

Standard Guide for Sampling Radioactive Tank Waste¹

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1. Scope

1.1 This guide addresses techniques used to obtain grab samples from tanks containing high-level radioactive waste created during the reprocessing of spent nuclear fuels. 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) (1).² Vapor sampling of the head-space is not included in this guide because it does not significantly affect slurry retrieval, pipeline transport, plugging, or mixing.

1.2 The values stated in inch-pound units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1 *ASTM Standards*:³ D1129 Terminology Relating to Water

3. Terminology

3.1 *Definitions*—For definitions of terms used in this method, refer to Terminology D1129.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *forced evaporation, n*—intentional concentration of a waste solution using heat or vacuum, or both, primarily to remove water or other solvents.

3.2.2 *pH modified*, *n*—a description of a solution where the pH is adjusted with either an acid or base material to achieve a desired pH level to minimize tank corrosion.

3.2.3 *soft sludge*, n—a sludge with a low viscosity where minimal sampling device pressure could be used to penetrate the sludge layer.

3.2.4 *sparge*, *n*—a process of delivering a chemically inert gas through fluids to displace materials for the purpose of mixing.

3.3 Acronyms:

3.3.1 *EREE*—Extended Reach End-Effector

3.3.2 HAST—Highly-Active Storage Tanks

3.3.3 LDUAs—Light-Duty Utility Arms

3.3.4 NPH—Normal Paraffin Hydrocarbons

3.3.5 ORNL-Oak Ridge National Laboratory

3.3.6 PTFE—Polytetrafluoroethylene

3.3.7 PVC—Polyvinyl Chloride

3.3.8 RFD-Reverse-Flow Diverter

4. Significance and Use

4.1 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 small due to the hazardous nature of the samples as well as their high radiation dose.

4.2 Many high-level wastes have been treated to remove strontium (Sr) or cesium (Cs), or both, underwent liquid volume reductions through forced evaporation or have been pH modified, or both, to decrease corrosion of the tanks. These processes, as well as waste streams added from multiple process plant operations, often resulted in precipitation, and produced multiphase wastes that are heterogeneous. Evaporation of water from waste with significant dissolved salts concentrations has occurred in some tanks due to the high heat load associated with the high-level waste and by intentional evaporative processing, resulting in the formation of a saltcake

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 $^{^{2}}$ The boldface numbers in parentheses refer to a list of references at the end of this standard.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

or crusts, or both. Organic layers exist in some waste tanks, creating additional heterogeneity in the wastes.

4.3 Due to these extraordinary challenges, substantial effort in research and development has been expended to develop techniques to provide grab 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 is provided in Table 1. These techniques will be summarized in this guideline with the assumption that the tank headspace is adequately ventilated during sampling.

5. Liquid-Only Sampling Techniques

5.1 Liquid only techniques are not common in tank waste sampling. More common are liquid samples captured by methods used primarily to obtain solid or slurry samples. However, some high-level waste tanks, such as the Highly-Active Storage Tanks (HAST) tanks at Sellafield in the United Kingdom, 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 HAST system design also allows for the agitation of tank contents to help obtain representative liquid samples (2).

6. Slurry/Liquid Sampling Techniques

6.1 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 decrease sampling bias.

6.2 A dip sample is taken by lowering a stoppered and weighted bottle into the waste to the desired depth. After the

TABLE 1 High-Level Waste Tank Sampling Methods				
Technique	Solid	Material Type Slurry	Liquid	Notes
HAST in- tank needle orifice	Cond	Oldity	X	Orifice as part of Reverse-Flow Diverter (RFD)
Bottle on a String		Х	Х	Dip sample
Vacuum Pump		Х	Х	
Auger	Х	Х		Only high viscosity slurries
Sample Cup	x	X		Manual system used at Savannah River Site to obtain salt-cake samples and hard sludges that don't slump.
Core Drilling — Rotary Mode (Hanford Sampler)	Х	x		Hard sludges and salt-cake.
Core Drilling — Push Mode (ORNL Soft Sludge Sampler)		Х	Х	Liquid or soft sludges.
Cylinder with retractable nose cone		Х	Х	Used at Savannah River Site for soft sludges and liquids.
Robotic Arm	х	Х	х	Material type captured is dependent upon the end- effector.
Hydraulic Mining	Х	Х	Х	0.0000
Hydraulic Scoop	х	Х	х	
Sample Thief (Bacon Bomb)			х	

bottle has reached the desired level, the stopper is pulled from the bottle and the liquid or slurry sample flows into the bottle. Ideally, the stopper is then closed and the bottle is pulled from the tank (3).

6.3 Dip sampling is limited to lower viscosity liquid and slurry materials and the effectiveness of sampling is highly dependent upon the size of the sample bottle inlet and the presence of saltcake layers which may prevent sampling access to lower tank levels. Further, sampling locations are limited only to vertical columns directly under a tank penetration, or riser. Particulates obtained from this method may be highly biased due to sample location and variations in settling velocity while sampling.

6.4 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 polytetrafluoroethylene (PTFE) 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 PTFE tubing to keep it vertical. A diagram of the vacuum-pump sampling system used at Oak Ridge National Laboratory (ORNL) is provided in Fig. 1 (4).

7. Solids/Slurry Sampling Techniques

7.1 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 in. This 6 in. limitation was driven primarily by a desire to reduce radiation dose. Some homogenization of the sample occurs while obtaining auger samples. These samples can only be taken directly beneath a penetration, or riser (5).

7.2 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.

7.3 Savannah River Site staff 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 Fig. 2 (6). The applicability of this method is limited to hard materials that will not flow or slump once collected in the sample device.

7.4 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

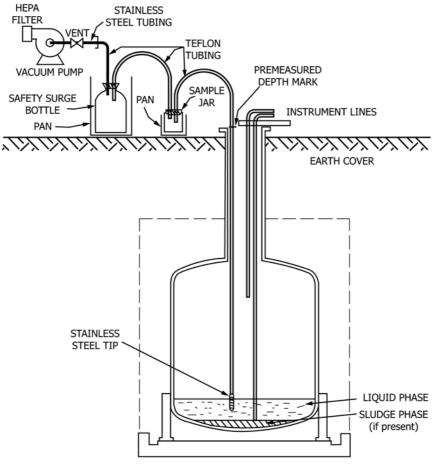


FIG. 1 Vacuum Pump Sampling System

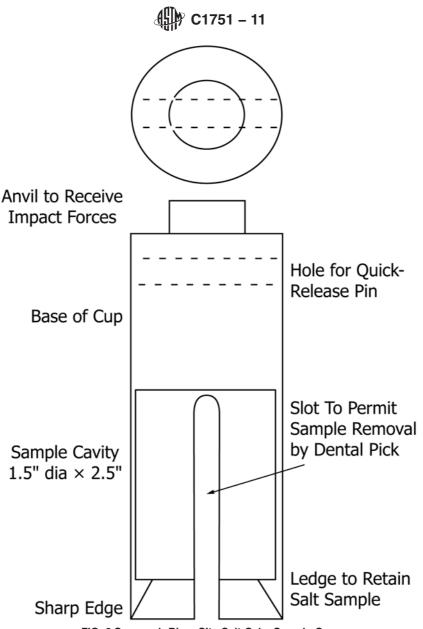


FIG. 2 Savannah River Site Salt-Cake Sample Cup

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.

7.5 The Hanford Sampler is based on a modified coredrilling 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 (7), (8). Liquid and solid samples are trapped in the sampler by a spring-actuated rotary valve (see Fig. 3). Two different sampler designs have been used, but both designs incorporated the spring-actuated rotary valve. The first design produced samples that were 19 in. long and 1 in. in diameter. The later design had a slightly larger diameter (1.25 in.). It is important to note the design length of this sampler was driven by operational space limitations of the existing hot cells at the time. Core samples can be taken at varying depths to obtain samples that comprise the entire depth of the waste. A sliding piston in the sampler controls the height of the sample being collected. 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. Nitrogen gas has also been used.

7.6 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 Fig. 4. 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 (4).

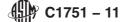
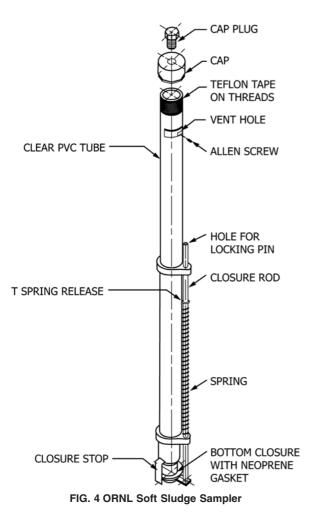




FIG. 3 Hanford Core Sampler



7.7 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 (9).

8. Other Sampling Techniques

8.1 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 risers. The LDUAs 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 lbs and can capture both liquids and solids (10).

8.2 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.

8.3 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 (6).

8.4 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. Mechanically controlled scoops are selected when the introduction of organic-based hydraulic fluids are not allowed in the tank.

8.5 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.

9. Sampling-Plan Design

9.1 As discussed in Section 4, 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.

9.2 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.

9.3 Locations for sampling may be selected randomly, deliberately chosen to represent the range of conditions observed in the field or unusual conditions of particular interest, or simply limited by the location of tank access ports. Randomly selected locations are appropriate for overall assessments of site conditions, in wastes where variations are random (for example, 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.

9.4 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 (**11**).

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 variability of the analytical methods. This heterogeneity may be due to any combination of the following factors: the nature of the solids, stratification caused by treatment in the tank, changes in the waste streams as the tank was filled, crystallization, mixing, temperature gradients, 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. Pierre Gy's theory offers an approach to minimize error in sampling particulate solids (12), (13).

10. Keywords

10.1 core sampling; high-level waste; radioactive waste; sampling tank waste; slurry sampling

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