Consensus Standardization of Slurry Simulant Development Process to Reduce Design Risk Within the DOE Complex – 14635

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ABSTRACT

A critical responsibility of DOE's Office of Environmental Management is the design, construction, and operation of equipment and facilities to process legacy radioactive waste slurries for safe, long-term disposal. The accomplishment of this mission requires the extensive use of physical and chemical simulants to represent the hazardous radioactive waste. Development and characterization of simulants can often be difficult to compare or utilize as questions exist as to the basis, techniques, or consistency associated with reported parameters. In addition, controversy has existed in the interpretation and application of the characterization data for the wastes that the simulants are intended to represent.

In January 2008, DOE's Office of Engineering and Technology, Office of Environmental Management (EM) sponsored a workshop on slurry handling, mobilization, retrieval, transport, mixing, and processing for the purpose of identifying technical vulnerabilities, reducing program risks, understanding and disseminating lessons learned as well as best practices, and determining areas for improvement. Following the workshop, a technical report was generated describing the workshop, recommendations, aspects of slurry-characterization methods, and design principles/practices for slurry mobilization and transport systems that are intended to allow for comparable results and system designs among DOE sites. The report contained follow-on efforts including a guideline for the development of simulants. A summary of the simulant development process is provided. Use of consensus material property measurements is essential to ensure comparable results from all actual and simulant-based tests conducted. Validation of waste simulant formulations, actual unit operations and integrated facility performance is critical if improvements in process prediction are to be achieved throughout the facility mission.

Development of consensus standards is compliant with federal law National Technology Transfer and Advancement Act of 1995 (Public Law 104-113). This act contains language on "Federal Participation in the Development and Use of Voluntary Standards." The act directs that "... all Federal agencies and departments shall use technical standards that are developed and adopted by voluntary consensus standards bodies, using such technical standards as a means to carry out policy objectives or activities determined by the agencies and departments." The federal law provided motivation for the simulant guideline to be developed into the consensus standard ASTM International C1750 in 2011.

Standard Guide C1750 has been applied to tasks within the DOE complex that include the evaluation of processes and equipment, development and verification of instrument and system designs, and to address safety issues. These applications provide test cases that can be used to evaluate lessons learned for reviewing and updating the standard before the five-year required review. Additional recommendations and conclusions are provided.

INTRODUCTION

DOE's Task

A critical responsibility of the U.S. Department of Energy's (DOE) Office of Environmental Management (EM) is the design, construction, and operation of equipment and facilities to process legacy radioactive waste, currently stored in underground storage tanks, for long-term disposal. This effort requires the mobilization, retrieval, transport, mixing, and processing of diverse waste slurries and includes waste streams of both Newtonian and non-Newtonian mixtures. Such slurries (i.e., the presence of solids in waste streams with complex rheology) can severely reduce expected process throughput and yield, and increase startup time and required investment as they are much more difficult than handling gases or liquids. The entire inventory of waste to be processed has not been fully characterized. To accomplish DOE's mission, a wide range of work activities are required that include:

- Evaluating and studying the physical mechanisms and/or chemical reactions that will occur to gain a better understanding of the problem at hand. This effort requires analysis of data associated with the current waste and conducting experiments associated with waste aging, handling, and processing to obtain input for the development of processes and equipment design.
- Performing characterization of the waste, which includes remote sampling of the material.
- Developing processes, operations, and procedures to retrieve, mix, condition, and stabilize the waste.
- Designing and selecting equipment components, instrumentation, and systems to perform mobilization, transport, and mixing operations.
- Performing design, scale-up, verification and commissioning activities for physical systems and process flowsheets.
- Resolving safety issues and developing contingency plans associated with both normal operations and off-normal events.

Application of Simulants

The accomplishment of the above mentioned work activities include the use of physical and/or chemical simulants. "Simulants," for the purposes of this paper, are materials designed to emulate specific types of chemical or physical behavior of actual radioactive or hazardous wastes. Simulated wastes can be developed to exhibit a limited set of important properties for a specific testing requirement (e.g., only pertinent physical properties) or developed with a broader range of chemical and physical properties and rheological behavior for testing a unit operation where both slurry chemistry and physical properties are important. Development of simulants is usually a necessary requirement due to the difficulty of working with radioactive or hazardous wastes which include the large costs of procuring and disposing of large enough batches to complete necessary testing in addition to significant environmental, safety and health issues dealing with actual wastes. Actual waste tests are typically conducted at beaker- and bench-scale while pilot-scale tests require non-radioactive simulants. Lack of validated simulants introduces significant risk into scaling unit operations and validation of process flowsheets potentially leading to process failure.

As pointed out in the National Research Council (NRC) report, *Advice on the Department of Energy's Cleanup Technology Roadmap: Gaps and Bridges* [1], testing with a complete range of actual radioactive wastes to cover all process limits in regard to chemical and physical properties and rheological behavior would require too much actual waste that is difficult to retrieve and handle; increases radiological safety hazards to laboratory staff; and in most cases, is too time consuming and costly. Regardless, proper simulant development is essential to ensure that the critical radioactive process streams characteristics are represented.

Developing simulants to enable process flowsheet design verification encompasses the need to reduce the technical risks associated with: 1) using simulants as surrogates for waste for design of full-scale waste processing facilities; and, 2) effectiveness of simulants (versus real waste) in independent unit operations and the impact on integrated facility performance.

To increase confidence for process scale-up, it is necessary to ensure that simulants are designed to emulate the specific chemical or physical behaviors of actual radioactive wastes. Critical to the success of simulant development is the need to use standardized chemical, physical, and rheological measurement methods for both the actual and simulant wastes. Use of consensus material property measurements is essential to ensure comparable results from all actual and simulant-based tests. Validation of waste simulant formulations and actual unit operations and integrated facility performance is critical if improvements in process prediction are to be achieved throughout the facility mission. In addition, the use of nuclear waste material property measurement consensus standards that are easily accessible to designers and regulators (states, EPA, NRC, etc.) is needed to expedite the process of moving from the design stage to an actual working facility.

The benefit of developing robust waste simulants is the ability to “scale-up” waste processing operations with confidence. Errors in basic data are a common cause of failed new technology projects. For example, failure to accurately predict the flow properties of slurries is a leading cause of process failure (e.g. pipeline plugging). Integration of actual waste data to develop simulants will reduce risk and enable more accurate process flowsheet and unit operations design. A key benefit will be to eliminate overly conservative and costly process designs and reduce operational risks.

Past Issues

Prior to the workshop, simulant development efforts within the DOE complex were often project/program specific. These project-specific activities often used terminology and characterization techniques unique to a program. Past work related to the development and characterization of simulants can often be difficult to compare or utilize as questions exist as to the basis, techniques, or consistency associated with reported parameters. In addition, controversy has existed in the interpretation and application of the characterization data for the wastes that the simulants are intended to represent. This is compounded further when attempting to pedigree these results within an NQA-1 program.

The benefits of developing accurate and reliable waste slurry simulants are discussed in the NRC report [1]:

> “The absence of adequate understanding of the behavior of process streams can necessitate overly conservative and costly process designs to minimize the risk of a process failure or the risk of unrecognized safety issues, which as a worst case can render a facility inoperable with the actual radioactive waste it was intended to process.”

Need for Standardization

A need has existed within the DOE complex to establish standardized methods and terminology for identifying and performing characterization of waste and representative simulants. Standardized practices are required for the development and makeup of simulants that are applicable to the processes and mechanisms being evaluated to assure the necessary and sufficient properties and behavior are represented. Simulant compositions formulated to test specific sub-unit operations such as pumps, slurry transport piping or mixer configurations may be tailored to only the chemical and physical properties that are known to affect specific key operating/processing parameters. In addition, the scale-up of simulant make-up and the application of simulants to various physical scales should be standardized.

DEVELOPMENT OF SIMULANT GUIDELINES

DOE's Office of Engineering and Technology, Office of Environmental Management (EM) sponsored a workshop on slurry handling, mobilization, retrieval, transport, mixing, and processing for the purpose of identifying technical vulnerabilities, reducing program risks, understanding and disseminating lessons learned as well as best practices, and determining areas for improvement. The workshop was held in January 2008.

Pacific Northwest National Laboratory (PNNL), Savannah River National Laboratory (SRNL), Oak Ridge National Laboratory (ORNL), and Idaho National Laboratory ((INL) coordinated the workshop. Attendees were comprised of engineers and scientists that had experience working on tank waste slurry issues, experts from commercial slurry-processing companies, industry experts, and members of academia. Dr. Art Etchells, Retired DuPont Fellow, and Dr. Nigel Heywood, BHR Group, conducted short courses on *slurry mixing* and *slurry rheology and transport*, respectively. Additional, invited keynote speakers included Dr. Rick Bockrath, Dupont; Dr. David Gottslich, Independent Project Analysis, Inc., Prof. David Boger, University of Melbourne; and Dr. Robert Cooke, Patterson & Cooke.

The objectives of the workshop were to raise the level of awareness of slurry handling and processing by: 1) focusing on the risks associated with slurry retrieval, mobilization, transport, pipeline plugging, and stratification of slurries in vessels; 2) providing technical education and expert commentary on slurry handling practices; and, 3) to generate a technical report documenting slurry transport issues and best practices to include mobilization and transport technologies [2].

Key points and recommendations from the workshop related to simulant development for slurry systems included:

- The ability to design and field effective retrieval systems is dependent upon having and understanding the chemical and physical properties of the waste slurries.
- Waste retrieval equipment currently utilized has little ability to control properties of the resulting slurries that are conveyed out of the tanks.
- If the basic data (i.e. process stream material characteristics) for which slurry-system development is based are wrong, the design will be flawed, often in fundamental ways.
- Errors in basic data are the most common cause of failed new technology projects.
- Characterization of slurry properties is necessary to reduce technical risk.
- Large-scale testing of slurry handling equipment using simulants is essential.
- Use of standardized guidelines are valuable to ensure comparable results from all actual radioactive waste and simulant-based tests conducted within the DOE complex.

- An aggressive slurry physical and rheological characterization campaign of tank waste is needed for proper design of slurry transport, mixing, and processing systems.

Following the workshop, a technical report [2] was generated describing the workshop, recommendations, aspects of slurry-characterization methods, and design principles/practices for slurry mobilization and transport systems that are intended to allow for comparable results and system designs among DOE sites. In addition, three guidelines and a rheology primer recommended by the workshop were developed to ensure standardized, comparable results from all tests on simulated or actual waste slurries conducted across the DOE complex. The guidelines and primer were included in the workshop report [2] as appendices and consisted of 1) Sampling of Radioactive Wastes (Appendix B); 2) Performing Measurements of Chemical, Physical, and Rheological Properties (Appendix C); 3) Simulant Development, Approval, Validation, and Documentation (Appendix D); and 4) Slurry Rheology ((Appendix E).

Joel M. Tingey of PNNL initiated the guideline for sampling radioactive waste based on outcomes from the workshop, while the other three write ups used previously developed material as a starting point to formulate the guidelines. The sources of the material developed before the workshop are discussed below.

The starting point for the guideline to measure chemical, physical, and rheological properties began within the Waste Treatment and Immobilization Plant (WTP) project at the Hanford Site in approximately 2002. The need for coordination of actual waste and simulant properties measurements was identified so that measured values and corresponding terminology would be consistent and defensible across all subcontractors providing data to the WTP project. To facilitate this need, an internal guideline document was developed, reviewed within the project, implemented, and later used to formulate the workshop recommended measurement guideline.

The genesis of the guideline for simulant development began with an internal WTP guideline generated in approximately 2003 and revised in 2010. The guideline was written by the Research & Technology (R&T) department to facilitate the need for coordination of simulant development work within the WTP project and ensure that the development and use of simulants would be coordinated, consistent, and technically defensible across R&T and into commissioning.

The starting point for the rheology primer was Appendix D (titled: Rheology Primer) of the WTP project report, “Final Report: Technical Basis for HLW Vitrification Stream Physical and Rheological Property Bounding Conditions” [3]. The initial work was conducted by PNNL in 2006 to help coordinate terminology use across the WTP project and supporting contractors.

As a result of the workshop on slurry handling, mobilization, retrieval, transport, mixing, and processing and to facilitate better waste slurry handling, DOE EM sponsored needed radioactive tank waste characterization, gap analysis and reporting on waste slurry properties. A comprehensive summary of the Hanford tank waste characterization data pertinent to safe storage, retrieval, transport, and processing operations for both the tank farms and the WTP was compiled [4]. The Hanford waste report provides data on important parameters including physical properties and rheological behavior of the waste pertinent to flow-sheet unit operations, (e.g., pumping, pipeline transport, and mixing). These properties of the as-stored Hanford waste, including both the liquid and solid phases, are presented by tank and waste type. The data in this report are useful for estimating process and equipment performance and providing a technical basis for development of simulants for testing. The report also includes a waste slurry/sludge properties design basis gap analysis used to determine what additional actual radioactive waste and associated properties are needed to be measured and represented using simulants.

This paper is intended to focus on the formulation of the guidelines and final consensus standard guide for simulant development. The following section provides an overview of the guidelines and methodology for simulant development.

SUMMARY OF SIMULANT GUIDELINES

Simulants are used for testing to reduce the cost and hazards associated with procuring, working with, and disposing of actual radioactive or hazardous waste. In addition, the use of simulants provides for greater control of pertinent properties and allow for variations in material properties (e.g., sensitivity studies) to be incorporated into the test program. Simulants are used for developing process designs, demonstrating that the waste process flowsheet is viable at the design process limits of the various unit operations, and to facilitate process scale-up. Again, accurate and sufficient characterization of the chemical and physical properties as well as the rheological behavior of the radioactive/hazardous material after each pertinent processing step is essential to have sufficient data to develop and validate simulants.

Designing, testing, and building properly functioning full-scale waste processing facilities will be difficult if: there is insufficient knowledge of the waste properties basic data, simulant development is not rigorous enough, or prior testing is too limited. The following sections outline the process for simulant development and provide key factors that should to be considered. The following outline is divided into four high-level tasks: 1) Simulant use definition, 2) Determination of simulant composition and properties, 3) Simulant design requirements, and 4) Simulant development and verification. The following sections focus on the work activities and do not discuss the quality assurance and documentation that must accompany such a process.

Simulant Use Definition

The first step in simulant development is to determine the use of the simulant, e.g., studying slurry transport in piping, particle segregation and size reduction, filtration, precipitation kinetics, or a combination of flowsheet process unit operations. The simulant use definition exercise will assist in determining the simulant characteristics (i.e., chemical and physical properties, rheological behavior, makeup) required for testing of various unit operations. Key waste characterization properties important to slurry retrieval, transport and processing options and relevant engineering correlations used to select and size equipment, e.g., pumps, piping and mixers, are discussed by Wells, et al. [4].

Defining the simulant use will identify the key issues to be addressed in the following steps associated with determining the simulant composition and specifying the simulant requirements. Factors and questions to be addressed in defining the simulant use include:

- Purpose of the results: safety analysis, longevity testing, erosion/corrosion, chemical conditioning (dynamic process).
- Nature of the processes to be evaluated and type of testing to be conducted. Is the simulant to be used for a single pass or batch operation or must material degradation issues be addressed?
- Range of processes to be covered – does the initial simulant have to meet specific requirements (e.g., properties) at specific points in the process history?

- Conservatisms – are average or most adverse conditions to be evaluated? Are bounding conditions needed such that multiple simulants will be required.

Another important factor associated with simulant use is whether some form of "scaling" will be applied for the testing. The term "scaling" has different meanings for different audiences as do the concepts of scale-up and scale-down. Kuhn et al. [5] provides an overview of various scaling approaches and the scaling process for application to waste mixing systems. At a high-level three basic approaches are defined:

- Pure similitude - where geometric, kinematic, and dynamic similitude are satisfied such that the performance of a scaled system is the same as the prototype at the same dimensionless locations and times. Relative to simulant development, the simulant properties would be based on those of the actual waste but in comparison may be significantly different. The simulant properties would also be scale dependent.
- Pure statistics – entails executing a statistically designed test matrix over the ranges of interest for the parameters that impact the performance metrics. Regression of the composite set of data provides an approximate but simple and general model of the performance metric/s. A performance metric is predicted from the multi-linear regression of log-log data, which is then used to scale up the data by evaluating the regression for the parameters of the prototype. Relative to simulant development, a simulant material is often applied to multiple geometric scales to obtain the desired data set.
- Pure Physics – relies on establishing physical models that describe in first principles the important phenomena of the process being evaluated. Existing knowledge is used to deduce geometric and kinematic ratios to be matched for scale down of the prototype.

For complex slurry systems, the final scaling strategy may be a combination of the three approaches. If a scaling strategy is being employed, it will have a significant impact on the development of the simulant. For scaling approaches, significant consideration needs to be given to the uncertainty in the actual waste properties and how they propagate through the process of scaling down a prototype system for experimental design and scaling up the results to predict final prototype performance. Scaled testing is usually conducted at multiple scales or large enough scale to achieve an acceptable level of risk and provide sufficient understanding of basic unit operations, both independently and as required, in an integrated fashion.

The Hanford Tank Operations Contractor Waste Feed Delivery Mixing and Sampling Program provides an example of a scaling strategy applied to testing with slurry simulants. Kelly et al. [6] provides results for three major performance categories: Limits of Performance (LOP), Solids Accumulation, and Scaled Performance. These tests utilized the simulants of Lee et al. [7]. Wells et al. [8] evaluated a specific subset of the Scaled Performance test data. In addition to the primary metric of transfer concentrations of undissolved solids, Wells et al. [8] also evaluated scaling with jet velocity for additional metrics including cloud height and effective cleaning radius to increase confidence in the transfer concentration test data scale-up. These additional performance metrics are significant because they are available for both small-scale and full-scale test data and predictions, whereas transfer concentrations of undissolved solids are not. Therefore, these additional metrics provided a way to test scaling relationships and they presented a means by which to relate expectations for other metrics to full scale. The test data indicated that the jet velocities used in the tests are lower than the jet velocities required to relate performance to full scale. Improved performance is therefore predicted for full scale, as undissolved solid concentrations in each batch are closer to the pre-transfer sample undissolved solid concentrations at higher jet velocities.

Simulant use definition is complete when:

- Process to be evaluated with the simulant has been fully defined.
- Range of operating conditions and performance metrics to be conducted have been specified.
- Use of the test results have been determined.
- Scaling strategies (if applicable) have been developed.

Determination of Simulant Composition and Properties

Simulant compositions should be based upon data from characterization of actual waste, e.g., actual Hanford tank waste data [4], and any available data from scaled testing using actual waste as the starting point. Combined or integrated unit operations flowsheet testing may require simulant waste streams that do not readily have actual waste testing from which to determine the actual composition. In this case, flowsheet models may need to be developed (depending on the complexity of the conditioning process) to ensure the correct additional stream compositions (e.g., recycle streams from other unit operations) are addressed and incorporated.

For simulants formulated to test specific sub-unit operations such as pumps, slurry transport piping, or mixer configurations, the composition may be tailored to only the chemical and physical properties and rheological behavior that are known to affect specific key operating/processing parameters. Identifying the key parameters on which the process of interest are dependent will aid in defining key metrics for the design and verification of the simulant. Consider separate scopes associated with flow of the waste stream through a pipeline. One scope is concerned with evaluating the slurry transfer through the pipe (e.g., identifying critical velocity for deposition and system pressure drop) while the other scope is focused on evaluating the abrasive wear on valve components. The material constituents and properties that dominate each process are not necessarily the same. The critical velocity will be impacted by the larger/denser particles while the abrasive wear is dominated by the harder material of sufficiently small enough size that it can be deposited between the moving parts of a valve. For simulants intended to define the limits of a process unit operation or specific piece of equipment, a range of appropriate simulant compositions may need to be developed to define the bounding operational limits.

Another consideration to be addressed at this stage of development is whether unique constituents of the simulant need to be characterized or separated from the slurry simulant as part of the test (e.g., concentration of a specific sentinel particle being mobilized or transferred by a system).

Determination of simulant composition and properties is complete when:

- Waste streams to be simulated and characterized have been identified including any combined or integrated unit operations flowsheet testing/modeling.
- Important parameters and metrics to be measured and determined have been identified as well as the corresponding ranges (e.g., rheogram for defined range of strain rates).
- Methodologies and procedures have been defined by which measurements will be made and metrics evaluated.

Simulant Design Requirements

The necessary and sufficient simulant properties to measure (should be the same for both actual and simulant waste) for each affected sub-unit piece of equipment, complete unit operation, or integrated set of unit operations must be determined for development of simulant design requirements. Design assumptions employed at the particular point in the waste conditioning process must also be specified. For example, design parameters for pumps, agitators, piping and vessels that would affect the simulant will need to be documented. Examples of key properties that may be critical to the conditioning process follow:

- Key processing properties. These will consist of the properties that are measured during testing of a piece of equipment or unit operation. Examples include critical pipeline transport velocity, erosion/corrosion, pressure drop, representativeness of sampling, filtrate flux rate, etc.
- Key chemical properties. The chemical properties of the simulant necessary to ensure a valid simulant is prepared, e.g., an ion exchange simulant may need to demonstrate different chemistry than an evaporator simulant.
- Key physical (including rheological) properties. Physical properties of the simulant necessary to ensure a valid simulant are prepared. Examples include particle size and density distributions: particle shape, size, hardness; yield stress and shear stress versus shear rate viscosity; etc.

Consultation with engineers/scientists/laboratory analytical chemists who are technically knowledgeable with all sub-unit pieces of equipment, complete unit operation, or an integrated set of unit operations as well as analytical analysis equipment to determine how close each measured property must be to the target for the important chemical analyses, physical properties, rheological behavior, and other characteristic metrics is necessary. This iterative process would be used to develop the acceptance criteria for the simulant to verify the simulant preparation procedure. Target acceptance criteria should be based upon actual waste characterization and testing data to the extent possible.

Simulant design requirements are complete when:

- Parameters to be used for simulant verification have been defined including the required uncertainty for the measurements and derived metrics. These need to be defined over the entire range associated with the scaling strategy and for any combined or integrated unit operations flowsheet testing/modeling.
- Interim metrics (if applicable) as well as associated uncertainties have been defined to monitor simulant degradation.
- Maintenance and handling requirements for the simulant have been defined as well as requirements for simulant replenishment or replacement.
- Simulant compatibility requirements with the test setup and environment have been identified and meet all permitting requirements.
- Recycling requirements for the simulant or simulant constituents have been defined as well as methods to meet the requirements.
- Disposal path for the simulant has been identified and determined to meet regulatory requirements.

Develop and Verify Simulant

Laboratory development and testing is conducted to develop a simulant recipe and demonstrate the simulant requirements are met. Some examples of ‘high level’ criteria may include:

- Substitutes used for radioactive species are appropriate;
- Correct ionic forms of waste components used (e.g., nitrates, nitrites, etc.);
- Charge balance completed appropriately;
- Physical properties of solids used are correct (e.g., phase, size, density, morphology,

hardness, etc.)

- Addition of chemicals, liquids, etc. is completed in the correct sequence to avoid unwanted chemical reactions;
- Important processing parameters, hold points for measurement confirmation, and other formulation requirements necessary to develop the final simulant were followed.

The final simulant properties are then be measured and compared with the previously determined acceptance criteria. All necessary and sufficient simulant properties should be within the acceptance criteria specifications. Initial testing of simulants is usually performed on batches generated at small-scale. An important requirement of simulant preparation procedures is that consistent results are obtained for significantly larger batches (e.g., pilot-scale applications). Verification of the preparation procedures for the full range of make-up requirements is an important aspect of the verification process that should not be over looked.

Verification and validation of simulates, which are defined in the following section “ASTM Standard Guide C1750” are critical. The validation methodology demonstrates and documents that the necessary and sufficient actual waste properties are mimicked. Standardized chemical, physical and rheological measurement methods should be used for measuring both the actual waste and simulant materials (ASTM C1752) [9] and should have been specified during the determination of the simulant composition.

Simulant development and verification is complete when:

- Simulant recipe/s satisfying the defined requirements has been developed
- Simulant make-up procedure and associated scale-up of make-up procedure have been verified to yield acceptable and consistent material.
- Simulant properties defined by requirements have been measured and are within acceptance criteria.
- Disposal path for final simulant recipe is verified to meet regulatory requirements

CONSENSUS STANDARDS

Federal agencies are using consensus standards developed by private sector standards development organizations (SDO) to demonstrate transparency, impartiality, and to save costs (e.g., not maintaining multiple, duplicative standards within federal agencies and utilization of previous efforts) to name a few benefits. Development of consensus standards is compliant with federal law which codified the Office of Management and Budget (OMB) Circular A-119 [10] through signing into law the National Technology Transfer and Advancement Act of 1995 (Public Law 104-113) [11]. This act contains language on "Federal Participation in the Development and Use of Voluntary Standards." The act directs that "... all Federal agencies and departments shall use technical standards that are developed and adopted by voluntary consensus standards bodies, using such technical standards as a means to carry out policy objectives or activities determined by the agencies and departments." The act also states that “…Federal agencies and departments shall consult with voluntary, private sector, consensus standards bodies and shall, when such participation is in the public interest and is compatible with agency and departmental missions, authorities, priorities, and budget resources, participate with such bodies in the development of technical standards.”

Development of consensus standards in general uses a process that encompasses several important characteristics. The standards definition and development process is open to all who are directly affected by the activity, i.e., comprised of representatives from all related groups of interest to the subject matter (e.g., manufacturers, end users, research and independent testing laboratories, code authorities, regulators, government agencies and academics). Impartiality is maintained so that federal or state agencies, private sector entities, stakeholders, or regulatory groups cannot dominate the process or final outcome. And lastly, the standards development process must include a procedure by which all relevant technical views are considered openly and an appeals process is incorporated into the process.

Participation in the standards development process provides for effective communication between government and regulatory agencies and entities that must complete the work. The consensus standards development process provides a flexible environment in which the private sector can work together with regulatory and governmental agencies to meet the public goals of protecting the environment, safety and health of the population. In summary, consensus standards development organizations provide an open process whereby all directly affected parties and technical experts have the opportunity to participate in developing consensus in an impartial and transparent manner [12, 13, 14].

DOE has adopted the guidance provided in OMB Circular A-119 and PL 104-113 [15]. As such, all three guidelines from the Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop have been developed and approved as consensus standards through ASTM International: 1) ASTM C1750 – 11 "Standard Guide for Simulant Development, Approval, Validation, and Documentation;" 2) ASTM C1751–11 "Standard Guide for Sampling Radioactive Tank Waste;" and 3) ASTM C1752–11 "Standard Guide for Measuring Physical and Rheological Properties of Radioactive Solutions, Slurries, and Sludges" [16, 17, 9].

ASTM INTERNATIONAL STANDARD GUIDE C1750

The consensus standard guide ASTM C1750 [16] promulgated from the DOE slurry handling workshop to facilitate nuclear facility design verification was developed "…to provide general considerations for the development, verification, validation, and documentation of high-level waste (HLW) tank simulants. Due to the expense and hazards associated with obtaining and working with actual wastes, especially radioactive wastes, simulants are used in a wide variety of applications including process and equipment development and testing, equipment acceptance testing, and plant commissioning. This standard guide facilitates a consistent methodology for development, preparation, verification, validation, and documentation of waste simulants."

The following general outline of the simulant development guide describes: "(1) defining simulant use, (2) defining simulant-design requirements, (3) developing a simulant preparation procedure, (4) verifying and validating that the simulant meets design requirements, and (5) documenting simulant-development activities and simulant preparation procedures."

1. simulant use definition;
2. simulant composition definition;
3. simulant design requirements;
4. simulant development test plan;
5. development of a simulant preparation procedure;
6. consideration for simulant scale-up and fabrication;
7. verification simulant meets design requirements; and,
8. documentation of simulant development, verification, validation, and preparation activities.

Note: definition of "simulant validation" and "simulant verification" from C1750 follow:

"*simulant validation*, *n*—establishment of documented evidence that confirms that behavior of the simulant adequately mimics the targeted actual waste behavior. Simulant

> validation can be expressed by the query, “Are you making the correct simulant?” and refers back to the needs for which the simulant is being developed.” and
> *“simulant verification*, *n*—establishment of documented evidence which provides a high degree of assurance that the simulant meets the predetermined design and quality requirements. Simulant verification can be expressed by the query, “Are you making the simulant properly?”

APPLICATION OF THE STANDARD GUIDE C1750 FOR SIMULANT DEVELOPMENT

A variety of recent projects within the DOE complex have applied ASTM C1750 for the development of simulants. The use of the standard has improved 1) the traceability and representativeness of simulant properties to those of the corresponding waste; 2) the selection and determination of applicable metrics for defining the simulant requirements; and 3) the evaluation, comparison, and utilization of simulant development efforts and results between programs and projects. Examples of simulant development efforts associated with equipment evaluation, instrument development, design verification, scientific study, and safety issue resolution are summarized below.

The purpose of the Hanford Tank Operations Contractor (TOC) Waste Feed Delivery (WFD) Mixing and Sampling Program is to mitigate the technical risks associated with the ability of the WFD systems to adequately mix and sample High Level Waste (HLW) feed to meet the Hanford WTP Waste Acceptance Criteria (WAC). Lee et al. [7] and Gauglitz et al. [18] used ASTM C1750 for guidance to ensure that the simulant selection is relevant to test objectives to satisfy DNFSB 2010-2 Sub-Recommendation 5, Commitment 5.5.3.5, “Definition and qualification of simulants for testing to establish tank farm performance capability” [19]. The results of this testing are presented in Kelly et al. [6]. The development of a test simulant for leakage testing of safety-significant isolation valves for Double Valve Isolation (DVI), Wells [20], built upon the simulant definition of Lee et al. [7]. The DVI simulant development report [20], provides a good example of the comparison of two different physical simulants that were developed to evaluate different phenomenon for the same waste. The example demonstrates the importance of selecting relative metrics for defining the simulant requirements. The final results of the DVI test effort are presented in Enderlin et al. [21].

The fluid velocity at which particle deposition occurs in slurry transport piping (critical velocity for deposition) is a key WAC parameter that must be accurately characterized to determine if the waste is acceptable for transfer to the WTP. The Ultrasonic PulseEcho (UPE) system was developed at PNNL to detect particle motion in mixing vessels and piping for the purpose of determining in real time when sediment is developing in pipes and vessels. The UPE system uses non-invasive ultrasonic transducers mounted to the outside bottom of pipes and vessels to detect sediment formation. The UPE system was qualified for flow loop application under two test campaigns at PNNL using simulants with properties that bound those of tank waste and a full-scale flow loop that represented that being designed by the Hanford TOC for waste feed characterization [22-24]. The performance of the UPE system was recently tested with simulants developed by Lee et al. [7] to assess the system’s ability to detect the onset of critical velocity in a Hanford Tank Waste Feed cold test flow loop designed for characterizing Hanford tank waste prior to transferring it to the WTP [25-26]. The ultrasonic results from performance tests conducted using the Lee et al [7] simulants in the Hanford Tank Waste Feed cold test flow loop were in good agreement with visual observations facilitated by segments of clear pipe in the test loop. The instrument development effort applied simulants generated via ASTM C1750 to advance prototypic instrumentation into a process worthy design. As a result, the UPE system was recommendation for future Hanford tank farm waste feed delivery projects [26].

The DOE Office of River Protection (ORP) has tasked PNNL and SRNL with supporting the development of the WTP Full Scale Vessel Testing (FSVT) Program. The objective of the FSVT Program is to test prototypic WTP vessels that employ pulse jet mixer (PJM) technology to obtain data for design verification that demonstrate the systems will adequately mix, transfer, and sample radioactive waste slurries. Within the FSVT Program, the National Laboratory Team has the responsibility to develop simulants to be used in testing. In general, the simulant development approach is consistent with the methodology outlined in ASTM C1750, with modification in the basic steps to accommodate for the development of a component simulant. Component selection and the determination of simulant composition are similar between this work and the aforementioned TOC effort.

In underground storage tanks containing radioactive waste, multiple settled layers that vary in physical properties (e.g. density, rheology) have formed. The different physical properties can be a result of differences in gas retention, degrees of settling, and amount of compaction in the various layers or differences in the solid particle compositions. Such a configuration can experience a Rayleigh-Taylor (RT) instability, in which a less-dense lower layer rises into a more-dense upper layer, resulting in a release of retained gas. PNNL is conducting experiments to study gas retention and release in Hanford waste due to potential buoyant motion within the settled bed of solids. The purpose of the work is to provide quantitative information for estimating the size of gas release events within Hanford double-shell tanks (DSTs) should a RT instability occur. Simulant development and selection for this effort has been performed per ASTM C1750.

One of the events postulated in the hazard analysis for the WTP and other DOE nuclear facilities is a breach in process piping that produces aerosols with droplet sizes in the respirable range. To expand the data set upon which the WTP accident and safety analyses were based and improve the WTP methodology [27], an aerosol, spray-leak, test program was conducted by PNNL. The test program collected aerosol generation data at two scales that used the same simulants designed to mimic the relevant physical properties projected for actual WTP process streams. The simulants were developed and validated per the 2010 revision of the WTP project simulant development guidelines, which include requirements beyond that of ASTM C1750. The small and large-scale testing and resultant data are described in Mahoney et al. [28], and Schonewill et al. [29], respectively. Follow-on test efforts are presented in Schonewill et al. [30] and Daniel et al. [31].

RECOMMENDATIONS

Review of the simulant guide development process and the recent application of the standard guide C1750 to DOE activities have led to the following recommendations.

- Development of additional consensus standards, which were discussed during the DOE slurry handling workshop, are needed to support development of new test methods to 'close' current technical gaps and include:
    - The need to link actual waste particles to their respective size and composition (density) (i.e. a direct determination of actual waste particles particle size and density distribution [PSDD]),
    - A shear strength test method and resulting measurement/s for slurries that provide relevant parameter/s for retrieval/mobilization, mixing, and blending activities for direct application in engineering equations.
    - Continuing the development of the rheology primer into a consensus standard.

- Review of the simulant development standard guide should not wait for the maximum required five year review cycle and instead should take advantage of recent DOE activities using a lessons learned approach to revise the Standard Guide.

CONCLUSIONS

State and government agencies, stakeholders, regulatory agencies, academia, private industry and national laboratories must work together to meet the goals of regulatory policy and laws for the safe removal and treatment of weapons production legacy nuclear wastes in a technically and economically sound manner under a set of commonly developed consensus standards. This helps facilitate that all goals pertaining to public health, safety, and protection of the environment are met.

An aggressive slurry physical and rheological characterization campaign of tank waste is needed for proper design of slurry transport, mixing, and processing systems because incorrect basic data used for the developed simulants lead to flawed design.

Three guidelines from the DOE EM sponsored "Slurry Retrieval, Pipeline Transport & Plugging and Mixing Workshop" were developed and approved as consensus standards through ASTM International:

1) ASTM C1750–11 "Standard Guide for Simulant Development, Approval, Validation, and Documentation;"
2) ASTM C1751–11 "Standard Guide for Sampling Radioactive Tank Waste;" and
3) ASTM C1752–11 "Standard Guide for Measuring Physical and Rheological Properties of Radioactive Solutions, Slurries, and Sludges."

DOE projects have begun applying the standards resulting from the workshop. The workshop and resulting follow-on efforts successfully demonstrated the resolution of identified design and development issues through the formulation of consensus standards.

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