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**In-Process Analysis Program  
for the Isolock Sampler  
at the Gunite and Associated Tanks,  
Oak Ridge National Laboratory,  
Oak Ridge, Tennessee**

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**APPROVALS**

**In-Process Analysis Program  
for the Isolock Sampler  
at the Gunite and Associated Tanks,  
Oak Ridge National Laboratory,  
Oak Ridge, Tennessee**

(BJC/OR-6)

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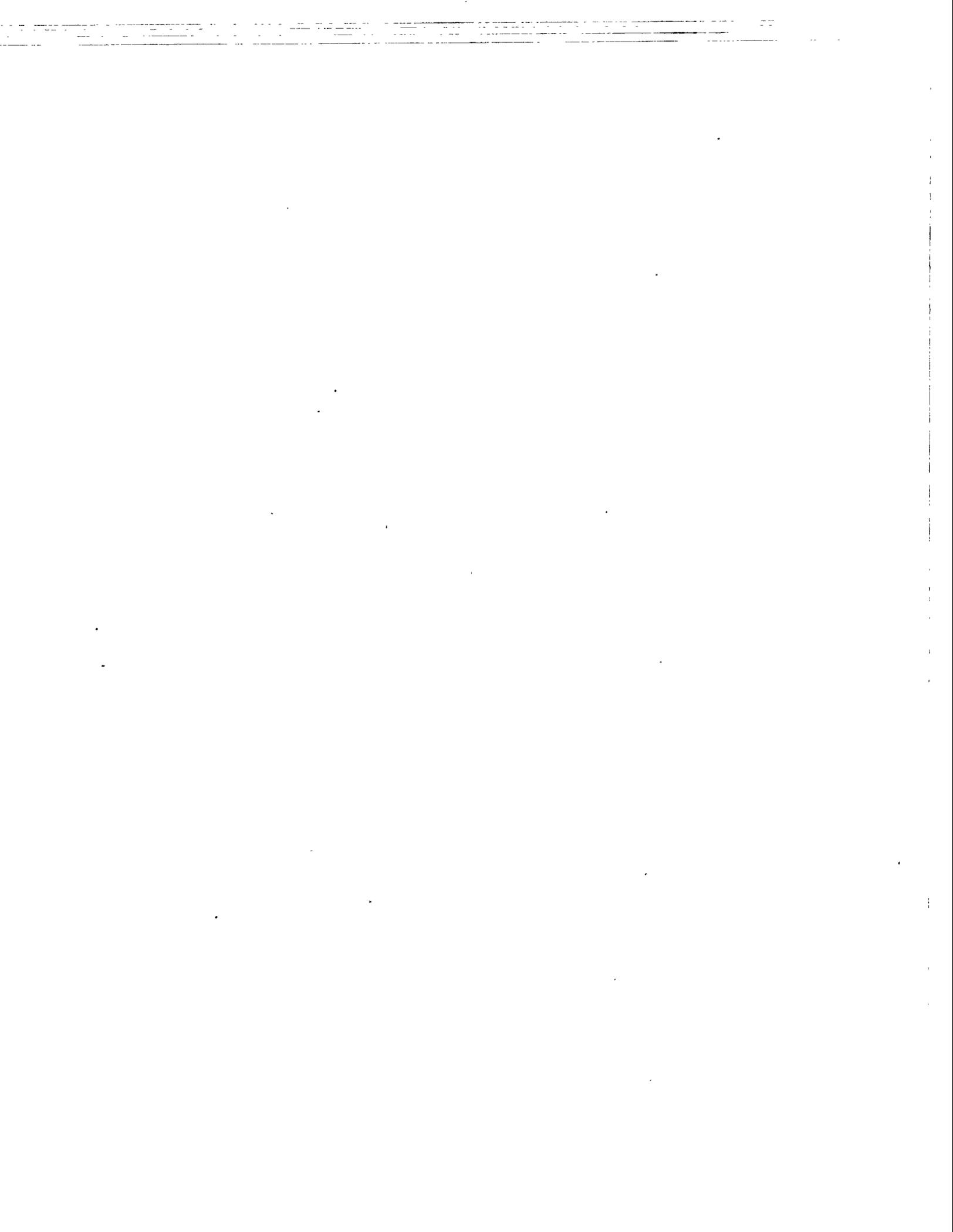
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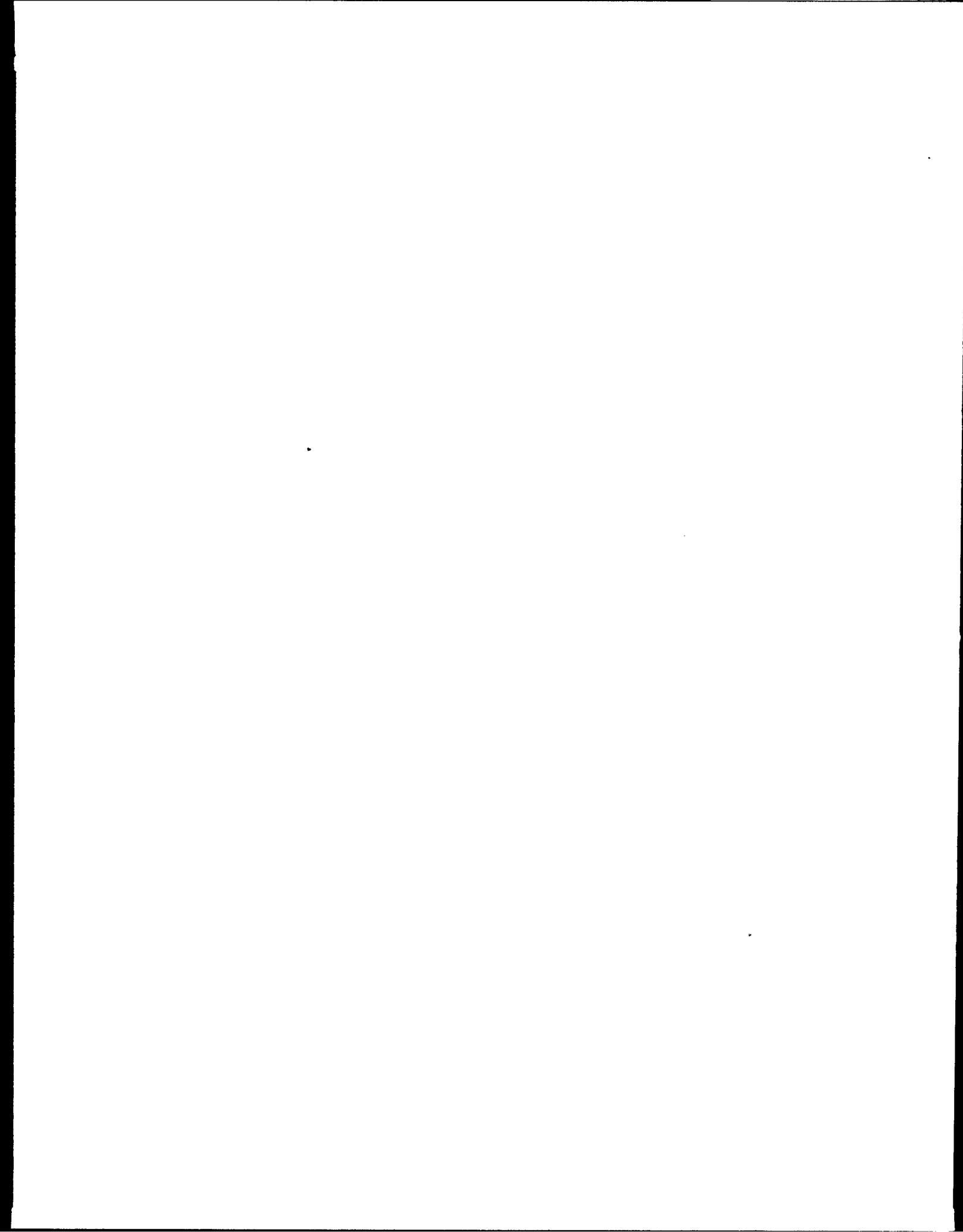
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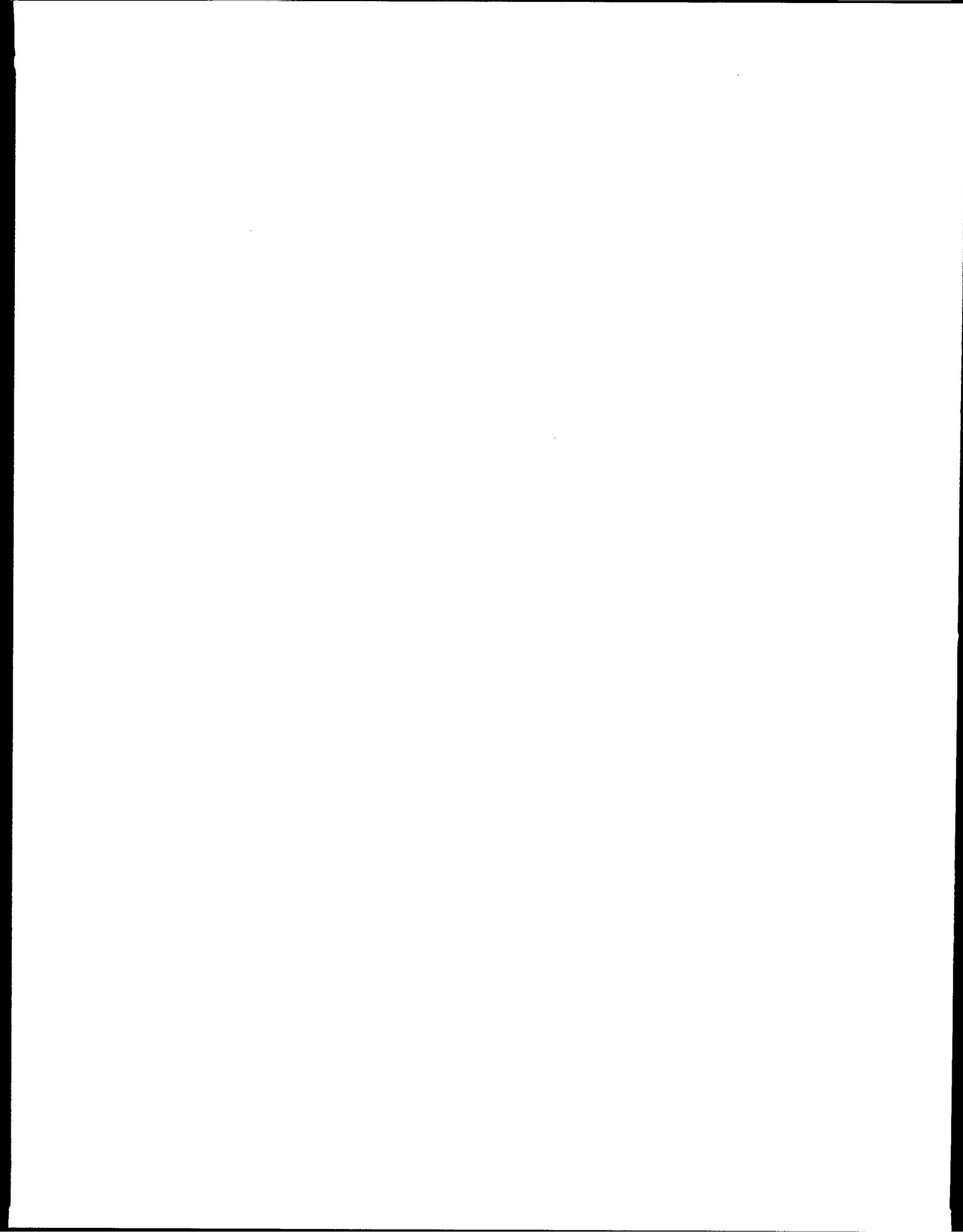
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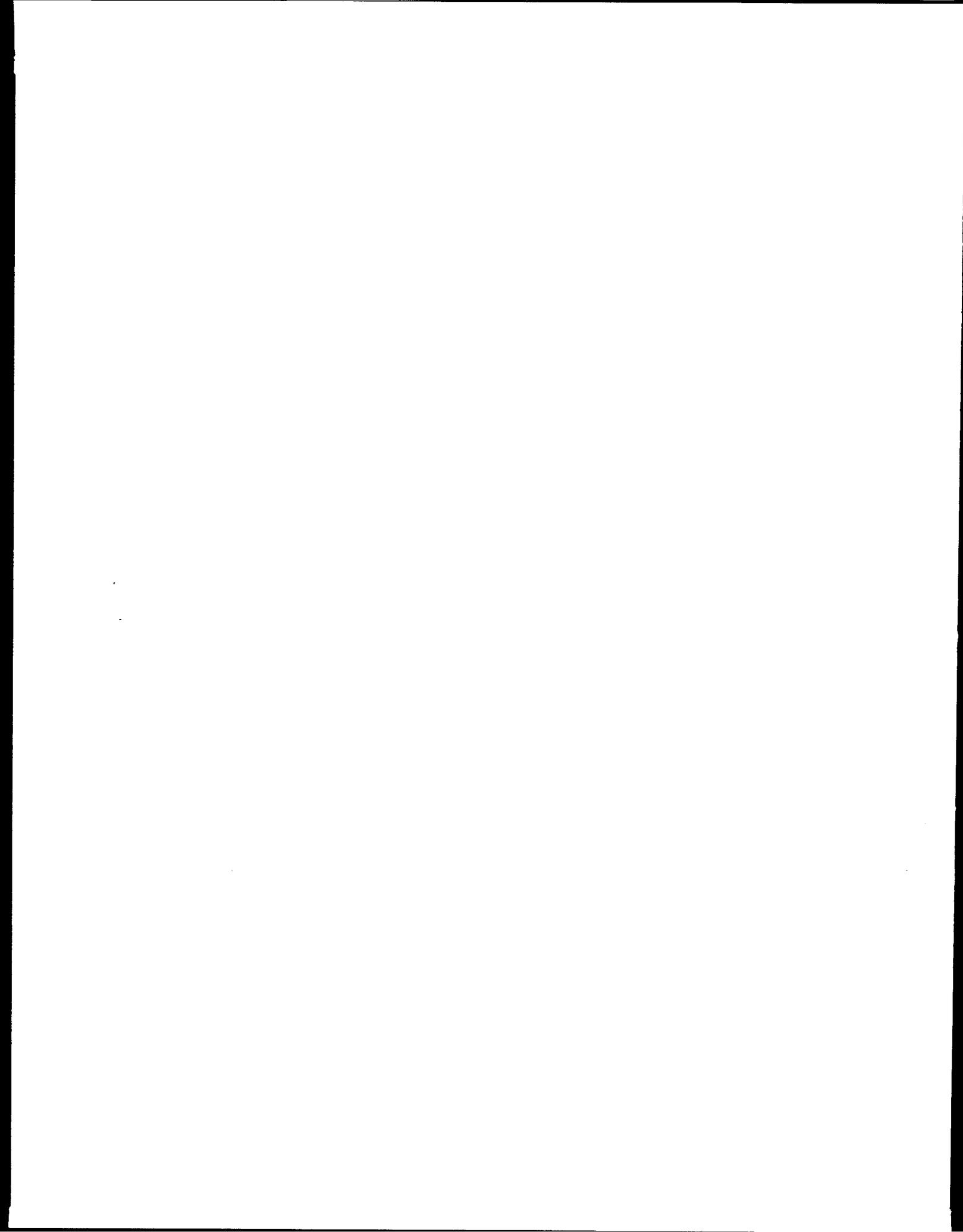
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## ABBREVIATIONS

|      |   |
|------|---|
| DOE  | U.S. Department of Energy                   |
| EPA  | U.S. Environmental Protection Agency        |
| GAAT | Gunite and Associated Tanks                 |
| ORNL | Oak Ridge National Laboratory               |
| RMAL | Radioactive Materials Analytical Laboratory |
| WD&C | Waste Dislodging and Conveyance (System)    |



## **EXECUTIVE SUMMARY**

The In-Process Analysis Program documents the requirements for handling, transporting, and analyzing waste slurry samples gathered by the Bristol Isolock slurry sampler from the Gunitite and Associated Tanks at Oak Ridge National Laboratory in Oak Ridge, Tennessee. Composite samples will be gathered during sludge retrieval operations, labeled, transported to the appropriate laboratory, and analyzed for physical and radiological characteristics. Analysis results will be used to support occupational exposure issues, basic process control management issues, and prediction of radionuclide flow.

# 1. INTRODUCTION

This In-Process Analysis Program document was prepared to identify the requirements for the Oak Ridge National Laboratory (ORNL) Gunite and Associated Tanks (GAAT) project in Oak Ridge, Tennessee. The goal of this plan is to provide data to estimate the number of curies and the volume of solids (i.e., sludge volume) removed from the tanks. These data will be used to support closure of the tanks, compare the expected versus actual waste volume and curies to aid in planning for the tank farm activities, and verify the performance of the in-line instrumentation (flow meter).

This investigation will use the Bristol Isolock<sup>®1</sup> slurry sampler to perform in-process sampling on waste slurry removed from GAAT. The Bristol Isolock slurry sampler will be used to collect slurry samples, and analysis will be performed at the Radiochemical Materials Analytical Laboratory (RMAL). Analyses will include total solids (% moisture); sample density (wet and dry); total curie content (gross alpha/beta, gamma spectroscopy); total radioactive strontium and cesium; and visual observations (color, texture, etc.). Data collected will be used to support occupational exposure issues, basic process control management issues, and prediction of radionuclide flow. Table 1 identifies the data quality objectives, data application, and requirements for the In-Process Analysis Program.

**Table 1. Data quality objectives for Gunite and Associated Tanks sampling**

| Question  | Data application   | Requirement   |
|---|--|---|
| 1. How many curies are removed from each tank?                              | Estimate the curies removed from each tank based on the concentration measured in the sample and the volume of slurry. The volume of slurry will be measured/calculated from instrumentation installed at the tank farm, and this will be multiplied by the concentration determined by laboratory analysis [(volume of waste) × (curie/volume)] | Compute the volume of waste times the radioactive concentration of sample in curies per volume.                                       |
| 2. What is the amount (volume and weight) of solids removed from each tank? | Evaluate waste removal system effectiveness/efficiency, estimate the volume of sludge removed from the tank, and compare to expected tank's volume.  | Determine % solids in the slurry and the wet and dry density of solids in the slurry, and use to compute volume and weight of solids. |

This plan documents those areas in which standard U. S. Environmental Protection Agency (EPA) procedures and protocols cannot be followed because of the nature of the tanks and their contents. Sampling considerations (personnel exposure and tank configuration limitations) are the same as those discussed in the Radiation Work Permit, the Tank Farm Operations Plan, and HAZWOPER plans; therefore, they are not addressed in this plan. Samples will be collected from the piping at the discharge of the waste removal equipment using the Bristol Isolock slurry sampler during sludge retrieval operations. Details of the sampling procedures for the tanks will

<sup>1</sup> Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply endorsement, recommendation, or favoring by the United States Government or any agency thereof.

be described in the Waste Dislodging And Conveyance (WD&C) System Procedure (GAAT-TS/P-122, Rev. 0). Standard ESP procedures will be used by field team for sample identification, chain of custody, sample tracking, data review, and sample security.

The transport of all samples will be according to the guidelines in the Transportation Plan for Movement of Radiological Samples. A Request for Exception form for the GAAT project is in response to the ORNL policy to comply with U.S. Department of Transportation requirements for packaging and transportation of samples on site. A Chain of Custody form will also accompany the sample through the process.

A report of results will be issued after all sampling and analyses have been completed.

For this effort, each tank in GAAT will be sampled during sludge removal operations using the Bristol Isolock slurry sampler. The slurry sampler collection logs and analytical data will be used to estimate the curie content retrieved from each tank.

## 2. RATIONALE, PRIORITY, AND ANALYSIS

Specific sampling parameters have been identified for each functional area: occupational exposure, basic process control management, and prediction of radionuclide flow. Table 2 identifies the basis for sampling in each functional area.

Table 2. Priority and rationale for Gunite and Associated Tanks sampling

| Item | Priority and functional area     | Basis for sampling  |
|------|----------------------------------|---|
| 1    | Basic process control management | Determine the characteristics and properties of the tank contents (total solids, % moisture, density of wet and dry solids, and total curie content of sludge and liquid). The information collected will be used to make basic process control comparisons about the operations.       |
| 2    | Operational exposure             | Determine the characteristics and properties of the tank sludge to estimate the potential operational exposure to the workers from the system.  |
| 3    | Prediction of radionuclide flow  | Determine the characteristics and properties of the pumped sludge material in comparison to the previously estimated sludge curies. The curie inventory of the sludges based on new samples could be used to predict flow of radionuclides from the tanks into the active waste stream. |

The Bristol Isolock slurry sampler will be used to collect composite slurry samples from the discharge pipe of the WD&C system. From the samples, three from each tank will be analyzed at RMAL, and three will be archived for future use.

The composite samples are to be collected and transported to RMAL. Each composite sample will be taken when sludge is being transferred through WD&C. Approximately 10 mL of material will be collected into a 500-mL sample bottle on an intermittent basis (approximately every 20 minutes of processing, not to exceed 375 mL per 500-mL bottle). The sampler will be operated remotely during waste removal operations. The sample will be removed, labeled, and packaged after the transfer system has been flushed and is not operating.

The operator will use the sampler to collect a composite sample into a 500-mL sample bottle. The 500-mL sample bottle will be transported to RMAL after a sample is retrieved. If the surface of the sample bottle exceeds 200 mrem/hr, the bottle will be transported in a shielded container. The Balance of Plant operator will also collect data to complement the sampling activities. Table 3 is a listing of data that will be collected from the graphical user interface during operations that are needed to support the laboratory data to make calculations. Any unusual observations will be noted in the shift supervisor's log.

**Table 3. Data collected from the graphical user interface during operations at the Gunitite and Associated Tanks**

| Question                                      | Analysis  | Operations information  |
|---|---|---|
| 1. How many curies are removed from the tank? | RMAL will analyze slurry samples collected and provide gross alpha, gross beta, total radioactive strontium, and gamma spectroscopy concentrations (Bq per unit volume of the sample) ( $3.7 \times 10^{10}$ Bq = 1 curie). It will be assumed that the samples collected are representative of the average concentration in the waste being removed from the tank. | Information needed from operations will be as follows:<br><ol style="list-style-type: none"> <li>1. Type of operation being performed from the logbook (wall cleaning, bulk sludge retrieval, heel removal, etc.).</li> <li>2. Duration of each activity from the logbook (start time and end time for bulk retrieval, etc.).</li> <li>3. Start and end time for sampler operation and cycle time.</li> <li>4. Initial and final tank level (volume) in the receiving tank for each type of operation (tank level at the start and end times in items 2 and 3).</li> </ol>  |
| 2. Waste removal efficiency/effectiveness?    | Laboratory analysis will provide the % solids and wet and dry density in the sample. It will be assumed that the sample is representative of the average % solids being removed from the tank.  | Information needed from operations will be as follows:<br><ol style="list-style-type: none"> <li>1. Change in volume of waste in the receiving tank (tank level at the start and end times).</li> <li>2. Clean water added to the tank by all operations: <ul style="list-style-type: none"> <li>• Decon (estimated flow rate and operating time for hand decon, FIR-141 for DSR),</li> <li>• Line flushing (FIR-102),</li> <li>• Jet pump operations (FIR-121), and</li> <li>• CSEE cutting jet operation (FIR-131).</li> </ul> </li> <li>3. The difference between the volume increase in the receiving tank and the volume of water added will represent the volume of waste removed from the tank.</li> <li>4. The % solids and wet and dry density of the solids in the sample will be used to evaluate the solid content (weight and volume) of the waste being removed and to determine waste removal efficiency.</li> </ol> |
| 3. Waste volume removed?                      | No data are required from RMAL.   | Information needed from operations will be as follows:<br><ol style="list-style-type: none"> <li>1. Change in level for the receiving tank (tank level at start and end time) for each operation.</li> <li>2. Clean water added to the tank by all operations: <ul style="list-style-type: none"> <li>• Decon (estimated flow rate and operating time for hand decon; FIR-141),</li> <li>• Line flushing (FIR-102),</li> <li>• Jet pump operation (FIR-121), and</li> <li>• CSEE cutting jet operation (FIR-131).</li> </ul> </li> <li>3. The difference between the volume increase in the receiving tank and the volume of water added will represent the volume of waste removed from tank.</li> </ol>   |

RMAL, Bldg. 2026 at ORNL will receive the slurry samples for radiochemical analysis. Standard procedures "Sample Management in the Radioactive Materials Analytical Laboratory, Building 2026 – CASD-OP-RML-AD02, Rev 1" will be followed (i.e., standard procedure for sample receipt, log-in and identity, chain of custody, sample tracking, data review, and sample security). The radiation level of the incoming sample will determine the location used to handle and prepare the samples in preparation for the laboratory analysis (hood or hot cell).

The material collected will be well mixed, and a representative aliquot will be taken, weighed, and put in solution. Samples will be analyzed for total solids (% moisture), density (wet and dry), gross alpha/beta, gamma spectrometry (interested in strontium and cesium), and visual observations (photo and video).

The laboratory sample handling and analytical procedures used will be the standard procedures employed at RMAL. The actual decision on what standard procedures will be carried out will depend on the radioactive reading from the samples. The appropriate methods used will be described in the laboratory's report of results to the GAAT Program. In all cases, any deviations from written procedures during the sampling or analysis will be documented. Samples will be retained according to the laboratory's standard operating procedure (CASD-OP-RML-Ad02), with the exception that no sample will be disposed of without written approval by the project manager. Table 4 discusses the issues to be addressed and the type of analysis to be performed on the samples.

**Table 4. Analysis summary for Gunite and Associated Tanks sampling**

| Issues to be addressed              | Type of sample                       | Types of analysis to be performed on each sample  | Detection limit <sup>c,d</sup> |
|-------------------------------------|--------------------------------------|---|--------------------------------|
| 1. Basic process control management | Isolock sludge sample <sup>a,b</sup> | - Gamma spectroscopy for all significant gamma emitters (CS-137 in particular)<br>- Standard strontium separation | 0.374 Bq/L<br>0.074 Bq/L       |
| 2. Operational exposure             |                                      | - Gross beta using liquid scintillation counting<br>- Gross alpha by dry plate counting                           | 0.15 Bq/L<br>0.11 Bq/L         |
| 3. Predictions of radionuclide flow |                                      | - Total solids (% moisture)<br>- Density (wet and dry)<br>- Visual observations (photo and video)                 | N/A<br>N/A<br>N/A              |

<sup>a</sup>Sample size may vary depending on sample tool effectiveness and collection method.

<sup>b</sup>Analysis protocols per Radioactive Material Analytical Laboratory (RMAL) ORNL Quality Assurance Project Plan. Constituents per RMAL-ORNL/QAPjP (QAP-X-96-CASD/RML-001).

<sup>c</sup>The detection limit for highly radioactive samples will vary and is dependent on such factors as dilutions, counting time, and background levels used during the measurements.

<sup>d</sup>These detection limits are based on the National Primary Interim Drinking Water Regulations.

### 3. ANALYSIS SCHEME FOR SAMPLES

Each sample will be subjected to a battery of analyses to collect information to address the issues of operational exposure, basic process control management, and prediction of radionuclide flow, as shown in Table 4. Table 5 lists the anticipated procedures, based on expected sample radiochemical characteristics for each analysis that will be used for this effort.

**Table 5. Analytical methods for Gunite and Associated Tanks sampling**

| <b>Methods</b>   | <b>Analysis</b>  |
|--|--|
| Sample Management in the Radioactive Materials Analytical Laboratory (CASD-Op-RML-AD02)                                      | Sample and data management   |
| Determination of <sup>89</sup> Sr/ <sup>90</sup> Sr in High Level Samples Using Extraction Chromatography (CASD-AM-RML-RA13) | Radioactive strontium sample preparation   |
| Operations of Packard MO 2500TR Liquid Scintillation Counter (CASD-AM-RML-RA12)  | Alpha- and beta-emitting radionuclides analysis  |
| Gross Alpha and Gross Beta Radioactivity in Drinking Water (CASD-AM-EPA-900.0)   | Gross beta and alpha radioactivity sample preparation  |
| Gamma-emitting Radionuclides in Drinking Water (CASD-AM-EPA-901.1)   | Gamma-emitting radionuclides sample preparation  |
| RMAL-ORNL Quality Assurance Project Plan QAPjP for the Characterization of Radioactive Waste (9QAP-X-96-CASD/RML-001)        | Quality assurance plan for laboratory analysis   |
| Data Acquisition and Processing System (CASD-AM-RML-RA04)  | Data acquisition and processing system   |
| Operation and Calibration of the Tennelec LB4000 (CASD-AM-RML-RA02)  | Gross alpha and gross beta analysis  |
| Density (wet and dry)  | Work Instruction for Percent Moisture and Density Measurement for the GAAT Sludges, dated June 25, 1997. |
| Total solids (% moisture)  | Work Instruction for Percent Moisture and Density Measurement for the GAAT Sludges, dated June 25, 1997. |

If needed, samples will be transported in a shielded carrier to a high-radiation-level hot cell, if external beta/gamma radiation levels exceed 200 mrem/hour. If beta/gamma radiation levels are less than 500 mrem/hour, samples will be transported to radiochemical laboratories that are contamination areas at RMAL. Transportation will be coordinated with Waste Management.

#### **4. ANALYSIS PROCEDURES FOR SAMPLES**

Figure 1 shows the anticipated analysis scheme that may be used to characterize the slurry samples. The sample that will be submitted to the laboratory is assumed to be slurry of liquid mixed with sludge that is being transferred through the WD&C. The samples will be removed from the sampling container and transferred to a hot cell or radiochemical hood for mixing. Before any sample treatment, the samples will be removed from their containment, photographed, and videoed and a physical description of the sample (color and consistency) will be documented. Upon conclusion of the analysis, remaining sample material will be archived for possible future characterization. This is the flow diagram depicting the overall analysis scheme for the samples retrieved with the Isolock Sampler at the Gunite and Associated Tanks. As soon as the samples are transported to the RMAL, this process will begin.

Concentrations of radionuclides will be determined on a 0.5-g (0.175-oz.) aliquot of sample. Aliquots of the digested sample will be taken for radionuclide analysis. Radionuclides will be analyzed using the ORNL procedures.

DOE-ORNL, WAG 1 Gunitite and Associated Tanks, Oak Ridge, Tennessee  
Drawing Date April 7, 1998

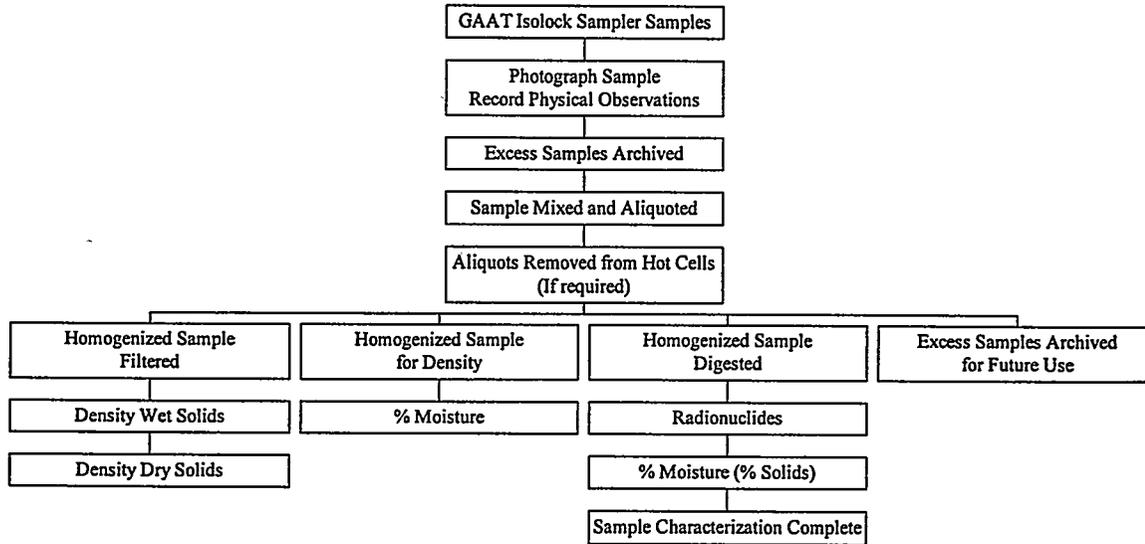


Fig. 1. Overall analysis scheme for the GAAT Isolock Sampler.

## 5. QUALITY CONTROL

Ordinarily, trip blanks are submitted with each shipment of samples to the laboratory. However, because only three composite samples per tank will be collected over an extended period of time and submitted to the laboratory, no trip blanks will be submitted to RMAL.

The quality control acceptance criteria for EPA methods from SW-846 may not always be applicable or practicable for high-radioactivity samples. Modifying the procedures may be necessary for as low as reasonably achievable considerations. All deviations and dilutions and the reason for the deviations and dilutions will be documented and reported by RMAL. Error propagation will be documented and reported.

All information generated during laboratory analysis will be filed in request folders, which will be retained by the laboratory. Copies will be made available for the tank project files. Documentation to be filed in the request folder for each analysis will include the following:

- sample identity;
- number and title of the standard procedure;
- detailed, step-by-step description of any deviation from the standard procedures;
- rationale for any procedural deviation;

- results of any analysis, including results of percentage recovery of each spike and percentage difference of each set of duplicates; and
- observations.

Review of data packages will be conducted using the laboratory's sample management standard operating procedures (CASD-OP-RML-AD02). This procedure includes security measures and the initiation and use of chain-of-custody forms.

Instrument logbooks will be maintained to record periodic balance and instrument maintenance. Daily instrument and balance checks will be recorded in these logbooks. Calibration data associated with specific analyses for this project will be filed in the analytical request folders.

Information pertaining to calibration standards, spiking standards, and reagents used will be retained in the logbooks. This information will include manufacturer, lot number, and expiration dates (if any) of stocks, dates prepared, and names of persons who prepared dilutions or dissolutions. At the completion of the project, all results and information of deviations from EPA SW-846 or ORNL analytical chemistry procedures will be compiled into a report. A discussion of the probable error for the analytical results will also be included in the report.

## **6. DATA MANAGEMENT**

Sampling data will be managed and validated in accordance with the GAAT Data Management Plan. The data will be compiled into a report and issued to team members.