Key Elements of Characterizing Savannah River Site (SRS) High Level Waste (HLW) Sludge Insolubles through Sampling and Analysis

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Abstract

Characterization of HLW is a prerequisite for effective planning of HLW disposition and site closure performance assessment activities. Adequate characterization typically requires application of a combination of data sources, including process knowledge, theoretical relationships, and real-waste analytical data. Consistently obtaining high quality real-waste analytical data is a challenge, particularly for HLW sludge insolubles, due to the inherent complexities associated with matrix heterogeneities, sampling access limitations, radiological constraints, analyte loss mechanisms, and analyte measurement interferences. Understanding how each of these complexities affects the analytical results is the first step to developing a sampling and analysis program that provides characterization data that are both meaningful and adequate. A summary of the key elements impacting SRS HLW sludge analytical data uncertainties is presented in this paper, along with guidelines for managing each of the impacts. The particular elements addressed include: a) sample representativeness; b) solid/liquid phase quantification effectiveness; c) solids dissolution effectiveness; d) analyte cross-contamination, loss, and tracking; e) dilution requirements; f) interference removal; g) analyte measurement technique; and h) analytical detection limit constraints. A primary goal of understanding these elements is to provide a basis for quantifying total propagated data uncertainty.

Introduction

Activities being performed to plan disposition of HLW and site closure performance assessment rely on effective waste characterization data. Such data are typically developed through a combination of sources, including accountability records, waste transfer records, in-situ physical measurements, constituent solubility expectations, application of radionuclide fission product yields, and laboratory data generated through analysis of real-waste samples. Because of the complexities related to waste variations, sampling access limits, radiological constraints, and analytical issues, consistent generation of useful, high quality real-waste analytical data is an ongoing challenge. This is particularly true for HLW sludge insolubles, due to their tendency to be heterogeneous based on changing waste inputs, temporal settling effects, and resistance to mixing by virtue of their solid form. Effective characterization of HLW sludge is important, because the sludge phase contains the primary long-term risk drivers, namely the long-lived alpha-emitting radioisotopes of plutonium and americium. Developing an understanding of how the characteristics of HLW sludge affect the analytical

requirements and resulting data is important when establishing a sampling and analysis program. The goal of this paper is to provide an awareness of the key elements impacting HLW sludge analytical data and to provide bases for understanding the primary sources of analytical data uncertainty.

Specific Objectives

1) Identify complexities of key elements affecting HLW sludge analytical data uncertainties. The key elements include:
   - Sample representativeness
   - Solid/liquid phase quantification
   - Solids dissolution
   - Analyte contamination and loss
   - Dilution requirements
   - Removal of interferences
   - Measurement techniques
   - Detection limit constraints
2) Provide guidelines for addressing complexities.
3) Address data uncertainties and identification of primary uncertainty drivers.

Discussion

Sample Representativeness

Obtaining samples that are representative of waste in storage tanks is important if the purpose of sampling and analysis is to draw quantitative conclusions about waste composition. Acquiring representative samples would be straightforward if the waste was totally homogeneous and/or if the number and size of samples collected were unlimited. However, in practice, HLW sludge matrices are typically heterogeneous with respect to both physical and chemical properties and subject to severe sampling limitations.

HLW sludge matrices are heterogeneous for several reasons. Material in a given storage tank has typically been accumulated over a long period of time and in the absence of mixing. Often the material is a conglomeration of multiple sources of waste. The net result is that the contents of a given waste tank are often stratified, due to a combination of waste differences, settling conditions, and temporal effects. Sludge at the bottom of a tank is normally very compact (high solids content), because of the long relative settling time (due to its greater age) and the higher static pressure imposed by the mass of overlying waste. In contrast, the top layer of sludge is generally less compact, because of the shorter settling time (due to the being more recently received) and the lower static pressure. The mineralogy of the material at the bottom of the tank is also expected to differ from that at the top, due to the difference in equilibration times affecting crystallization.

Limitations associated with HLW sludge sampling are severe for many reasons. Managing radiation dose is a necessity, both in the field during sampling activities and transport, and at the laboratory. Because of the high potential dose associated with HLW

sludge, constraints of sampling access, sample size, and number of samples are "a given." Consequently, the majority of sludge sampling events require dependence on remote sampling devices, which are prone to operational limitations. SRS HLW tanks were built for the purpose of storing large volumes of waste over an extended period of time, with the objectives of being structurally sound and capable of effectively containing the waste. These characteristics do not necessarily lend themselves to sampling convenience and flexibility. Most of the HLW tanks have a capacity of approximately one million gallons and often only one to two waste access points. With this small number of access points, it is very difficult to collect samples from most sites within the tank. Furthermore, the maximum practical sample size (several liters) is small compared to the total volume of waste (hundreds of kgal).

If one assumes that each batch of waste received into a tank is relatively homogeneous and the sludge in a given batch settles in a distinct layer, there is the possibility that lateral variations would be smaller than vertical variations. If this is the case, sampling of vertical waste "cores" could provide an acceptable basis for understanding compositional variations, in lieu of abundant lateral samples. The alternative is to sample the waste following/during effective mixing of the material. However, mixing of the waste is normally not practical until just prior to removing the waste from the tank, which is later than desired from a planning perspective.

Regardless of the sampling protocol, it is imperative to collect multiple samples (from different locations, if possible) to provide a basis for identifying the magnitude of compositional variations.

Guidelines

- Homogenize sludge via in-tank mixers during sampling or just prior to sampling, if possible
- If mixing is not possible, collect full length vertical core samples (from top to bottom of sludge layer), to assure sludge strata from all depths are sampled
- If full length core sampling is not possible, consider collecting grab samples from random locations in the tank, but recognize that grab samples may not be representative

Solid/Liquid Phase Quantification

One goal of characterizing sludge at SRS is to quantify the mass of insolubles that will be incorporated into glass at the Defense Waste Processing Facility (DWPF). This mass has a direct effect on the number of glass canisters that will be produced, which in turn has a direct effect on DWPF life cycle costs and schedule.

In order to project inventories of sludge insolubles, three key pieces of information are necessary. The first is data identifying the total volume of sludge in a tank. This is typically acquired using physical in-tank measurements of sludge depth coupled with tank geometry information. The second is data quantifying the composition of insolubles in the sludge. This is typically acquired through a combination of process knowledge and real waste analytical data. The third is data quantifying the solid/liquid

(S/L) phase distribution of the waste. This is typically acquired in the laboratory through physical measurements of real waste samples and becomes the primary basis for converting insoluble constituent concentrations to insoluble constituent inventories. Because S/L distribution plays such a key role in projecting mass of insolubles, it is critically important that the distribution be identified accurately.

Accurate quantification of S/L distribution is a challenge for many reasons. One of these is the presence of matrix heterogeneities, which introduce large variations between replicate samples and impede the practicality of obtaining representative samples. Another is due to temperature differences between the conditions of the waste in the tank (~30 C), the conditions during transport and storage (0 – 40 C, depending on the season), and the conditions of the laboratory environment (~ 23 C). These temperature differences affect the constituent solubilities and could have a significant potential impact on the S/L distribution measurement.

Another challenge associated with quantification of the S/L distribution is tied to laboratory sample size limitations and use of remote manipulators, both associated with radiation dose issues. S/L phase measurements are typically performed on aliquot volumes of 100 mL or less. In this volume range, sample phase losses due to adherence to sample container walls, phase separation methodology, transfer/measurement equipment, or volatilization could have a significant impact on the final measurement result. In addition, constraints associated with manipulators have the potential for reducing sample handling control and visibility, which could have a detrimental effect on results.

Proven measurement techniques and accountability are required when performing S/L distribution measurements, because of the large potential for skewing results high or low and the high impact that skewing has on projecting insoluble mass projections. The goal is to produce results that 1) are consistent with theoretical expectations based on the composition of the sludge and 2) allow discernment of the relative uncertainty of the result. This will be necessary to effectively project anticipated insoluble mass inventories, along with defendable lower and upper bounds.

Guidelines

- Perform phase determinations on the largest aliquot volumes and largest number of independent samples practical
- Minimize temperature differences between field and lab conditions and minimize sample storage times prior to laboratory measurements to the greatest extent practical
- Compare laboratory results with theoretical expectations based on chemical and physical characteristics
- Record details of laboratory methods, conditions, and observations, for purposes of comparison
- Incorporate uncertainties into insoluble inventory projections

Solids Dissolution

Insoluble solids in HLW sludge are those solids that have low solubilities in neutral to alkaline aqueous solutions. Most of the insoluble solids in HLW sludge are comprised of hydroxides and oxides of metals such as iron, aluminum, uranium, and manganese. Prior to performing constituent analyses, the insoluble solids need to be dissolved into an aqueous solution, to make their form consistent with the needs of the laboratory methods.

Several different solids dissolution methods are available. These methods typically involve use of aggressive conditions to assure dissolution proceeds to completion (or near-completion). Use of strong acids, oxidizing agents, and/or flux forming agents is usually required to achieve adequate dissolution. Additionally, conditions of elevated temperature and pressure may be required.

Dissolution of solids presents several challenges because the conditions necessary to dissolve one constituent may cause loss or prevent dissolution of another constituent. For example, the extreme temperature conditions needed to dissolve many silicates and mineral oxides could result in loss of potentially-volatile constituents such as manganese, mercury, and tin. In contrast, the conditions necessary to dissolve and retain the volatile constituents would in some cases be insufficient to dissolve other constituents.

Because of the different behaviors of the different chemical constituents, use of more than one dissolution method is often required. Typically, this would include at least one method performed at slightly elevated temperatures/pressures and at least one method performed at extremely elevated temperatures. An example of the former method is one utilizing aqua regia (a 3:1 mix of concentrated hydrochloric acid and nitric acid), performed in a pressure vessel at approximately 70 C, and then allowed to cool to ambient temperatures before retrieving the solution. This method provides an effective means of solubilizing many potentially-volatile constituents of importance, including mercury and radioisotopes of selenium, technetium, and iodine.

An example of a method for dissolving non-volatile refractory insolubles is one utilizing a sodium peroxide fusion reaction, performed at approximately 1000 C, under ambient pressure conditions. After cooling, the solids produced through the fusion reaction are transferred to the aqueous phase through nitric acid dissolution. This method provides an effective means of solubilizing many acid-insoluble alloys of metals such as iron, nickel, and chromium.

Dissolution testing is needed to demonstrate that a given dissolution method is acceptable for a given constituent. For a dissolution method to be deemed acceptable, three primary criteria must be met: 1) the method results in complete dissolution or near-complete dissolution of the constituent of interest; and 2) the method results in negligible loss of the contaminant of interest; and 3) interferences from constituents associated with the dissolution reagents and equipment must be negligible or manageable.

Guidance:

- Choose/develop dissolution methods targeting the constituents of interest
- Perform dissolution tests to determine if the methods are acceptable

- Perform sample dissolutions using replicates and multiple methods, when possible, to provide bases for comparing results
- Incorporate standards/tracers into dissolutions, as possible, to quantify constituent dissolution efficacies and yields

Analyte Contamination and Loss

Samples processed in the laboratory are subject to both cross-contamination and losses. Cross-contamination occurs when constituents from an external source are inadvertently introduced into a sample, increasing the quantity of constituents in the sample. Potential sources of cross-contamination include contaminated laboratory equipment, other samples undergoing processing in the same vicinity at the same time, and/or "sloppy" work practices. Depending on the magnitude of the cross-contamination, the impact to samples could be negligible or significant. The net result of cross-contamination is that it biases constituent concentrations high.

In contrast to cross-contamination, losses reduce the quantity of constituents associated with a sample and therefore bias constituent concentrations low. Losses occur due to various physical and chemical processes, some of which are related to "sloppy" work practices. Physical sources of losses include adherence to laboratory ware, spillage, and spattering. Chemical sources of losses include incomplete reactions (incomplete solids dissolution and/or incomplete constituent separations), sorption, volatilization, and/or precipitation. As in the case of cross-contamination, the magnitude of the losses could be negligible or significant.

Contamination and loss effects must be understood and quantified in order to choose the appropriate laboratory protocols to negate their effects. Use of "blanks" is an established laboratory practice for identifying cross-contamination. "Blanks" are simulated samples known to be free of the constituents of interest. "Blanks" are processed and analyzed alongside of the real samples, and provide a means for gauging whether constituents have been introduced into the samples via cross-contamination. If the "blanks" are found to be free of the constituents of interest, there is a basis for concluding that cross-contamination was negligible. In contrast, if the "blanks" are found to contain the constituents of interest, there is a basis for concluding that cross-contamination was impactive and a means for quantifying the extent of the cross-contamination. Potential means of overcoming cross-contamination impacts include use of larger sample aliquots or less sample dilution, with the end goal of making the contributions from cross-contamination small compared to the constituent concentrations in the sample.

Quantification of losses can often be accomplished through use of constituent-specific tracers and/or standard additions. These materials are added to the sample at the start of the laboratory processing and provide a means for identifying the quantity of the constituent that remains at the time of the analysis. This approach can be utilized provided: 1) an appropriate tracer or standard is available for the constituent of interest; 2) the amount of tracer or standard added to the sample is appropriate to allow quantification of the recovery; and 3) the purity of the tracer or standard is such that it does not compromise quantification of other constituents of interest; and 4) traceability of the tracer or standard is maintained. In the absence of appropriate tracers/standards, other

more labor-intensive approaches may be used to track constituent losses. Analysis of a progressive range of sample sizes (serial dilutions) offers one potential approach. Note that losses associated with some constituents are more difficult to track than others, depending on the relative abundance of the constituent, the analytical demands, and the accessibility of appropriate tracers and standards. Regardless of the tracking approach, once contaminant loss is quantified, analytical results can be adjusted to take the losses into account.

Guidelines:

- Hone lab processing protocols to minimize potential contamination and losses
- Quantify contamination and losses associated with constituents of interest
- Adjust experimental conditions, as necessary, to overcome cross-contamination and loss issues
- Do not report analytical data where issues remain unresolved

Dilution Requirements

Prior to analysis, HLW samples typically require dilution (following dissolution) for the purposes of: 1) reducing radiation dose; 2) adjusting constituent concentrations so they fall in the proper detection ranges; and 3) providing sufficient volumes of material to accommodate all required analyses. Dilution requirements are a function of the sample composition and the aliquot size. Samples containing high concentrations of dose drivers will typically require more dilution than samples containing low concentrations, assuming similar dose implications. A minimum aliquot size will be necessary to facilitate laboratory handling and processing. However, if the radiological content of the sample is low, a larger aliquot size may be required to quantify the radiological constituents. Such an increase in aliquot size could impact the dilution requirements.

Most analytical measurement techniques have clearly defined detection ranges, bounded by minimum and maximum detection limits. Very high constituent concentrations typically require dilution to fall below the maximum detection limit, whereas very low constituent concentrations may already be below the minimum detection limit, even in the absence of dilution. Clearly, in cases where high analytical sensitivity is required, unnecessary dilution should be avoided. Additionally, dilution makes analytical measurements more prone to cross-contamination issues, due to lowering of the constituent concentrations, as well as introducing another potential source of contamination, the diluent solution.

Although dilution is a relatively simple process, it is one that contributes additional uncertainty to the analysis. Part of this uncertainty is routine – the part due to the uncertainty of the diluent volume measurements and the assumption that mixing of the diluent with the sample digest solution is complete. The other part is less routine – the part associated with changing dilution protocols from what is considered "standard" (for example, if the normal lab protocol is a 1000:1 dilution and the protocol is changed to a 100:1 dilution, there is an increased potential for a data processing error).

Guidelines:

- Use dose measurements and process knowledge to determine extent of dilution that is needed
- Do not dilute the sample beyond what is needed to meet routine dose limits and to provide sufficient material to accommodate all required analyses
- Consider larger sample aliquots and less dilution for constituents subject to analytical sensitivity constraints or cross-contamination issues

Removal of Interferences

HLW sludge is a complex matrix, comprised of an extremely large number of stable and radioactive constituents. Because analytical measurements are based on properties that are not entirely unique to each constituent, there is a need to remove those constituents that interfere with a given analysis, prior to the analysis. Otherwise, the analytical result will be biased by the presence of the interfering constituent.

Removal of interferences is accomplished through physical, thermal, and/or chemical processes dependent on the properties of the interfering constituent(s) in question. Physical removal processes are typically based on differences in size, mass, and volume. Examples of such processes include sieving, centrifugation, and filtration. Thermal processes are based on differences in melting points, boiling points, combustion energy, and enthalpy. High temperature ashing for removal of organics is one example. Chemical processes are based on electron configuration and molecule size. Examples include oxidation, reduction, precipitation, extraction, ion exchange, and sorption.

Established methods for removing interferences from many common sample matrices are well documented in the open literature. Methods for removing interferences from HLW sludge insolubles are less well documented and are dependent on sludge composition, which can vary from site to site and from tank to tank. In most cases, the established methods will need to be honed/revised and validated in order to be applied to HLW sludge matrices. Effective validation of these methods will typically require laboratory tests performed on HLW sludge matrices, with sufficient quality assurance measures to demonstrate analytical effectiveness. Such quality assurance measures include standard protocols such as use of blanks, tracers/standards, serial dilutions, replicates, and interlab comparisons.

Guidelines:

- Hone/validate interference removal methods for HLW sludge matrices
- Use sufficient quality assurance measures to demonstrate method effectiveness
- Revise methods as necessary to overcome newly identified interference issues

Measurement Techniques

Quantification of the constituents of interest in HLW sludge requires use of a combination of measurement techniques capable of meeting the analytical objectives. These needs include considerations of accuracy, convenience, expense, and throughput,

as well as issues related to available laboratory equipment and space. Unfortunately, no single measurement technique has the capability of quantifying all constituents. This is due to the wide range of differences between the properties used to quantify the various constituents. Examples of these properties include molecular mass, molecular charge, molecular size, absorption/emission characteristics, and radiological characteristics. Furthermore, each technique has inherent strengths and weaknesses, so identification of the "best" available technique is often subjective.

Because of the large number of considerations affecting selection of measurement techniques, it is clear that analytical objectives and constraints must be clearly understood before reaching a decision. Regardless of which techniques are selected, laboratory personnel must expect that measurement problems requiring resolution will routinely be encountered, and that effective quality control measures will be necessary to recognize the problems, as well as demonstrate measurement effectiveness.

Measurement Techniques Routinely Used for Quantifying Key HLW Sludge Constituents:

Inductively Coupled Plasma Emission Spectroscopy
Inductively Coupled Plasma Mass Spectroscopy
Atomic Absorption Spectroscopy
Alpha Spectroscopy
Gamma Spectroscopy
Liquid Scintillation Counting or Gas Proportional Counting

Guidelines:

- Choose appropriate measurement techniques based on an understanding of analytical objectives and constraints
- Consistently use quality control measures to identify measurement problems and to demonstrate measurement effectiveness
- Resolve measurement problems as they are identified

Detection Limit Constraints

Nearly all instrumental measurement techniques have lower and upper detection limits bounding the concentration ranges over which analytical measurements are expected to be effective. Uncertainties of the measurements are typically lower for concentrations falling in the middle of the acceptable measurement range rather than those falling within the range but approaching the lower or upper bounds. For these reasons, it is useful to estimate constituent quantities prior to performing analyses, so sample aliquot size and dilution protocols can be chosen appropriately. Otherwise, the analytical results may not meet the data needs.

In order to estimate the constituent quantities prior to the laboratory analyses, an adequate understanding of the HLW source material is required. Such understanding can be developed from a) waste receipt accountability records combined with process knowledge and theoretical constituent relationships and/or b) existing analytical data for other similar HLW. Development of an understanding of the source material is typically

labor intensive, requiring both significant lead time and sizable resource demands. However, in the absence of such an understanding, determination of the appropriate laboratory protocols is subject to guesswork and therefore expected to be iterative.

Because each measurement technique has its own inherent detection limit constraints and multiple measurement techniques are typically required to meet analytical needs, it may not be possible to choose one aliquot size/dilution protocol that fits all analyses. Confounding this situation are two other factors, sample size limitations related to radiation dose and differences in constituent recoveries. Nonetheless, available information should be used to best advantageous, when possible, to maximize the chance that sample protocols will yield effective measurements.

Guidelines:

- Identify measurement detection limits and optimal concentration ranges
- Estimate waste constituent concentrations
- Choose sample aliquot size and dilution protocols to support effective measurements
- Iterate as necessary to produce acceptable analytical results

Propagated Data Uncertainty

In order to effectively utilize analytical data as a basis for characterization, the limitations and uncertainties of the data must be identified and understood. This means quantifying the total propagated uncertainties associated with the data, as well as the individual uncertainties associated with each element of the sampling and analysis process. Such quantification is vitally important for two reasons: 1) it provides a basis for projecting the overall uncertainty of a given characterization; and 2) it provides a basis for identifying which elements of the process drive the overall uncertainty, and whether changes in the sampling and analysis protocols are warranted in future characterization activities. The end goal of quantifying the uncertainties is to assure that the analytical data are sufficient to meet the characterization needs.

In the case of HLW sludge insolubles, each of the key elements addressed in this document has a significant potential impact on the overall data uncertainty. Within this context, a discussion of the primary considerations associated with each of the key elements is summarized below. Note that uncertainties fall into two general categories, random uncertainties and determinate uncertainties. The recommended approach for estimating the random uncertainties is the same for all key elements – through determination of standard deviations between results of multiple sample analyses (analyses performed on distinctly different samples) or replicate sample analyses (analyses performed different aliquots of the same sample). In contrast, the recommended approach for estimating determinate uncertainties is through: a) application of known deviations based on calibration data (this applies to traceable standard materials or routine physical measurement tools such as pipettes, graduates, and balances); b) determination of deviations between analytical results and standard values (this applies to analyses performed on traceable standards); c) determinations of deviations between analytical results of independent analytical results. Formulae for

computing total propagated uncertainties, while not presented in this document, are available in many sources in the open literature.

The impact of sample representativeness is highly dependent on whether the waste is homogeneous or heterogeneous. If the sludge is relatively homogenous (due to consistent waste receipt conditions and/or adequate mixing conditions), the likelihood of collecting representative samples is relatively high and the number of samples required to yield modest uncertainties is relatively low. In contrast, if the sludge is relatively heterogeneous (due to inconsistent waste receipt/settling conditions, in the absence of mixing), the likelihood of collecting representative samples is relatively low and the number of samples required to yield modest uncertainties is relatively high. In the case of relatively homogeneous sludge, sample representativeness may not be the primary element driving the overall uncertainty. However, in case of relatively heterogeneous sludge, it is very likely that sample representativeness (or lack of representativeness) would be a primary driver of overall uncertainty.

A common means of quantifying the impact of sample representativeness is through analysis of multiple samples, collected from distinctly different locations from within a given waste tank. Specifically, this means collecting samples from various depths and various lateral positions. Deviations between the results of the various samples provide a measure of the combined effects of sampling uncertainties and analytical uncertainties. As such, it is necessary to understand the magnitude of the analytical uncertainties to discern the relative contribution due to sampling.

Uncertainties associated with solid/liquid phase distributions can also be significant, due to a) thermal and temporal effects which can alter the characteristics of the sludge and b) the inherent physical measurement difficulties associated with high solids content samples. Quantification of these uncertainties can best be achieved through a combination of replicate measurements (measurements on "identical" sample aliquots) and use of multiple solids/liquid quantification techniques (for example, centrifugation and filtration).

Uncertainties associated with solids dissolution methods are highly variable, due to the differences between the dissolution properties of the various constituents and the various matrix effects. Effective means of monitoring these uncertainties is through use of appropriate matrix standards and/or simulants processed through the applicable dissolution method and analyzed for the constituents of interest.

Analyte contamination and loss are highly contaminant-specific and method-specific. As such, uncertainties associated with these elements are also highly contaminant-specific and method-specific. An effective means of quantifying these uncertainties is through deviations between a) "blanks," for contamination issues and b) standard recoveries, for loss issues.

Uncertainties associated with sample dilution are typically minor, assuming that dilution attributes are accurately recorded and accounted for. However, in cases where dilution protocols are different than the norm, there is an increased chance of incorrectly accounting for the dilution. Routine use of normal quality assurance practices offers one means of minimizing the chance of such an error, should it occur.

Interferences removal processes can have a significant impact on uncertainties, particularly when sample compositions are outside of the norm and analyses are performed in the absence of appropriate yield tracers or standards. Under these

conditions, it is possible than constituent yields will be much less than expected and unidentified. The best means of quantifying such uncertainties is through use of tracers or standards, which provide a direct measure of constituent removal.

Uncertainties associated with a given measurement technique can be potentially significant, particularly if a) the chosen measurement technique is not optimized for the constituent of interest, b) an interference is present, or c) if the constituent quantity approaches a detection limit or is outside of the detectable range. An effective means of quantifying these uncertainties is through replicate analysis of standards, preferably standards that are free of interferences.

Conclusions

1) Effective sampling and analysis of HLW sludge requires a clear understanding of the data objectives, waste expectations, and analytical processes prior to the start of the characterization activity.

2) Recognition of potential issues/uncertainties related to all key elements of the sampling and analysis process will facilitate characterization efforts and provide a basis for optimizing analytical work.

3) Identification of overall data uncertainties is an integral part of the characterization process.