cesium by pumping the waste through a series of ion-exchange columns loaded with crystalline silicotitanate (CST). The CST, originally developed by Sandia National Laboratories personnel in association with Union Oil Products Corporation (UOP), has both a high affinity and selectivity for sorbing cesium-137 from highly alkaline or acidic solutions. For several years now, DOE has funded work to investigate applying CST to large-scale removal of cesium-137 from radioactive tank wastes.

The experimental work on CST has involved both batch and column tests with a variety of simulated and actual waste solutions. This work has been done to establish the general applicability of CST for effective removal of cesium-137 and to develop process flowsheet parameters for such applications. The experimental work has also included tests to establish that cesium-137-loaded CST material can be satisfactorily converted to a durable borosilicate glass either by itself or in admixture with typical Savannah River or Hanford Site sludges.

As a result of full-scale column tests and early results from the immobilization effort, the CST technology was perceived as moving from one technology maturity phase to another (from demonstration to implementation). The Tanks Focus Area (TFA) performed a review of the CST technology to confirm whether it was ready for routine implementation. The analysis was performed by five experts in pretreatment and immobilization. Information was presented by the Technology Integration Managers (Phil McGinnis for pretreatment and John Plodinec for immobilization) and principal investigators (Doug Lee, Doug Hendrickson, Daro Ferrara, Mary Andrews, Ted Krause, and MaryLou Balmer). The gate review objectives were two-fold. The first objective was to take a collective look at the developmental work on CSTs to date as well as planned CST development efforts. The second objective was to identify the technical issues that must be resolved or additional CST development that must occur before "closing the book on the development of CSTs" for use by end-users. Three criteria were used to achieve these objectives.

1. Are the performance characteristics sufficiently defined and validated (or will be defined and validated by the end of the fiscal year) for end-user selection for process feasibility testing and/or deployment?

2. Is sufficient information available to the end-users to estimate life-cycle and capital costs associated with the use of CST in pretreatment and immobilization processes?

3. Have significant issues related to safety, environmental protection, and disposal in the use of CST been identified and addressed?

The responses of the CST Gate Review Group were overwhelmingly "yes" to the three criteria listed in the previous paragraph. They believe that sufficient work has been done to close developmental work on CST and turn the remaining site-specific tasks over to the users. A brief discussion of the group and the meeting is in Section 2.0. The review group's findings are presented in Section 3.0. The recommendations from the group and the responses made are in Section 4.0. Issues and concerns are in Section 5.0

2.0 Review Group Introduction

The TFA Technical Team in December 1996, chartered a five-member CST Gate Review Group. The members of the CST Gate Review Group were as follows:

Rudy Carreon, DOE's Richland Operations Office
Joseph A. Gentilucci, Independent Consultant
Wallace W. Schulz, Independent Consultant
John L. Swanson, Independent Consultant
E. Thomas Weber, Independent Consultant

The last four listed members are also members of the TFA Technical Advisory Group.

For several years now, DOE has sponsored work to investigate applying CST to large-scale cesium-137 removal from a host of radioactive wastes now stored at Hanford, Idaho, Oak Ridge, and Savannah River Sites. The experimental work has involved both batch and column tests with a variety of simulated and actual waste solutions to establish the general applicability of CST for effective removal of cesium-137 and to develop process flowsheet parameters for such applications. The experimental work has also included tests to establish that cesium-137-loaded CST can be satisfactorily converted to a durable borosilicate glass either by itself or in admixture with typical Savannah River or Hanford Site sludges.

The CST Gate Review Group convened in Richland, Washington, on January 14 - 15, 1997, to judge if CST has been sufficiently developed and tested for an end-user to determine if large-scale deployment of this sorbent is a technically and economically viable option. Scientists and engineers from several national laboratories and sites provided an excellent status of experimental results concerning CST use for radioactive tank waste pretreatment and immobilization of potential secondary waste streams resulting from pretreatment. However, information concerning synthesis and physical properties of the engineered form of CST were not presented.

The CST Gate Review Group met in executive session on January 15, 1997, to make judgments concerning the CST demonstration-to-implementation gate. This report presents the review group's findings and some recommendations for additional experimental work to be completed in the remainder of FY97 or, if necessary, in FY98.

3.0 Findings from the Review Group

The four non-DOE members of the CST Gate Review Group responded individually to three questions regarding the status of development of CSTs. The group first modified the wordings of the three draft questions provided by the TFA Technical Team to the following:

1) Are the performance characteristics sufficiently defined and validated (or will they be defined and validated by the end of the fiscal year) for end-user selection for process feasibility testing and/or deployment?

2) Is sufficient information available to the end-users to estimate life-cycle and capital costs associated with the use of CST in pretreatment and immobilization processes?

3) Have significant issues related to safety, environmental protection, and disposal in the use of CST been identified and addressed?

The four group members then voted "yes" or "no" on each of these questions separately for pretreatment and immobilization. These votes were made by secret ballot with no discussion other than that which had occurred during the modifications of the questions. All votes were "yes" with the exception of one "no" vote in each of the following four cases:

a) Question 2 for both pretreatment and immobilization.
b) Questions 1 and 3 for immobilization.

The issues, especially where there had been a "no" vote, were then discussed by the group members. It was found that the "no" votes for Question 2 resulted from one member having a different perception from the other members regarding the type of estimate implied in the question. When the issue was discussed in terms of a "conceptual design level" estimate, the "no" voter became comfortable with changing his vote to a "yes."

The "no" votes on Questions 1 and 3 for immobilization resulted from one reviewer responding on a different basis than the other three reviewers. The "no" vote was based on the feeling that some significant questions remained unanswered, at least for application at some sites. The "yes" voters agreed with many of the specific concerns, and indeed had other concerns of their own (in pretreatment as well as immobilization). These considerations were concluded to be of secondary importance and will be discussed in later sections of the report. The group consensus is that the current level of development was sufficient for end-users to decide whether they were interested in pursuing the development and use of CST, and responded to the question in that context.

All group members feel that the completeness of the current body of data for an end-user's application varies widely among end-users. It is felt that the data needed for application at Oak Ridge will be essentially complete by the end of FY97. The data needed for application at Hanford, Savannah

River, and Idaho sites are thought to be less complete; however, the current data are certainly judged to be sufficient for end-users at these sites to decide whether their unique situations warrant continued evaluations of the use of CST. The review group recognizes the potential need for additional development testing to resolve site-specific issues.

In summary, the responses of the CST Gate Review Group were overwhelmingly "yes" to the three questions listed in the first paragraph of this section. This does not mean that they feel that no further development work needs to be done to have CST applied at the candidate sites. Partial lists of the perceived development needs are presented in subsequent sections of this report. Recommendations on the source of funding (e.g., EM-50, EM-30, private contractor) for such development work are considered to be beyond the charter of this gate review group.

4.0 Recommendations and Responses

4.1 Pretreatment

Recommendation 1: The concept of disposing of a cesium-loaded CST as a low-level waste needs more complete and careful consideration before it is carried very far. This concept may be "regulatorily allowable" for material from the Oak Ridge Site but not for material from the other tank waste sites. Even when such disposal might be allowed, workers need be concerned with factors other than the maximum allowed cesium-137 concentration in low-level waste. For example, the loaded CST must also not contain transuranic (TRU) element activity in excess of 100 nCi/g. Evaluation of this effect must consider TRU elements that are associated with solids present in the feed as well as TRU elements that are in solution.

Response to Recommendation 1: The TFA has created a supernate team with which to address issues. An earlier review by the supernate team did not result in feedback to the TFA on this issue. The TFA has no indication that TRUs are sorbed on CST, nor does the TFA have evidence that TRU downstream of a filter is a problem. This will be discussed with the supernate conference call in more detail.

Recommendation 2: The potential importance of the loading of chromium on CST should be evaluated, first through paper study evaluation and then by experimental measurements as indicated. For example, is the solubility of chromium (III) in a waste solution high enough that, if it were all sorbed by CST during cesium removal, the loaded CST would contain sufficient chromium to cause a problem in subsequent vitrification operations, or to require the loaded CST to be managed according to Resource Conservation and Recovery Act of 1976 or Comprehensive Environmental Response, Compensation, and Liability Act of 1980 regulations? The potential for competition of CST sites between small molar concentrations of cesium and much higher concentrations of chromium needs to be carefully considered.

Response to Recommendation 2: The sludge investigations note the chromium is almost exclusively chromium (VI), not chromium (III). We have done flow tests on samples from two Oak Ridge Site tanks and four Hanford Site tanks, as well as several Savannah River Site samples, with no indication of chromium competition. An earlier review by the supernate team did not identify chromium competition as a problem.

Recommendation 3: Conduct, preferably yet in FY97, a series of CST column tests with several (three to four) simulated (cesium-137 traced) Hanford and Savannah River Site waste solutions to evaluate cesium sorption efficiency over a wide range of superficial flow velocities. The range of superficial flow velocities investigated should embrace those expected in large-scale CST column operation at the Savannah River and Hanford Sites.

Response to Recommendation 3: The TFA has requested the Efficient Separations and Processing Crosscutting Program at the Oak Ridge Site to consider this recommendation. The Cesium Removal Demonstration has run at 6 CV/hr, which is high.

Recommendation 4: The status of computer modeling of cesium sorption by CST should be evaluated to see if additional development by the model originators is needed. This work probably cannot be started before the end of FY97, but it could be an important activity and continuation should be considered.

Response to Recommendation 4: Sandia National Laboratories has not made the model available to either the Efficient Separations and Processing Crosscutting Program or TFA yet. This was discussed with the crosscutting program at their midyear review on the first week of April 1997.

Recommendation 5: Conduct "paper" studies to compare presently available and new (obtained in remainder of FY97) CST column performance data with predictions of the Texas A&M "thermodynamic" model to more closely estimate the mass of CST needed to pretreat various suites of Hanford tank wastes, e.g., double-shell tanks only, double-shell tank plus single-shell tank saltcake. A key issue in consideration of the use of CST to pretreat Hanford tank waste is the mass of cesium-loaded CST that will require handling and disposal.

Response to Recommendation 5: The TFA does not have access to the model. This is a worthy study. We will investigate contracting with Texas A&M to do this for us.

Recommendation 6: Additional large-scale testing of CSTs is thought to be necessary to develop more complete information on factors such as pressure drop, height/diameter ratios, and attrition of the sorbent during transfer into a column and during column operation. This testing should be done under conditions closely matching those thought to be desirable by the end-users, and can be done satisfactorily in nonradioactive environments.

Response to Recommendation 6: The TFA does not have the resources to do this in FY97.

Recommendation 7: Make a strenuous effort to obtain quantitative data for the behavior of strontium-90 and TRU elements in the scheduled CST column test with Hanford Site complexant concentrate waste.

Response to Recommendation 7: This request has been made to Pacific Northwest National Laboratory and is part of their test plan.

Recommendation 8: Request the personnel at the Oak Ridge Site who are performing or will perform the CST column demonstration test with actual Melton Valley Storage Tank waste to conduct their tests in a manner that facilitates collection of engineering-scale data, e.g., different column height/diameter ratios, different superficial flow velocities.

Response to Recommendation 8: This request has been made to the Cesium Removal Demonstration team.

4.2 Immobilization

Recommendation 1a: For planned FY97 tests to define glasses containing CST and Hanford Site neutralized current acid waste (NCAW), obtain NCAW simulant that contains noble metals or add noble metals to incorporate this issue in the test program.

Recommendation 1b: For crucible tests to assess glasses containing Hanford sludges plus CST, pursue obtaining simulants from Pacific Northwest National Laboratory for the most relevant Hanford high-level waste (HLW) streams in addition to NCAW, such as

- simulant that would closely approximate the HLW composition envelope defined in the Hanford privatization request for proposal
- simulant reflecting the blend of all waste per the Hanford tank waste reference flow sheet
- Tank 106-C simulant.

Response to Recommendations 1a and 1b: These recommendations are accepted without reservation. They have been forwarded to the investigators associated with the CST immobilization tasks, and the recommendations as being addressed as part of the FY97 work scope.

Recommendation 2: Savannah River Technology Center should utilize prior data on vitrification of Savannah River Site sludge streams processed in Integrated Defense Waste Processing Facility Melter System (test facility), mini-melter and crucible tests to validate FY97 data on feasibility of CST plus sludge processing.

Response to Recommendation 2: These recommendations are accepted without reservation. They have been forwarded to the investigators associated with the CST immobilization tasks, and the recommendations as being addressed as part of the FY97 work scope.

Recommendation 3: Savannah River Technology Center should also assess the relevance of FY97 test data obtained using glass-forming chemicals in relation to Defense Waste Processing Facility (at Savannah River Site) process development testing and plant processes using frit.

Response to Recommendation 3: These recommendations are accepted without reservation. They have been forwarded to the investigators associated with the CST immobilization tasks, and the recommendations as being addressed as part of the FY97 work scope.

Recommendation 4: Establish plans for a new program task, which will extend beyond currently planned FY97 work, to develop a low-temperature process for an alternative CST-only waste form.

Response to Recommendation 4: The TFA will not implement this recommendation at this time. None of the tank sites have indicated a need for a low-temperature process for a CST-only waste form. Unless a user comes forward, the TFA believes that resources should not be directed toward this activity. However, by implementing recommendation 6, the TFA may spark the development of such a need.

Recommendation 5: Perform leach testing to include measurements of actual cesium release from different CST waste forms for comparison and to support disposal/storage analyses (i.e., unconsolidated CST, CST-only glass, and any alternative CST waste forms). This could involve modifying the Product Consistency Test (PCT) or Toxicity Characteristic Leaching Procedure (TCLP) testing protocols currently in use, or establishing additional relevant test methods.

Response to Recommendation 5: The TFA conditionally accepts this recommendation. Mary Andrews of the Savannah River Technology Center will compare the PCT responses of cesium-loaded CST and of CST-only glass. However, because there is no other CST waste form currently available, this is the only comparison that will be made at this time. If other CST waste forms are developed in the future, this data will be used for comparison with the performance of those forms as well.

Recommendation 6: TFA should undertake an initiative to open innovative cesium (and possibly strontium) disposal considerations in conjunction with potential applications of CST technology. This should be aimed toward bringing focus within the DOE complex on innovations for cost-effective, long-term storage or disposal of cesium and strontium isolated from tank wastes and transformed into stable waste forms. This could be started through sponsorship of a study to identify existing and potential new scenarios for long-term storage or disposal of separated nuclides with relatively short (tens of years) half lives.

Response to Recommendation 6: The TFA enthusiastically accepts this recommendation. Finding alternative disposal paths for separated radionuclides (potentially including secondary waste streams) could ultimately lead to significant savings for the DOE, without compromising health or safety.

5.0 Issues and Concerns

5.1 Pretreatment

Some members of the CST Gate Review Group were concerned that there is insufficient knowledge of the amount and properties of "fines" present in typical "as received" CST. Similarly, our knowledge of the extent of CST attrition to be encountered in plant-scale column operation is very limited. True, we have assurances from UOP representatives that the rate and extent of CST attrition in engineering-scale operation will be well within acceptable limits. And, early column tests that have been performed to date support the UOP contention. Still, there are no quantitative data on "as received" fines or attrition-derived fines that a design engineer can use to provide an assured operational column system.

Planned column tests with Hanford saltcake and concentrated complexant waste are of high priority, and should be completed in FY97 to complete the "initial feasibility" studies. Of the two, the concentrated complexant test should be given higher priority.

The review group was pleased to learn that CST column tests will be done with actual Hanford Site complexant concentrate and did not anticipate that aqueous soluble organic compounds in such waste will compromise sorption behavior of cesium-137. But, such compounds may (likely will) seriously interfere with removal of strontium-90 and TRU elements. An opportunity to obtain data on the behavior of strontium-90 and TRU elements should not be missed because of analytical or other difficulties.

More specific applications (e.g., removal of cesium from sludge leach solution) should not be tested until later, if/when flowsheet planning indicates that separate processing of such a stream is likely.

5.2 Immobilization

The reviewers' assessment of the status of development for immobilization of cesium-loaded CST was subdivided into three areas of consideration. The first area includes CST-only vitrification, where the greatest focus of TFA effort has occurred; the second covers vitrification of CST combined with tank HLW sludge feed; the third covers CST-only alternative waste forms.

The TFA accepts the CST Gate Review Group's findings. In response to the findings, the TFA does not intend to carry out any further development (after the FY97 tasks are completed) related to the immobilization of CST unless this becomes an identified need for one of the tank sites.

The TFA agrees that all of the concerns expressed on immobilization are valid. All of them have been passed on to the investigators responsible for immobilizing CST. As discussed with the gate review group during the review, several of these apply only to specific end-users. The FY97 tasks will address these issues only in the context of the end-users for the results of these tasks. For example, the current CST immobilization tasks will not directly address the implications of noble metals in Hanford wastes.

However, these issues will be addressed for the Savannah River Site and Oak Ridge Reservation wastes. When a Hanford Site user for CST comes forward, issues such as these will be addressed in the Hanford context.

5.2.1 CST-only Vitrification

Review Conclusion:

After completion of the FY97 work (including the vitrification test with radioactive Melton Valley Storage Tank sorbent), CST-only vitrification is considered a viable option for immobilization.

Further Considerations:

Heat loads associated with cesium loading of the waste form should be assessed for any applicable technical limits or relationships to disposal site requirements. This should include assessment of the potential for de-vitrification of the glass.

Work to date has not addressed the implications that actinides sorbed on the CST may have on the final waste form. This is not expected to be an issue for glass quality, but there are implications for waste classification and compatibility with disposal site restrictions.

Absorption on CST of species with low solubility in the glass (e.g., chromium) should be evaluated by the end-user in relation to CST sorption behavior with his feeds.

5.2.2 CST and Sludge Vitrification

Review Conclusion:

After completion of planned FY97 work, CST plus sludge vitrification is considered a viable option, with varying degrees of confidence from site to site. (Note that this conclusion is not based on consideration of total HLW glass volumes and costs for different site-wide flow sheets, but pertains only to technical feasibility of processing at CST loadings in balance with cesium removal needs and typical waste form specifications.)

Further Considerations:

The reviewers agree with the technical approach used in the TFA task to assess CST loadings in sludge plus CST glasses.

Considering data and experience associated with prior testing, there is a relatively high level of confidence in technical viability of processing Savannah River Site HLW sludges in combination with CST. Investigators should consider how testing with glass-former chemicals relates to processing with frit, as in the Defense Waste Processing Facility.

For CST in Hanford NCAW sludge, taken in the context of previous Savannah River Site (and Pacific Northwest National Laboratory) work on this feed stream, there should be a fair level of confidence in technical viability. Implications of noble metals in Hanford HLW sludges should be addressed for CST-sludge glasses, relative to previous results with sludge-only vitrification.

There is a relatively low level of confidence in feasibility of vitrification with CST of other HLW feeds at Hanford and other sites (e.g., Idaho National Engineering and Environmental Laboratory), without performing additional crucible and small-scale melter tests with specific sludge plus CST simulants. The other Hanford HLW sludge streams are not well defined. Any necessary additional work could be done after the waste sludge types are identified.

The reviewers agree with the conclusion of TFA program managers, that further investigation of molten salt processes for separating cesium from CST is not warranted, due to the success in achieving high CST loadings in waste glasses.

5.2.3 CST-only Alternative Waste Form

Review Conclusions:

There is no incentive to develop another ceramic waste form based on use of alternative thermal densification processes.

A significant cost saving is potentially available if a low or ambient temperature process can produce a consolidated CST waste form acceptable for greater-than-Class-C disposal or long-term storage. Future work on a low-temperature process for consolidation of a CST-only waste form, such as inorganic impregnation or low-temperature mineralization, appears justified.

Further Considerations:

There seems to be little or no data on leachability of cesium-137 from unconsolidated CST, which will apparently be sent directly from Oak Ridge to a disposal site. Also, there does not seem to be any data on long-term stability (thermal, radiation, etc.) of CST loaded with radioactive cesium. Such information needs to be available to assess the suitability of loaded CST for disposal or long-term storage.

An alternative, monolithic waste form obtained at low temperatures could have advantages over unconsolidated CST including greater leach resistance, acceptability for lower cost long-term storage, capability to comply with transportation regulations with lower cost packaging, and an improved basis for greater-than-Class-C disposal acceptance.

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