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Quality Assurance Plan for Final Waste Forms Project in Support of the Development, Demonstration, Testing and Evaluation Efforts Associated with the Oak Ridge Reservation's LDR/FFCA Compliance

T. M. Gilliam
C. H. Mattus

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ORNL/TM-12724

Chemical Technology Division

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T. M. Gilliam
C. H. Mattus

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APPROVAL SIGNATURES

This Quality Assurance Project Plan has been approved by:

Project Leader: Thomas M. Gilliam Date: 8/1/94

DDT&E Program Coordinator: T. B. Conley Date: 8/2/94

Program QAS: S. E. Lewis Lambert Date: 8/3/94

EDS Section Head: S M Johnson Date: 8/3/94

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LIST OF ACRONYMS

ANS	American Nuclear Society
ASTM	American Society for Testing and Materials
AVID	Accelerated Vendor Inventory Delivery
CARS	Corrective Action Report and Status (form)
CCB	continuing calibration blank
CCV	continuing calibration verification
COC	chain of custody
CTD	Chemical Technology Division (Oak Ridge National Laboratory)
DDT&E	development, demonstration, testing and evaluation
DOE	U.S. Department of Energy
DQO	data quality objective
EDS	Engineering Development Section (Chemical Technology Division)
EPA	U.S. Environmental Protection Agency
FFCA	Federal Facility Compliance Agreement
I&C	Instrumentation and Controls Division (Oak Ridge National Laboratory)
ICB	initial calibration blank
ICP	inductively coupled plasma (atomic emission spectroscopy)
ICSA	interference check standard A
ICSAB	interference check standard B
ICV	initial calibration verification
IDL	instrument detection limit
LCS	laboratory control sample
LDR	land disposal restrictions
MDL	method detection limit
MMES	Martin Marietta Energy Systems, Inc.
MS	matrix spike
MSD	matrix spike duplicate
ORNL	Oak Ridge National Laboratory
ORR	Oak Ridge Reservation
OSOR	Office of Safety and Operational Readiness
PB	preparation blank
PE	performance evaluation
QA	quality assurance
QA/QC	quality assurance/quality control
QAPjP	quality assurance project plan
QAPP	quality assurance program plan
QAS	quality assurance specialist
R&D	research and development
RCRA	Resource Conservation and Recovery Act
RPD	relative percent difference
S/S	solidification/stabilization
TCLP	Toxicity Characteristic Leaching Procedure
UCS	unconfined compressive strength

ABSTRACT

This quality assurance project plan specifies the data quality objectives for Phase I of the Final Waste Forms Project and defines specific measurements and processes required to achieve those objectives. Although the project is funded by the U.S. Department of Energy (DOE), the ultimate recipient of the results is the U.S. Environmental Protection Agency (EPA). Consequently, relevant quality assurance requirements from both organizations must be met. DOE emphasizes administrative structure to ensure quality; EPA's primary focus is the reproducibility of the generated data. The ten criteria of DOE Order 5700.6C are addressed in sections of this report, while the format used is that prescribed by EPA for quality assurance project plans.

1. BACKGROUND

The purpose of this quality assurance project plan (QAPjP) is to specify the project objectives for Phase I of the Final Waste Forms Project and to define specific measurements and processes required to achieve those objectives. This section provides the reader with the background information necessary to understand the format of the QAPjP; Sects. 2 through 12 present the plan.

This project is funded by the U.S. Department of Energy (DOE), but the ultimate recipient of the results is the U.S. Environmental Protection Agency (EPA). Consequently relevant quality assurance (QA) requirements from both organizations must be met. QA requirements of DOE are found in DOE Order 5700.6C, Quality Assurance,[1] while requirements for EPA are found in EPA/600/8-91/005, *Preparation Aids for the Development of Category III Quality Assurance Project Plans*.[2] Although the two requirement documents are compatible, the emphasis and format are different. DOE Order 5700.6C emphasizes the administrative structure to ensure proper QA, while EPA/600/8-91/005 emphasizes the reproducibility of the generated data.

Even though this QAPjP was found to be a category III, the format used for the document is that of a category II project plan as presented in EPA/600/8-91/004 (ref. 3). The relationship between sections in this document and the ten criteria of DOE Order 5700.6C is shown in Table 1.

Table 1. Relationship between the ten criteria of DOE Order 5700.6C and sections in this document

Criterion no.	Description	Applicable sections
1	Program	3.1, 3.2, 3.3
2	Personnel training and qualification	3.5
3	Quality improvement	11, 12
4	Documents and records	5.1, 7.2, 7.3
5	Work processes	5.2
6	Design	5.3
7	Procurement	5.4
8	Inspection and acceptance testing	5.4.2, 7.1
9	Management assessment	8, 12
10	Independent assessment	9

2. PROJECT DESCRIPTION

On June 12, 1992, DOE Oak Ridge Operations signed a Federal Facility Compliance Agreement (FFCA) with EPA Region IV regarding Oak Ridge Reservation (ORR) mixed wastes subject to the land disposal restrictions (LDR) provisions of the Resource Conservation and Recovery Act (RCRA). The LDR/FFCA establishes an aggressive schedule for conducting treatability studies and developing treatment methods for those ORR mixed (radioactive and hazardous) wastes listed in Appendix B to the Agreement. These are wastes for which treatment methods and capabilities have yet to be defined. Requirement 5 of the Agreement states the following:

> . . . DOE shall submit to EPA for review and approval a plan for the treatment of the LDR prohibited wastes identified in Appendices 1B, 2B, and 3B. This plan must identify the treatment strategy for such wastes to meet LDR treatment standards and must include a schedule, not to exceed two (2) years after the submittal of this plan (i.e., March 1995), for the evaluation and prioritization of treatment method options, treatability studies if required, and technology development.[4]

At an upper management level, this requirement was satisfied by the DOE *Strategic Plan*.[5] In the *Strategic Plan*, the wastes are divided into two categories:

1. those wastes, listed in Appendices 1A, 2A, and 3A of the Agreement, for which treatment methods and facilities exist that meet the LDR standards; and
2. those wastes, listed in Appendices 1B, 2B, and 3B of the Agreement (hereafter referred to as Appendix B wastes), for which no treatment methods or facilities exist that meet the LDR standards.

A development, demonstration, testing and evaluation (DDT&E) program has been initiated to provide those efforts necessary to identify treatment methods for all of the wastes that meet Appendix B criteria. The DDT&E program has assembled project teams to address treatment development needs in a variety of areas, including that of Final Waste Forms (i.e., stabilization/solidification processes). As more definitive characterization data on the waste are obtained, any wastes newly classified as Appendix B will be so identified in updates to the FFCA and will be included in treatment development programs.

In the context of this project, solidification refers to treatment that renders the waste a "solid." Solidification encompasses technologies such as filtration, drying, and calcination that remove liquid and result in a dry or "solid" residue. Coating the dry waste with a polymer or similar substance would be considered solidification within the context of this project. Stabilization, which may also involve solidification, refers to treatment which involves reaction(s) with the waste constituents of concern to render them nonhazardous or to convert them to a chemical form which is less hazardous. As most processes involving stabilization also involve solidification, they are referred to as stabilization/solidification (S/S) processes. The Final Waste Forms Project gives priority to the more traditional S/S processes, but it does not exclude technologies such as filtration and drying. Waste-form types that this project is actively considering include grout (e.g.,

cement-based technology), glass (e.g., vitrification technology), and organic binders (e.g., polymer encapsulation).

It is not clear, at present, what the final form and composition of the yet-to-be-determined treatment method residues will be and which waste streams will require, or are candidates for, S/S. In addition, ongoing waste characterization has not progressed to the point where potential waste candidates for direct S/S can be readily identified. This process consists of reviewing existing waste records, conducting generator interviews, and performing sampling and analysis, where required, to acquire more detailed information on waste matrices and contaminants. As these data are obtained, candidate waste forms will become more apparent, and regulatory requirements for treatment of specific wastes can be clearly defined.

The purpose of this document is to define the QA requirements to be used in Phase I of the Final Waste Forms Project. In Phase I, treatability studies will be performed to provide "proof-of-principle" data to establish the viability of S/S technologies. This information is required by March 1995. The treatability studies will be performed using a mixture experiment. For each waste stream studied, 16 to 20 formulations will be tested and the data generated will feed the statistical design to define an area of possible formulations having the requested properties. The statistician needs the true variability for all the data generated; therefore, no data should be rejected during Phase I unless a procedure deviation occurs during a part of some test. In such an instance, this part of the test must be repeated. Among the formulations tested, three will be true replicates; that is, all the steps involved during the sample preparation and testing will be performed independently.

In Phase II, further treatability studies, some at the pilot scale, will be performed to provide sufficient data to allow treatment alternatives identified in Phase I to be more fully developed and evaluated, as well as to reduce performance uncertainties for those methods chosen to treat a specific waste. The focus of these Phase II efforts will be to obtain the necessary data to design the process module(s) and optimize the ability of the waste-form formulation to accommodate expected variations in both feed characteristics and process operations. While Phase I will concentrate primarily on laboratory- or bench-scale studies, Phase II will focus on laboratory-scale studies, as well as on bench- and pilot-scale demonstrations.

2.1 THE PROCESS

This project will evaluate three generic types of S/S processes: grout, vitrification, and organic binder. Initially, the project will focus on grout processes. The exact composition of the waste forms (i.e., the waste-form formula) to be evaluated will evolve over the course of the project. When these compositions have been determined, they will be reported in accordance with the requirements outlined in Sect. 5.3.1. The evaluation process for data acquisition and analyses for all three S/S processes is shown in Fig. 1. As Fig. 1 indicates, the waste of interest will be combined with the appropriate additives and treated at selected process conditions to produce an S/S product. The product will then be subjected to specified tests; the data will be collected routinely and subsequently analyzed and compared against the performance objectives.

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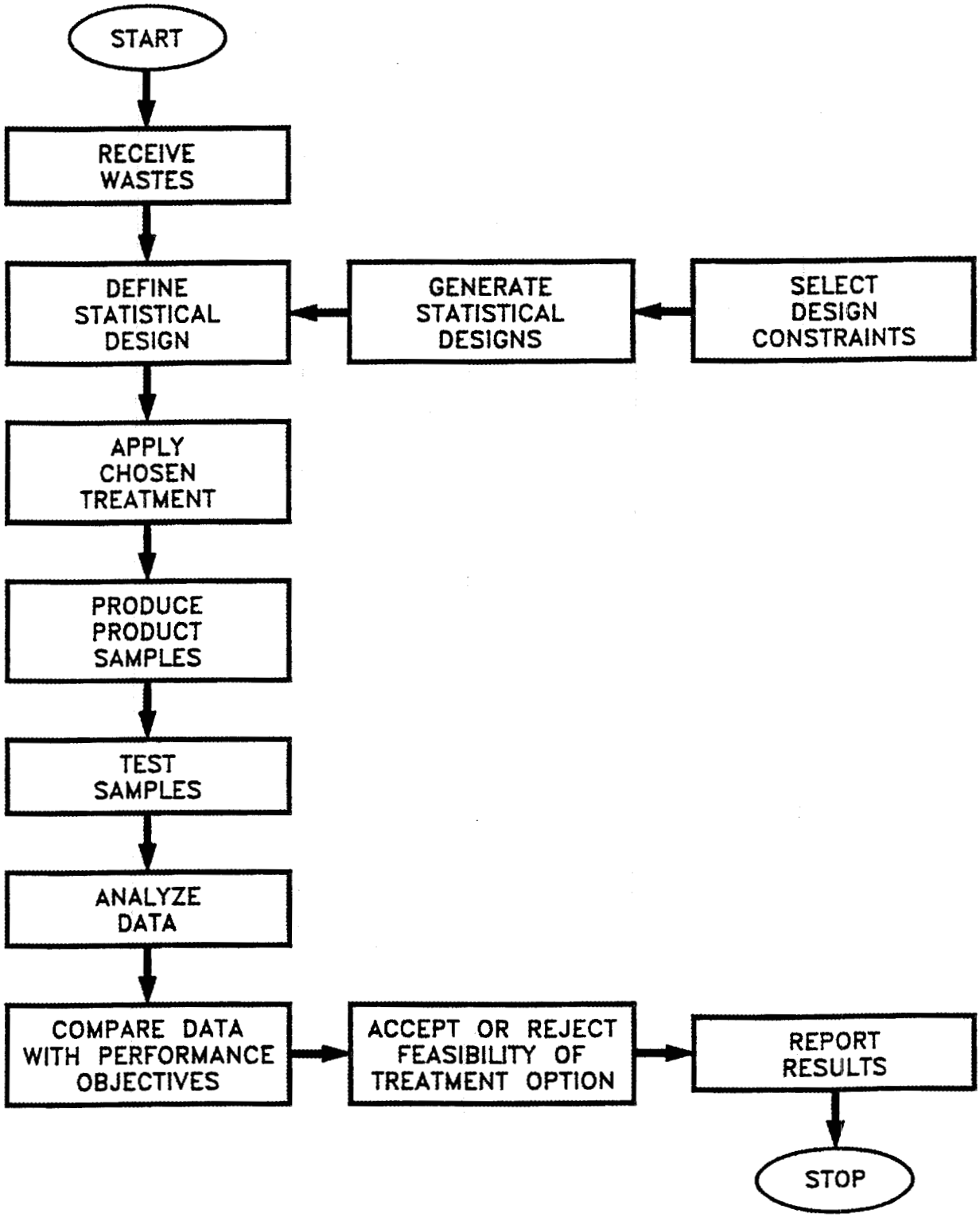

Fig. 1. Simplified logic diagram of the project's data acquisition and evaluation process.

2.2 PERFORMANCE OBJECTIVES

The objective of this project is to establish the feasibility of S/S as an option for disposal of selected Appendix B wastes. The feasibility of S/S is established by meeting criteria detailed in *Final Waste Forms Project: Performance Criteria for Phase I Treatability Studies*[6] in a technically defensible manner. As discussed in ref. 6, the test methodology is generic, although acceptable test values are waste code specific. Consequently, the primary objectives are for the S/S product to meet acceptable values (as defined in ref. 6) for the following:

1. free-standing liquid in accordance with a modified American Nuclear Society (ANS) 55.1, and
2. extract concentration in accordance with the Toxicity Characteristic Leaching Procedure (TCLP).

Secondary objectives of the project involve obtaining qualitative data to provide guidance for further studies and conceptual design. These include the following:

S/S Product	Raw Waste
Unconfined compressive strength	Moisture content
Viscosity (qualitative observation)	Bulk density
	TCLP

Another secondary objective is to model values for TCLP, free liquid, and compressive strength to provide a means for interpolation of the resulting data.

2.3 EXPERIMENTAL DESIGN

As shown in Fig. 1, the exact nature (e.g., composition of the waste form) will be specified by the statistical experimental design. Development of the design is an iterative process that will be documented in accordance with Sect. 5.3.1. Until completion of the design, the exact number of measurements cannot be specified.

3. PROJECT ORGANIZATION AND RESPONSIBILITIES

3.1 PLANNING

The Final Waste Forms Project is complex, involving personnel in the Engineering Development Section (EDS) within the Chemical Technology Division (CTD) at Oak Ridge National Laboratory (ORNL), other Martin Marietta Energy Systems (MMES) personnel, and subcontractors. In addition, it involves both line management within EDS and program management within the DDT&E program. This project is governed by the program QA requirements set forth in the *Quality Assurance Program Plan for the Development, Demonstration, Testing and Evaluation Efforts Associated with the Oak Ridge Reservation's LDR/FFCA Compliance.*[7] This quality assurance program plan (QAPP) requires the development of a QAPjP for the Final Waste Forms Project; this document meets that requirement.

As the work performed in this project will be managed and conducted primarily by EDS personnel, the project elements, as described in this document, are consistent with those in *Oak Ridge National Laboratory Engineering Development Section Quality Assurance Plan for Research and Development.*[8]

The QA requirements described in this document are utilized in a graded application and affect the quality and reliability/credibility of research and development (R&D) and the resultant investigative data and documentation. These activities include the functions of attaining quality objectives and ensuring that an appropriate QA program scope is established. Activities affecting quality include personnel training and qualification, design control, procurement, material handling and storage, audits and surveillances, testing, R&D, investigative activities, documentation, identification of deficiencies and corrective actions, QA record keeping, and self-assessment. The procedures described in this document are specific to this project.

This project has been evaluated and determined to be a QA Category III Project in accordance with ref. 2: that is, this project is one that "produces results used to evaluate and select basic options, or to perform feasibility studies."[2,3]

3.2 ORGANIZATION

This project involves both program and line management. DDT&E program management is responsible for defining and/or approving project objectives and scope as well as for defining and ensuring compliance with program QA requirements applicable to the project. EDS line management is responsible for ensuring compliance with specified QA requirements during day-to-day operations.

The organizational structure for DDT&E program management is delineated in the program QA plan,[7] while the organizational structure for EDS line management is delineated in the EDS QA Plan.[8] The project organizational structure is shown in Fig. 2. Only project personnel and direct interfaces with the project are discussed in Sect. 3.3. Other levels of both line and program management personnel and responsibilities are as defined in refs. 7 and 8.

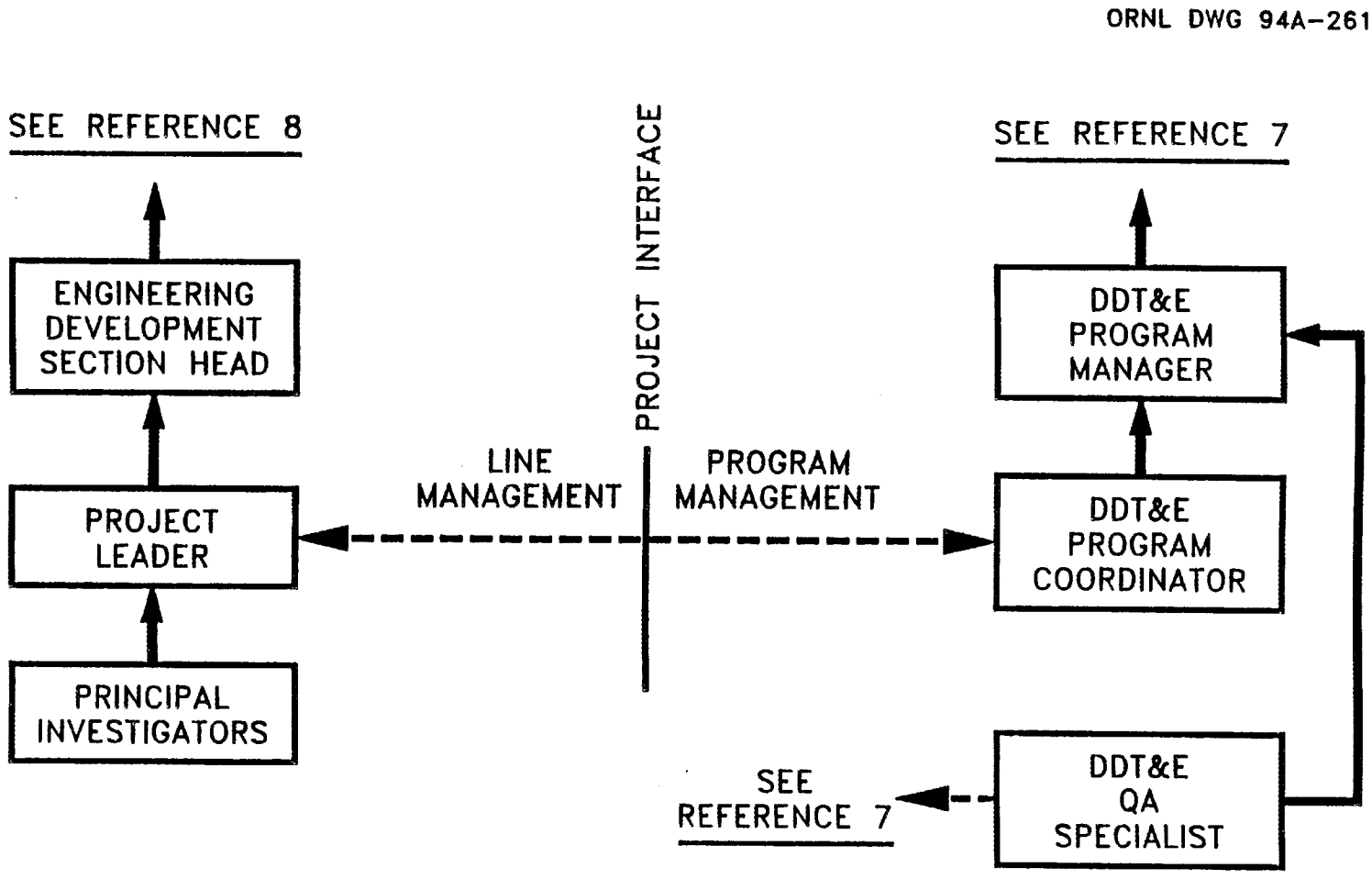

Fig. 2. Project organization chart.

3.3 RESPONSIBILITIES

Overall responsibilities, authority, and accountability for major organizational functions within the project are outlined in this section. The implementation, achievement, and maintenance of quality is the responsibility of the EDS line organizational management and personnel performing the work. It is the responsibility of all personnel performing work within this project to execute their tasks in accordance with this plan. Quality is verified by individuals or organizations that are not directly responsible for performing the work. In the context of this section, DDT&E program personnel are included in the definition of "personnel not directly responsible for performing the work."

3.3.1 DDT&E Program Coordinator (or Program Coordinator)

The DDT&E program coordinator is responsible for implementing the DDT&E QA Program; interpreting and implementing applicable state and federal codes, standards, and regulations relevant to the DDT&E effort within this project; ensuring that plans generated by this project are in conformance with the program management plan; approving all project-related plans, including this document and work plans; and serving as the interface between this project's EDS line management and DDT&E program management.

3.3.2 Project Leader

The project leader is responsible for the following activities:

- establishing the technical scope necessary to formulate and prepare project-specific plans;
- approving task plans, procedures, and reports generated by this project;
- interfacing with the program quality assurance specialist (QAS) to ensure that the appropriate level of quality assurance/quality control (QA/QC) is applied to this project and each task within the project;
- ensuring that proficiency of all personnel is being maintained in the performance of assigned tasks;
- serving as the interface between EDS line management and the program coordinator as well as the project interface with all subcontractors directly supporting this project;
- assisting EDS line management and DDT&E program management with the coordination of responses to any audit/surveillance/self-assessment findings on QA matters;
- providing input to the program coordinator on identifying and/or resolving quality problems; and
- overseeing management, costs, schedule, QA, and technical performance of the project.

The project leader shall review the status of each project task on a monthly basis and document that status in monthly reports to the program coordinator and shall ensure that both programmatic and line QA requirements are satisfied.

3.3.3 Principal Investigator (or Task Leader)

The principal investigator or task leader reports to the project leader and has the primary responsibility and authority to ensure that all aspects of project activities are conducted in accordance with this document. The principal investigator is responsible for preparing task-specific QAPjPs when required by the project leader; ensuring that each employee performing activities under a project task is qualified to perform this work in accordance with requirements in this document; generating, controlling, and storing QA activity records in accordance with this QAPjP; maintaining the calibration status of measuring and testing equipment; and preparing a status summary for each assigned task on a monthly basis for the project leader.

3.3.4 Section Head

The EDS section head is responsible for implementation of self-assessments, safety assessments, and MMES policies and procedures that are applicable to this project and approves all stand-alone plans and documents generated by this project.

3.3.5 Program QAS

The program QAS is responsible for assisting the project leader on all matters related to quality; reviewing project documents such as task plans, subcontractor QAPjPs, and procedures to ensure that appropriate quality requirements have been specified; and evaluating and reporting the compliance and effectiveness of QA activities within this project through review of, or participation in, readiness reviews, audits, surveillances, and corrective action efforts.

3.4 READINESS REVIEW

The requirements for the Operational Readiness Review process, as defined in Energy Systems procedure ESP-OP-551, Operational Readiness Process,[9] shall be implemented in a manner consistent with a QA Category III project. Operational Readiness Review, as it relates to this project, will consist of (1) approval of this document, (2) approval of performance criteria, and (3) approval of operational procedures prior to initiation of the treatability studies to be conducted in MMES laboratories. In addition, at the discretion of the program coordinator, an audit or surveillance as described in Sect. 9 may be required prior to the startup of the treatability studies. Compliance with these requirements will be documented by the project leader in a letter to the program coordinator, who will countersign the letter indicating his acceptance of the review.

In addition to treatability studies conducted in MMES laboratories, similar studies will be conducted off-site by subcontractors as separate tasks under this project. As part of the Operational Readiness Review, the program QAS must determine whether the subcontractor QAPjPs are in compliance with QAPP requirements. Acceptance of these plans will be documented by a letter from the program QAS to the project leader. In addition, at the discretion of the program coordinator, program QAS, or project leader, an

audit or surveillance as described in Sect. 9 may be required prior to startup of the treatability studies.

3.5 PERSONNEL TRAINING AND QUALIFICATION

Personnel performing work or managing activities within the scope of this project shall be trained and qualified as appropriate to ensure job proficiency. Training of personnel shall be performed in accordance with Chemical Technology Division procedure CTD/PM/TRN-1, Management of Training Programs.[10] Four major types of training shall be provided to each project team member: mandated, job specific, development, and project specific.

3.5.1 Mandated, Job-Specific, and Development Training

Mandated training is that required by DOE and by corporate orders, regulations, and laws. Job-specific training ensures that an employee has the skills and knowledge to perform the job assigned. Development training is professional development that provides skills and knowledge to improve the quality of work or productivity.

The principal task associated with this project is performing treatability studies in a chemical laboratory that contains radioactive materials. The training for such work is considered "routine," not project specific. Each project team member will take the appropriate required training (i.e., mandated, job specific, or development) for this type of work. Specifically this work will require the MMES training modules related to performing work with treatability study samples, hazardous chemicals, and radioactive materials. Such training will be identified and recorded by, or be accessible through, the CTD training coordinator.

3.5.2 Project-Specific Training

The "routine" training modules are designed to ensure proficiency and to promote sound environment, safety, and health practices in the workplace. Additional training related to this specific project includes this document as well as all Standard Operating Procedures for laboratory work, which are considered an addendum to this document. The project leader will verify compliance with this required training and document that training in a letter to the program QAS.

4. QUALITY ASSURANCE OBJECTIVES

Performance objectives are described in Sect. 2.2. This section describes the specifications that measurements must meet in order to achieve these performance objectives. Such specifications are defined to be QA objectives. Phase I will be a screening phase in which various waste-form compositions are studied for a given waste stream. At this date, plans are for the mixture experiment to examine 16 to 20 formulations for each waste stream, with 3 of the formulations being true replicates. These replicates will have the same compositions, but all the steps of preparation and testing will be performed independently. Because the statistician needs to evaluate the true variability in the model, all the data generated will be valid and none will be rejected. A rerun will be conducted only if a deviation from procedure occurs during preparation or testing.

4.1 DETERMINING QA OBJECTIVES

One of the primary performance objectives is to provide technically defensible data. Typically, data adequacy is defined by characteristics such as precision, accuracy, and completeness:

- **Precision.** The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is estimated by means of duplicate/replicate analyses.

- **Accuracy.** The closeness of agreement between an observed value and an accepted reference value. When applied to a set of observed values, accuracy will be a combination of a random component and a common systematic error component.

- **Completeness.** The ratio of the number of measurements taken that meet QA objectives for precision and accuracy to the total number of measurements.

However, to assess the adequacy of these specifications, it is important to document their rationale. Specifications may be based upon regulatory requirements, experience, and project-specific technical requirements.

4.1.1 TCLP

The primary performance objective is to meet acceptable TCLP values to allow land disposal under existing regulations.[6] As previously stated, this project is considered a QA Category III Project. A review of refs. 11 and 12 indicates that the appropriate data quality objectives (DQOs) applicable to these data are DQO Level III. DQO Level III is described as follows: ". . . analyses are performed at an established laboratory in accordance with accepted methodology and internal laboratory QA program procedures. Analyses are designed to provide results within the accuracy of routine laboratory procedures. . . ."[11] For this project, "within the accuracy of routine laboratory procedures" is taken to be that reported in SW846 (ref. 13). The minimum reported

quantified values of the TCLP extract analysis—by any appropriate method, but expected to be primarily inductively coupled plasma (ICP)*—are specified as 0.5 times the LDR threshold values of the RCRA constituents of interest (which are waste specific).

For the analytical measurements, precision and accuracy are determined by the QA requirements of the method followed. Precision of 20% and accuracy of 25% can usually be achieved on many matrices,[13] but these parameters, especially for accuracy, are matrix dependent. If one parameter is outside the limits, the data will not be considered unusable as long as the reason for the deviation of the criterion can be documented. For example, if a spike recovery is outside the acceptability window, a postspike on the digested sample can be analyzed; if the recovery is within 25%, a matrix interference can be suspected. Such data will be considered valid for this project.

4.1.2 Free Liquid

The primary performance objective is to have no free liquid as determined by a modification of a standard test method.[14,15] For this project, the modification involves use of a smaller sample size as well as a smaller container (250-mL graduated cylinder).

Free liquid will be evaluated only for precision and completeness. Accuracy is not relevant for this test. The precision will be appreciated by comparing the results obtained on the true replicates run for each waste stream. Considering the need for true variability for the statistical model, no acceptability window is defined for this criterion. Completeness (see Sect. 4.3) should be 100% considering that all measurements will be performed and will provide results for subsequent data reduction.

4.1.3 Unconfined Compressive Strength (UCS)

As for free liquid, UCS measurements will be evaluated only for precision and completeness. The precision will be appreciated by comparing the results obtained on the true replicates run for each waste stream. For the same reasons as for free liquid, no acceptability criterion exits for this parameter. Completeness should be 100%, considering that all measurements will be performed and will provide results for subsequent data reduction.

4.1.4 Additional Secondary Performance Objectives

As discussed in Sect. 2.2, secondary objectives include viscosity on the freshly mixed grout and the following measurements on the raw waste: moisture content, bulk density, and TCLP. Because the TCLP data obtained on the "as-received" waste will be used for comparison with those obtained from the S/S product, the DQOs will be the same as those as described in Sect. 4.1.1. The intended end use of the other measurements is to provide order- of-magnitude estimates of the resulting values. Consequently, DQOs for these measurements are not appropriate and will not be assigned for this project.

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*In this report *ICP* denotes inductively coupled plasma/atomic emission spectroscopy.

4.2 QUANTITATIVE QA OBJECTIVES: PRECISION, ACCURACY, AND COMPLETENESS

All procedures generated during this project which involve data generation and/or acquisition will address the precision, accuracy, and completeness of the method employed. Procedures are prepared and approved as described in Sect. 5.2.1. QA objectives for precision and accuracy (discussed in Sect. 4.1) as well as those for completeness (discussed in Sect. 4.3) are summarized in Table 2.

Analytical data of DQO Level III will be generated following the Modified EPA SW846-1311 TCLP extraction procedure and the SW846-6010A method for analysis by ICP.[13] Mercury analysis will be performed by the cold vapor method [SW846-7470 (ref. 13)]. The data will not be validated, but they all will be reviewed by the analyst prior to reporting to allow spurious data to be detected, flagged, and (if necessary) reanalyzed. This review will check the acceptability of the QC parameters, ensure that calculations are correct, and detect transcription errors.

4.3 COMPLETENESS

Completeness, as used in Table 2, is defined as the value (multiplied by 100 for percent) derived by dividing the number of measurements taken in accordance with procedures specified in this document and resulting in data that meet the QA objectives for accuracy and precision by the total number of measurements taken. Completeness is therefore a measure of acceptable or valid measurements as defined in this section.

4.4 IMPACT OF QA OBJECTIVES

These objectives are specified primarily to guide the principal investigator in procedure development: that is, these objectives define bounds on measurement data, which, if exceeded, require the measurement to be retaken. Failure to meet the QA objectives upon remeasurement does not necessarily mean that the data must be rejected and that corrective action must be taken as described in Sect. 11.

The primary performance objective of this project is to establish the feasibility of S/S as a treatment option for a given waste. As such it must be shown that the S/S product meets LDR requirements as described in ref. 6. Requirements in ref. 6 were taken directly from pertinent regulations, particularly 40 CFR 260 through 265 and 40 CFR 268. Precision and accuracy requirements are not specified in the regulations.

4.5 QUALITATIVE QA OBJECTIVES: COMPARABILITY AND REPRESENTATIVENESS

Comparability, as defined in ref. 2, is "the degree to which one data set can be compared to another." In the context of this project, comparability applies to two areas: (1) intralaboratory and (2) interlaboratory. Intralaboratory comparability will be achieved by use of consistent methods and by traceability of standards to a reliable source as described throughout this document. In the context of this section, MMES is the

Table 2. Quality assurance objectives for data accuracy, precision, and completeness including Data Quality Objective Level III requirements for chemical analysis[a]

Measurement	Method	Reporting units	Precision[b] (%)	Accuracy[c] (%)	Completeness[d] (%)
Free-standing liquid	Modified ANS 55.1	vol %	NA[e]	NA[e]	100
Unconfined compressive strength	ASTM C109 or equivalent	psi	NA[e]	NA[e]	100
TCLP extraction	Modified SW 846-1311	NA	NA	NA	100
Arsenic	SW 846-6010A	μg/L	±20	±25	80
Barium	SW 846-6010A	μg/L	±20	±25	80
Cadmium	SW 846-6010A	μg/L	±20	±25	80
Chromium	SW 846-6010A	μg/L	±20	±25	80
Lead	SW 846-6010A	μg/L	±20	±25	80
Mercury	SW 846-6010A	μg/L	±20	±25	80
Silver	SW 846-6010A	μg/L	±20	±25	80

[a]Only the most common species are listed in this table with respect to Toxicity Characteristic Leaching Procedure (TCLP) analyses. The species of interest are waste code dependent. The precision, accuracy, and completeness presented in this table apply to all TCLP species, independent of waste code.

[b]Precision objectives for free liquid, unconfined compressive strength, and TCLP extraction are not applicable for Phase I considering the need for true variability for the statistical model.

[c]Accuracy objectives for the TCLP concentrations are given as percent recovery of laboratory matrix spikes.

[d]Completeness is based on the number of valid measurements divided by the total number of measurements taken (see Sect. 4.3).

[e]Not applicable, as described in Sect. 4.1.

reference laboratory. Subcontractors will achieve intralaboratory comparability in a manner similar to the reference laboratory, but as described in their approved QAPjP.

Due to budget and schedule constraints associated with this project, it is not feasible for all subcontractors and the reference laboratory to use exactly the same equipment. In some cases, this will also result in using different methods such as American Society for Testing and Materials (ASTM) C39 (ref. 16) or ASTM C109 (ref. 17) for determining UCS. Therefore, interlaboratory comparability will be achieved by duplicating data sets on at least one waste stream at both the subcontractor and reference laboratory.

Representativeness, as defined in ref. 2, is "the degree to which a sample or group of samples is indicative of the population being studied." Determining the representativeness of the "as-received" waste sample to be used in the treatability study by this project, when compared with the waste population it represents, is beyond the scope of this project and will not be addressed in this document. The representativeness of split samples sent to the reference laboratory and those of the subcontractors will be achieved by ensuring that all such samples come from the same field sample. These samples will be homogenized by the laboratories receiving them. The representativeness of the S/S formulations tested, as compared with the population of possible formulations, will be ensured by use of a statistical design to determine formulations to be evaluated. The statistical design will be used by all laboratories participating in this project.

5. PERFORMANCE

5.1 DOCUMENTS AND RECORDS

In this context documents are data, physical samples, plans, procedures, or reports that are being used to support ongoing project activities. Records are any document that provides evidence of results of a completed activity.

5.1.1 Documents

The project leader and the program QAS will determine documents to be managed. Document management will include the identification of documents as well as the designation of the person responsible for storage, the location of storage, and the duration of storage. Guidance is provided in the appendix to this document.

5.1.2 Records

The project leader and the program QAS will determine the records to be managed. Records management will include the identification of records as well as the designation of the person responsible for storage and the location and duration of storage. Guidance is provided in the appendix to this document. The program QAS will identify all records to be considered QA records. All QA records will be maintained by the DDT&E QA records coordinator. QA records will be transmitted to the records coordinator by the project leader with a cover letter detailing the transmitted records. The records coordinator will countersign the letter to signify receipt. The letter will be placed in a technical notebook by the project leader.

5.1.3 Technical Notebooks

Technical notebooks will be managed as described in Sect. 5.2.1.2. Upon completion, technical notebooks become QA records and will be transmitted to ORNL Laboratory Records, where they will be stored for a period of 25 years. The project leader documents the transmittal in a letter to the records coordinator, indicating the notebook number and providing a description of its contents.

5.2 WORK PROCESSES

The principal product of this project is laboratory-scale experimental data. The processes and equipment involved in the generation of these data must be managed and controlled in such a manner as to ensure the validity and quality of the product. This section details the requirements for control of processes and equipment utilized by MMES personnel. Subcontractors working on this project will be subject to control of processes and equipment as described in their respective QAPjPs. Subcontractor QAPjPs are subject to approval as described in Sect. 3.4 of this document.

5.2.1 Plans, Procedures, and Drawings

Plans, procedures, and drawings detailing the methodology for data acquisition will be developed, as appropriate, by the principal investigator. Such documents will be approved by the project leader, the program coordinator, and the program QAS and will be considered an addendum to this document. A listing of all these documents, their storage locations, and the duration of management will be prepared by the project leader. The need for project-specific procedures will become more apparent as this project progresses. However, at a minimum, procedures will be developed which address implementation of laboratory-scale processes, material management, and laboratory-specific entrance requirements. Procedures requiring approval in a manner different from that described here are discussed in the following subsections.

5.2.1.1 Problem Safety Summaries

A problem safety summary (or summaries) will be prepared by the principal investigator prior to initiating experimental work. The safety summary will detail the work processes involved, the potential hazards associated with performing this work, and the safety precautions taken to minimize exposure to personnel. The problem safety summary will be approved by the project leader, the EDS section safety officer, and the EDS section head.

5.2.1.2 Management of Technical Notebooks

All pertinent experimental data will be entered into technical notebooks in accordance with Sect. 2.1.5 of the QAPP. These notebooks will be identified by number and assigned to the principal investigator. The section office will maintain a master list of all notebook numbers and the individual responsible for maintaining the notebook. The project leader will review the notebooks quarterly, at a minimum. This review will be documented by signature and date entered directly into the notebook.

5.2.2 Identification and Control of Items

Each individual assigned to this project is responsible for ensuring that all samples and equipment are used as intended. As used in this section, the term *items* encompasses the following:

- **Laboratory samples and generated data.** Stabilized materials will be generated as part of this project for testing and evaluation. These samples and the subsequent data obtained on or from them will be prepared by MMES personnel and subcontractors. To ensure understanding of the obtained data, a uniform sample identification system will be developed and used by all personnel working on this project. The identification system will be developed and a procedure for its implementation prepared and approved in accordance with the requirements outlined in Sect. 5.2.1.

- **Laboratory equipment.** All major equipment items used in sample preparation and testing will be identified and labeled by the principal investigator. The list of equipment and its location will be maintained by the project leader.

- **Waste material.** Legal requirements are associated with the management of the "as-received" waste as well as with the management of all of the stabilized samples and residues subsequently generated from the received waste. The waste material sample identification code has been addressed previously in this section. The principal investigator will prepare a procedure detailing the methodology for properly managing and controlling these waste materials. The procedure will be approved in accordance with Sect. 5.2.1.

5.2.3 Handling, Storage, and Shipping

The principal investigator will prepare a material management plan detailing the methodology for handling, packaging, and shipping "as-received" treatability study material and resulting residuals. This procedure will be approved in accordance with Sect. 5.2.1.

5.2.4 Control of Measuring and Test Equipment

The principal investigator will specify the means and frequency of calibration for all equipment, when applicable, identified in Sect. 5.2.2. Minimum requirements are described in Sect. 6. A listing of this information will be maintained by the project leader. The listing will clearly differentiate between calibration with primary and secondary standards and will specify which is required. Depending upon the complexity of the calibration methods, a procedure may be deemed necessary. Such a procedure would be initiated and approved as described in Sect. 5.2.1.

5.3 DESIGN

Design control, as it relates to this project, pertains to those processes intended to define, control, and verify technical investigations performed within this project (i.e., treatability studies). This section is intended to supplement, not to replace, requirements detailed in other sections of this document and should not be considered complete in itself.

5.3.1 Experimental Design

The experimental design governing the data to be generated during the treatability studies will be generated by commercially available software packages and is referred to as a mixture experiment. The principal investigator will develop the design constraints, and the project leader will approve them. Utilizing these constraints, the software package will develop multiple experimental designs. The principal investigator will select the appropriate design, which will be approved by the project leader. Verification/validation data points will be a part of the design. The resulting experimental data will be analyzed by the software, and empirical models will be developed. The accuracy of the models will be assessed by (1) statistical methods and (2) review by the principal investigator. The results of this evaluation will be documented by the principal investigator in a research report. The resulting report will be reviewed and approved as described in Sect. 7.2.3.

5.3.2 Software

The software used in the generation and analyses of the experimental design is commercially available and has been used extensively by others. Consequently, the software package will require no validation as to its functionality with respect to its intended use.

5.4 PROCUREMENT

Procurement documents, purchase requisitions, and UCN Form 1127, Request for Individual Consultant or R&D Subcontract, shall provide an adequate description of the scope of work to be performed; specification of applicable design bases; technical, quality, and administrative requirements; and requirements applicable to environmental, safety, health, and security as appropriate. Completed procurement packages on noncapital items must be reviewed and approved by the project leader and section head as indicated by initials or signatures on the procurement document. Procurement packages for capital items (i.e., ≥$5000) must be reviewed and approved by the project leader, section head, and program coordinator. Procurement documents which specify QA requirements will be reviewed and approved by the program QAS and documented by initials or signature on the procurement document.

5.4.1 Accelerated Vendor Inventory Delivery (AVID) System Purchases

AVID purchasing authority for each individual project team member will be specified by the section head. All such purchases related to equipment to be used directly in the acquisition of samples or data must be reported to the project leader.

5.4.2 Receipt Inspection

Upon receipt of purchased items, ORNL Receiving will inspect the items to verify that the number of received items, as labeled or identified, is in accordance with the number of items on the purchase requisition. Verification will be in the form of a receiving report to the requestor. Upon receipt, it is the responsibility of the requestor (generally the principal investigator) to verify the accuracy of the receiving report, the acceptability of the physical condition or appearance of the items, and the conformity to the procurement specifications. The conformance of the item to procurement specifications must be verified no later than 30 days after receipt, unless otherwise stated on the original purchase requisition. Nonconforming items are to be handled in accordance with Sect. 11.2.1.

5.4.3 Documentation

Upon acceptance, the purchase requisition, receiving report, and all related documentation become a project record (see Sect. 5.1). If the item is deemed noncritical to the generation of samples or data (e.g., pencils and paper), then the record need not be kept longer than the time required to verify the correct costs to the project account. If the item or services are critical to the generation of samples or data, then the information

package must be managed as a project record for an extended period of time. When the project leader deems that procured items or services are critical to the generation of samples or data, the information package will be considered a QA record and transmitted to the records coordinator for storage. At a minimum the information shall be stored for the duration of the item's warranty period. Receipt of capital equipment items will be considered QA records and transmitted to the program records coordinator for storage. Both the transmittal and the receipt of the capital equipment QA records will be documented and a copy kept by the project leader for the duration of the project.

6. SAMPLING, ANALYTICAL, AND CALIBRATION PROCEDURES

Numerous procedures will be developed during the course of this project, many as a result of "lessons learned" during experimentation or data reduction. Consequently, the need for procedures cannot be quantified at this time, and flexibility must be maintained. This section provides guidance to the principal investigator as to the required use of standard procedures and identifies technical areas which must be addressed by additional procedures to be developed in accordance with Sect. 5.2.1. It should be noted that previously developed procedures which can be referenced in the literature are considered standards in this context.

6.1 SAMPLING

The quality and reliability of the data generated during the treatability study are dependent on the representativeness, homogeneity, and/or consistency of the samples used for testing. It is recognized that the presence of debris (e.g., rocks) in the "as-received" waste sample may preclude the use of standard procedures. However, the principal investigator will develop procedures or document methods used for the following processes.

- **Achieving homogeneity.** The following sources of samples for testing must be homogenized prior to testing: (1) the "as-received" waste sample (prior to any subsequent testing or treatment), which will be screened to pass through a 4.75-mm sieve; (2) waste forms reduced in particle size to pass through a 4.75-mm-size sieve for subsequent TCLP testing; and (3) TCLP extracts and aliquot subsamples to be analyzed by ICP.

- **Curing of grout samples.** Properties and characteristics of grout waste forms are sensitive to curing time (of sampling in this context) due to the fact that the chemical reactions taking place within the waste form have not been shown to reach thermodynamic equilibrium even after centuries. To ensure consistency between data sets, UCS, drainable water, and TCLP measurements will be taken on grout waste forms after 28 ± 3 days of cure. In addition, the sample size and shape for drainable water measurements (modification of ANS 55.1) will be specified for use throughout the duration of the project.

- **TCLP testing.** The TCLP, as described in SW846 (ref. 13), generates an unacceptable quantity of liquid waste. Consequently, the sample size will be reduced, while keeping the leachate:sample ratio the same as that in the standard. The waste-form sample to be size reduced to pass through a 4.75-mm sieve will be approximately 50 g. The subsample to be subjected to the TCLP will be approximately 10 g. The extract collected from the TCLP test will be homogenized; transferred to an appropriate container; and preserved, as required, for the subsequent constituent analyses.

Table 3 provides recommended preservative, container type, and maximum holding times for typical constituents of this study.

Table 3. Recommended digestion volume, preservative, container type, and holding time for typical analytical measurements of the Toxicity Characteristic Leaching Procedure extracts

Parameter	Recommendation
Digestion volume required	100 or 45 mL
Preservative	HNO_3 to pH <2
Container type	Plastic or glass container rinsed with 1:1 HNO_3 and 1:1 HCl
Maximum holding time	6 months for all metals except mercury, which is 28 days

Source: SW-846, Test Methods for Evaluating Solid Waste, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C., 1986 (updated July 1992).

6.2 SAMPLE CUSTODY

Chain-of-custody (COC) forms accompanying the waste "as received" will be maintained by the principal investigator. Upon return of this material to the generator, a copy of the accompanying COC form will be placed in a technical notebook.

Sample custody will be maintained in accordance with the material management procedure, described in Sect. 5.2.2. Unless required by the material management procedure, additional COC will not be required for transfer of samples between laboratories dedicated to this project (A17, A21, A29, B29, and B33 in Building 4500N and 17 and 19 in Building 4505). Any sample sent for testing and/or analyses external to these laboratories requires a COC. The proper COC will be that used by the Analytical Services Division at MMES.

All samples generated for this project will be properly labeled in accordance with the sample identification procedure, described in Sect. 5.2.2. The label shall follow the sample until it is discarded or returned to the generator.

6.3 ANALYTICAL PROCEDURES

Requirements discussed in this section are applicable solely to performance of the TCLP and subsequent analysis of the extract. The requirements are based primarily on procedures found in SW846. A summary is provided in Table 4 (found at the end of Sect. 6.).

6.3.1 TCLP Extraction Procedure

This extraction will be performed following a modified version of SW846-1311. The modification involves the amount of extraction fluid and the sample weight, as well as the size of the sieve used. The quantities indicated in SW846-1311 are divided by a factor of 10 to minimize the amount of waste generated. Consequently, the extraction will be performed using 10 g of sample and 200 mL of extraction fluid. Because of the use of a much smaller quantity of sample during the modified extraction procedure, the sample will be sieved to pass through a 4.75-mm-opening sieve. The reduced size was chosen to ensure that the sample test is more representative of the original sample.

6.3.2 ICP Analytical Measurement

Prior to analyses, the TCLP extracts need to be digested following the appropriate prescribed method in SW846. The appropriate digestion method for TCLP extracts is SW846-3010A, but a microwave digestion method is also presented in the proposed method, SW846-3015. Any of these digestion methods may be used during this project; however, the microwave digestion method is expected to be routinely used. The digestion method must be recorded by the principal investigator. For samples other than TCLP extracts, the digestion methods are SW846-3010A and SW846-3015 (proposed) for aqueous samples and SW846-3050A and SW846-3051 (proposed) for soils, sludges, and solid waste samples.

TCLP extracts will be analyzed by ICP in accordance with SW846-6010A. SW846-6010A states that "water samples which have been prefiltered and acidified will not need acid digestion as long as the samples and standards are matrix matched": that is, the digestion step may be eliminated provided that the standards used for calibration are prepared in the same TCLP extraction fluid as the analytical samples. Whether the TCLP extracts are digested or the standard is prepared in the TCLP fluid will be determined upon performing a method development. It will be important to verify that the sensitivity of the instrument has not been significantly altered by the TCLP fluid matrix. Also background interferences may increase the baseline to a level which is not acceptable. At the present time, it is assumed that a digestion step is required.

6.3.3 Method Detection Limit (MDL)

The MDL of a constituent is defined as the minimum concentration of the constituent (e.g., arsenic) that can be measured and reported with 99% confidence that the constituent concentration is greater than zero. MDL is determined from analysis of a sample in a given matrix type containing the constituent.

For operational purposes, when it is necessary to determine the MDL in the matrix, the appropriate one-sided 99% *t*-statistic should be multiplied by the standard deviation obtained from a minimum of three analyses of a matrix spike containing the constituent of interest at a concentration 3 to 5 times the estimated MDL. (See Sect. 10.4.)

6.3.4 Instrument Detection Limit (IDL)

The IDL is the lowest concentration of a constituent, above background, that an instrument can reliably detect. For metal analysis, IDL is determined by analyzing seven consecutive measurements of the target constituent at a concentration 3 to 5 times the IDL suggested by the manufacturer, on 3 consecutive days. Three times the average of the standard deviation obtained from the 3 days' analyses defines the IDL.

6.3.5 Cleaning Procedure for Laboratory Glassware and Sample Containers

The glassware used for the preparation of the sample for metal analysis and the bottles containing the analytical samples are required to undergo a special cleaning procedure. The cleaning procedure (as per SW846) is as follows:

> . . . after being cleaned with a low or no-phosphate detergent, the glassware is rinsed abundantly with tap water, soaked in 1:1 nitric acid, rinsed with tap water, soaked with 1:1 hydrochloric acid, rinsed with tap water followed by rinsing with Type II water.[18]

Precleaned containers can be purchased and are QC checked by the vendor prior to shipment to the customer.

6.3.6 Reagents and Standards

All reagents should be analytical grade or better. Water used in the preparation of analytical standard should be ASTM Type I (ref. 18). Standards of ICP quality from two separate vendors will be used. Intermediate standard solutions for instrument calibration will be prepared at least every month for concentrations above 1 mg/L. Intermediate standard solutions with concentrations below 1 mg/L should be prepared at least twice a month and should be monitored daily when the ICP is active.

6.4 CALIBRATION PROCEDURE AND FREQUENCY

As in Sect. 6.3, requirements presented in this section pertain to performing the TCLP and subsequent leachate analyses by ICP. The requirements are consistent with SW846 requirements for a DQO Level III project.

When applicable, all primary instruments must be calibrated prior to use as a measurement device by comparison with a known reference material or instrument. All sample measurements must be made within the calibrated range of the instrument. If the calibration process reveals that an instrument is outside the specified range of acceptability, it should not be used until adequate measures are taken to correct the problem and a valid calibration is obtained. Had data been acquired with an instrument potentially outside of the acceptable calibration range, an evaluation should be made of the impact on the quality of the data generated and eventual measures should be taken to correct or flag these data.

6.4.1 Calibration of Standard Laboratory Equipment

Standard laboratory equipment should be calibrated as follows.

- **Thermometer.** The calibration should be performed by comparing the readings of the thermometer with the readings of a certified thermometer at two temperatures. The difference between the two readings for each temperature should not exceed 2°C. This calibration should be performed before the first use and then annually thereafter. (Certified thermometers are excluded.)

- **Balance.** Prior to first use and then semiannually thereafter at a minimum, mass balances will be calibrated by Instrumentation and Controls (I&C) Division personnel against a set of certified Class S weights.[19] Certification by I&C personnel will be denoted by a label on the balance, with records of the calibration kept by I&C Division. The user will perform one check (consisting of two weights) each day that the balance is in active use. Because this calibration is performed only when the balance is used, calibration records will not necessarily exist for each workday. The calibration records will be kept in a balance logbook, one for each balance.

- **pH meter.** The instrument will be calibrated at least once a day while it is actively in use. The pH meter will be calibrated using two pH standards in the range of the expected measures. The calibration will be verified using a third pH standard. These pH standard solutions are readily available commercially.

- **Piston plunger operated volumetric apparatus (pipettes).** The pipettes will be calibrated using a gravimetric method.[20] A beaker will be placed on the plateau of an analytical balance, and ten measurements of the same quantity of water will be added in a beaker. The weight corresponding to these ten individual measurements will be recorded. The volume is calculated based upon the knowledge of the density of water at specific temperatures and corrections for air buoyancy. The calculated volume should be in the range of acceptability provided by the manufacturer of the pipette. When used for analytical work, the pipettes should be calibrated quarterly.

6.4.2 Calibration of the ICP

The calibration requirements discussed in this section (further detailed in Sects. 6.4.2.1 and 6.4.2.2) are based on the manufacturer's recommendations and the requirements of SW846-6010A. The discussion details several procedures which must be repeated if data are outside specified limits. Initiation of this repetition is considered part of the routine calibration procedure, and its occurrence does not constitute the need for corrective action as described in Sect. 11.

All measurements must be within the instrument linear range where spectral interference correction factors are valid. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain QC data confirming instrument performances and analytical results. The analyst will check for interferences, correct them when possible, or document them when they cannot be corrected.

Calibration standards are prepared containing all metals for which analyses are requested. Triplicate burns will be performed for each sample or QC parameter, and the average value will be used. During active use, the ICP will be calibrated prior to each analytical run or every 24 hours, whichever comes first.

6.4.2.1 Initial Calibration

One blank and one standard sample are analyzed for determining the calibration curve. The concentration of the standard for each constituent should be at a level encompassing the anticipated range of measurement in the analytical samples or at a level recommended by the manufacturer for the specific instrument. Following the calibration, the highest concentration is reanalyzed as a sample, and its result should agree within 5% of the original value.

Immediately following the calibration, an initial calibration verification (ICV) and a subsequent initial calibration blank (ICB) are analyzed. The ICV should be from an another source (e.g., another vendor) than the original standard and should be within ± 10% of the true value. The ICB value should be below the reporting limit. If these conditions are not met, the standardization should be repeated until these two controls are within acceptable limits.

The ICP interference check standards A and B (ICSA and ICSAB) verifying the absence of spectral interferences will be analyzed for each run or every 8 hours, whichever comes first. The initial ICSA and ICSAB are performed following the first ICV and ICB and must also be performed at the end of the run before the final continuing calibration verification (CCV) and continuing calibration blank (CCB) (see Sect. 8).

6.4.2.2 Continuing Calibration

The initial calibration is verified on a regular basis during the run. For every ten analytical samples, a CCV and a CCB are analyzed. The CCV value should agree within 10% of the true value. The CCB should be below the reported detection limit for each analyte; however, if a blank is found to be slightly contaminated, the analyst might still accept it considering all the parameters of the analysis. If these conditions are not fulfilled, the analysis should be stopped, the instrument should be recalibrated, and the analytical samples following the last valid calibration should be reanalyzed.

Table 4. Summary of standard methods used as operating procedures

Method	Method subject[a]	Method type	Reference no.
Standard Method 2540. G	Determination of the moisture content	Gravimetric	21
ASTM C305-91 and C109-92	Mixing and casting of the sample molds	—	22, 17
ASTM C109-92[b]	Unconfined compressive strength	Physical testing	17
ANS 55.1[c] or ASTM C940-89	Free-standing liquid determination	Volumetric	14, 15
EPA Method SW846-1311[c]	Toxicity Characteristic Leaching Procedure	Leaching	13
EPA Method SW846-3010A	Acid digestion of aqueous samples and extracts for total metals for analysis by FLAA or ICP spectroscopy	Digestion	13
EPA Method SW846-3050A	Acid digestion of sediments, sludges, and soils	Digestion	13
EPA Methods SW846-3015 and SW846-3051 (proposed)	Microwave-assisted digestion (proposed)	Digestion	13
EPA Method SW846-6010A	ICP	ICP	13

[a]FLAA = flame atomic adsorption; ICP = inductively coupled plasma (atomic emission spectroscopy).

[b]Or equivalent.

[c]Modified as described in this document.

7. DATA REDUCTION, VALIDATION, AND REPORTING

Process and product inspections, as normally defined for manufactured items, will not be applicable to this project. Only data and documentation from technical analyses will be routinely generated. Inspection of new equipment used in the generation of data is addressed in Sect. 5.4.2. Surveillance and/or audit, when required, will be conducted in accordance with Sect. 9.

7.1 DATA REDUCTION AND ACCEPTANCE

The principal product from this project is data. Acceptance testing, as it relates to this project, involves testing and acceptance of the equipment utilized in the generation of the data and the data themselves. Test procedures (i.e., the methodology used in conducting the test) will be developed by the principal investigator in accordance with Sect. 5.2.1.

7.1.1 Equipment Operation

Measuring and test equipment will be calibrated and maintained in accordance with Sect. 5.2.4. Test procedures will specify the method and frequency of secondary calibration. Performance of this acceptance testing is the responsibility of the principal investigator and will be documented in a notebook.

7.1.2 Experimental Design

Experimental design is an iterative process dictated by both objective design constraints and subjective experience of the personnel developing the design. The rationale for the selected design and for decisions leading to that design shall be documented by the principal investigator. Documentation will consist of a letter from the project leader to the program coordinator.

7.2 DATA REVIEW AND VALIDATION

The data generated during Phase I of this project will not be validated following technical review protocol defined in EPA Laboratory Data Validation Functional Guidelines. However, the data will be reviewed as described in this section.

7.2.1 Data Validation

All analytical data will be checked by the individual generating the data to ensure that all QA/QC requirements are met. The principal investigator or project leader will review the data as well as the calculation (i.e., data reduction) prior to use in the statistical design. If data are found to be outside the specified control limits, the principal investigator will clearly flag these data to notify the user.

Data generated by this project will be entered into a computer data management system for subsequent analyses (i.e., data reduction). It is the responsibility of the principal investigator to verify the accuracy of the transcription of the raw data (from technical notebooks, etc.) to the data management system. Verification will be documented by a letter to the project leader accompanying the electronic data file containing these data.

7.2.2 Data Analyses

Data analyses will consist primarily of the generation of empirical models to allow interpolation of the data. The precision of the models will be reported, and the rationale for determining the precision will be documented. Documentation will be in the form of a research report, which will be prepared in accordance with Sect. 7.2.3.

7.2.3 Reports

Ultimately the experimental data, their reduction, and the validity of the data reduction will be detailed in a document which will undergo a process known as technical review. These documents may take the form of a letter report, a research report, or an external publication. Preparation of the report is the responsibility of the principal investigator. The technical review must be performed by a minimum of two persons who are independent of the work performed and are qualified to judge the technical adequacy of the material contained in the report. These reviewers will be selected by the project leader and the program coordinator and approved by the section head. The review will be documented on a technical review form (Fig. 3). The principal investigator is responsible for resolving all technical comments received. The section head or his designee will resolve any disputed comments. After technical comments are resolved, the technical review forms will become QA records. At a minimum, these records will be maintained by the program records coordinator until the subject document has been published.

7.3 DATA REPORTING

Documents meeting the requirements of Sect. 7.2.3 may be released or published. Documents to be released or published will be selected by the project leader or the program coordinator. These documents may take the form of a letter report, a research report, or an external publication. Prior to release, the document is to (1) undergo editing and (2) be approved by the project leader, the program coordinator, and the section head, as documented by initials or signatures on the Document Clearance form (UCN-10369).

As an exception, the Program Manager[7] may release documents in draft form prior to completion of all steps delineated in Sects. 7.2.3 and 7.3. In order to exercise this exception, the program manager or his designee is responsible for implementing the following steps prior to the release of the draft report: (1) the word "DRAFT" will be prominently displayed on each page of the document; (2) the status of the document and its intended use will be detailed on the document cover page; (3) the document release will be approved by the project leader, the section head, the program coordinator, and the program manager as documented by initials or signatures on the Document Clearance form; and (4) a copy of the document and its distribution will be forwarded to the

EDS DOCUMENT REVIEW

To:

From:

Subject: Please review the attached document for clarity, technical accuracy and objectivity, based on its class. Feel free to comment on the makeup and mode of presentation, but you need not be concerned with minor grammatical errors (and the like) since the document will be submitted for editorial review.

I would greatly appreciate your completing the review within two weeks and returning the completed form to me. Thank you.

Title:

Authors:

Class: ____ Memo (CF, etc.) ____ ORNL/TM ____ ORNL-

____ External Publication

REVIEWER'S RECOMMENDATIONS

This report should be submitted for publication

_____ Unchanged

_____ After minor revision (as indicated on returned report)

_____ After major revision (see comments)

Comments (use additional pages if necessary):

_____

Reviewer Date

Fig. 3. Technical review form.

program records coordinator to be maintained as a QA record. The record will be maintained until (1) the distributed copies are returned or (2) the document has been released in accordance with Sects. 7.2.3 and 7.3.

8. INTERNAL QUALITY CONTROL CHECKS

This section is intended to provide guidance to the principal investigator, who is responsible for developing and implementing procedures as described in Sect. 5.2.1. Therefore, this section specifies procedures or procedural requirements, where possible, and highlights areas where additional procedures are required.

8.1 ADMINISTRATIVE

EDS personnel selected by the section head will perform quarterly safety inspections. The section head may initiate self-assessments on any pertinent topic. In addition, technical audits sponsored by Energy Systems senior management, audits sponsored by Martin Marietta Corporate, and audits sponsored by DOE may be performed and scheduled independent of this project. The findings of all such inspections will be reported to the project leader. The project leader will report to the program QAS any finding deemed to affect the quality of this project. All findings will be subject to corrective action requirements as described in Sect. 11.

The project leader will "walk his space" at least once a week to verify that experimental work is being performed in accordance with this plan. Results of these inspections will be discussed at routine project team meetings at least once a month. Significant findings will be reported to the program QAS and the section head.

It is the responsibility of all employees to ensure that individual tasks performed in support of this project are executed in a manner consistent with this QAPjP. If a problem is identified that has the potential to seriously affect the quality of work or the health and safety of the worker or to adversely impact the environment, all project participants have the authority to immediately shut down operations.

8.2 QC CHECKS FOR PHYSICAL TEST MEASUREMENTS

8.2.1 Replicates

For the physical tests of UCS and free-standing liquid, replicate samples will be prepared and measured to determine the precision achieved with the instruments or method. In this project, the various tests are performed to feed a statistical design. The statistician sets up 3 replicates among the 16 to 20 formulations that will be tested for each waste stream. These replicates are true replicates for the same formulation: two different blends, mixes, and pours will be prepared.

8.2.2 Blank and Spike

There are no spiking requirements or blank measurements for the physical tests.

8.3 QC CHECKS FOR THE TCLP EXTRACTION PROCEDURE

8.3.1 Blank

The TCLP procedure followed in Phase I is a modification of the SW846-1311: 10 g of sample will be extracted in 200 mL of extraction fluid, with <4.75-mm particles used for the extraction. The containers used for the extraction will be 250-mL nalgene bottles, which will be discarded after each use. Therefore, the criterion for the TCLP blank will be to run a blank (only the extraction fluid without sample) every batch or every 20 samples (whichever comes first). In the event that both fluids are used in the same batch, two blanks will be run, one with each fluid.

8.3.2 Matrix Spike

A matrix spike will be performed for each waste type, and a minimum of one matrix spike will be analyzed per analytical batch (i.e., a group of samples which behave similarly with respect to the sampling or the testing procedures and which are processed as an unit). The spike will be added after filtration of the TCLP extract and before sample preservation.

8.4 QC CHECKS FOR ICP ANALYSES

8.4.1 Duplicates

For analytical data, a duplicate consists of two aliquots from the same sample which will undergo the same process (as the sample) for digestion and subsequent analyses. The frequency of duplicates will be 5%, that is, one duplicate for every 20 samples or one duplicate for every analytical batch, whichever is greater. The acceptance criterion is to obtain a relative percent difference (RPD) which agrees within 20% of the original value.

8.4.2 Spikes

In accordance with SW846, analytical data generated by ICP require the following types of spikes: (1) matrix spike (MS) and matrix spike duplicates (MSDs) and (2) spiked blanks (i.e., ICV, CCV). The procedure for each type of spike is discussed in this section.

8.4.2.1 Matrix Spike and Matrix Spike Duplicate

An MS is an aliquot of a sample which is doped (spiked) with a known amount of each of the analytes of concern and which undergoes the same preparation procedure as the actual sample (e.g., digestion and analysis). The effect of the sample matrix on the analytical method can be monitored by calculating the percent recovery of each analyte. The acceptance is ± 25% of the known value. An MSD can also be used to check the precision instead of analyzing a duplicate sample. The frequency rate is 5% or one spike and/or spike duplicate for every 20 samples or every analytical batch, whichever is greater. There is no need to spike for Ca, Mg, K, and Na when analyzing water samples and for

Al, Ca, Fe, Mg, K, and Na when analyzing soil samples.[23] Also, if the concentration of the analyte in the sample is greater than 0.1%, no spike of the analyte is necessary.

8.4.2.2 Instrument Calibration Verification

At the beginning of each run following the calibration, a standard of known concentration (ICV) whose origin is different from that used for the calibration is analyzed. This ICV should agree within 10% of the actual value.

8.4.2.3 Continuing Calibration Verification

Every ten samples, in order to verify the calibration, a spiked blank of known concentration (CCV) is analyzed. The concentration of the CCV should agree within 10% of the expected known concentration. A final CCV should also be performed at the end of an analytical run.

8.4.2.4 Laboratory Control Sample (LCS)

An LCS is a sample of known concentration which is processed through the entire preparation and analysis procedures to ensure that the methods are providing good and accurate performance independently of the matrix. The measured concentration of the LCS should agree within 20%, with the exception of antimony and silver, which do not have limits.[23]

8.4.2.5 QC Sample

A QC sample should be prepared in the same matrix as the calibration standards at 10 times the IDL and in accordance with the instructions provided by the manufacturer. No acceptability criterion exits for this parameter.

8.4.3 QC Parameters

8.4.3.1 Linear Range Analysis Standard

A verification of the linear range is performed quarterly. All the samples analyzed during this time should have concentrations that do not exceed this upper limit; otherwise, they should be diluted and reanalyzed. The result of the upper limit concentration of the linear range should agree within 10% of the known value.

8.4.3.2 Interference Check Standards A and B

ICSA and ICSAB have to be performed at the beginning and at the end of each run (after the initial ICV and ICB and before the final CCV and CCB) or every 8 hours, whichever comes first. ICSA and ICSAB are analyzed to verify the absence of spectral interferences. The measured concentrations should agree within 20% of the established mean value. If the recovery is outside the 80-120% window, the analyze should be terminated, the problem corrected, the ICP recalibrated, and the samples reanalyzed.

Termination and restart under these circumstances are considered routine procedure and do not require corrective action as discussed in Sect. 11.

8.4.3.3 Interelement Correction

Correction factors for spectral interference due to Al, Ca, Fe, and Mg (at a minimum) will be performed annually for all wavelengths used for each analyte reported by ICP.

8.4.3.4 Serial Dilution

Once for each new matrix or when an unusual matrix is encountered, a fourfold dilution of a sample should be performed. The results of this dilution should agree within 10% of the original determinations for all analytes if they are in concentrations >10 times the IDL after dilution. This dilution is performed to ensure that no problem exists with chemical and/or physical interference for the matrix considered.

8.4.3.5 Postdigestion Spike Addition

Once for each new matrix or when an unusual matrix is encountered, a postspike should be performed. An analyte spike added to a portion of a prepared sample or to its dilution should be recovered to within 75% to 125% of the known value. Like the serial dilution, the postaddition is performed to ensure that no problem exits with chemical and/or physical interference for the matrix considered.

8.4.3.6 Performance Evaluation (PE) Samples

Known QC check samples or PE samples obtained from an outside source (EPA, the National Institute of Standards and Technology, or a commercial vendor) with concentrations provided by the vendor should be run at least once during the project. A certified standard reference material should be run twice a month to verify the accuracy of the calibration and standards. This reference material does not need to contain all the analytes to be analyzed by the instrument.

8.4.4 Blanks

8.4.4.1 Preparation Blank (PB)

The PBs are subjected to the same preparation and analysis steps as normal analytical samples. One PB should be prepared for every batch of samples or for every 20 analytical samples, whichever is first. The PB is an important indication of any contamination occurring during the analysis process. If one PB is found to be grossly contaminated, the samples might be reprepared, especially if the samples show the same contamination in analyte and concentration as the PB.

8.4.4.2 Calibration Blanks (ICB and CCB)

ICB and CCB are calibration blanks consisting of deionized water containing the same amount of acid as the analytical samples. Their purpose is to confirm the calibration and to prove that the blank reading is below the reporting limit. The ICB is analyzed (1) at the beginning of the run following the initial ICV and CCB, (2) after the CCV for every ten analytical samples, and (3) at the end of the analytical run. The acceptance for ICB and CCB is to obtain concentrations below the reporting limit. If one blank value is over the reporting limit, the analysis should be terminated, the ICP should be recalibrated, and the samples following the last valid blank should be reanalyzed. The need for repeat analyses under these circumstances is considered routine procedure and does not require corrective action as discussed in Sect. 11. Table 5 provides a summary of the QC indicators for the ICP analysis. However, the analyst may decide to accept a contaminated (i.e., concentration above detection limit) blank if this acceptance does not impair the final quality of the data.

Table 5. Performance indicators for the inductively coupled plasma method

Performance indicator	Frequency of analysis	Criterion
Highest standard	Immediately after calibration	95-105% of the true value
Initial calibration verification (ICV)	Immediately after the highest standard	90-110% recovery
Initial calibration blank (ICB)	Immediately after ICV	Below reporting detection limit
Interference check samples (ICSA and ICSAB)	Immediately after ICB	80-120% recovery (ICSAB)
Quality control sample	Analyze after ICSAB, concentration 10 times the IDL[a]	None
Continuing calibration verification (CCV)	Analyze every 10 analytical samples	90-110% recovery
Continuing calibration blank (CCB)	Immediately after each CCV	Below reporting detection limit
Linear range standard	Quarterly	Not applicable
Interelement correction	Annually	Minimum: Al, Ca, Fe, and Mg
Preparation blank (PB)	One per digestion batch or every 20 samples	Below reporting detection limit
Laboratory control sample (LCS)	One per digestion batch or every 20 samples	80-120% recovery, except Ag and Sb
Matrix spike (MS)	One per digestion batch or every 20 samples	75-125% recovery
Duplicate (dup) or (MSD)	One per digestion batch or every 20 samples	≤ 20% RPD[b]
Postaddition spike (PS)	One for every new or unusual matrix	75-125% of the known value
Serial dilution (DL)	One for every new or unusual matrix	90-110% recovery if concentration of analyte is >10 × IDL

[a]Instrument detection limit.
[b]Relative percent difference.

9. PERFORMANCE AND SYSTEMS ASSESSMENTS

This section describes the methods and policies designed for planning, performing, and reporting audits and surveillance activities to verify compliance with all aspects of the QAPP[7] and to determine its effectiveness. Audits and surveillances, as described in this section, represent an independent assessment by program management and are in addition to routine line management assessments described in Sect. 8.1.

9.1 SURVEILLANCES

Surveillances[24] are narrower in scope than audits and are designed to provide a detailed look at a specific area of interest. The program coordinator is responsible for establishing a surveillance schedule for this project in consultation with the program QAS and project leader. Surveillances will be conducted by the program QAS and by other personnel technically competent in the area of interest who have been selected by the program coordinator. All findings will be reported to the project leader, who will initiate corrective action in accordance with Sect. 11. Surveillances shall be performed by using project/program documents and highlighting the items to be verified and/or compiling checklist questions. Surveillance results shall be documented in a report that will be issued on a timely basis by the program QAS. Copies of the report will be distributed to the program manager,[7] program coordinator, section head, and project leader.

9.2 AUDITS

Audits[25] are potentially broad in scope and may address any and all areas of the QAPP[7] and this document. The program coordinator is responsible for establishing an audit program for this project in consultation with the program QAS and project leader. Audits will be conducted by the program coordinator or his designee. All findings related to the audit will be reported to the project leader, who will initiate corrective action in accordance with Sect. 11.

9.3 SUBCONTRACTORS

Audits and surveillances will be conducted on subcontractors supporting this project in the manner described in Sects. 9.1 and 9.2. However, corrective action will be initiated in accordance with the subcontractor's approved QA plan and is subject to approval by the program QAS, the project leader, and the section head.

9.4 SCHEDULE

The schedule and frequency of audits and surveillances will be determined by the program QAS and/or the program coordinator. The minimum frequency of audits and surveillance for this project is as follows.

- A surveillance of project activities within MMES will be conducted quarterly, at a minimum, by the program QAS or his designee. Surveillance of project activities being conducted by subcontractors will be performed at a frequency determined by the project leader.

- Audits of project activities within MMES and subcontractor activities involving experimental work will be conducted annually, at a minimum, by the program QAS or his designee.

10. CALCULATION OF DATA QUALITY INDICATORS

Numerous terms to be used during this project to quantify data precision, data accuracy, and completeness are discussed in Sects. 4, 6, and 8. This section provides the principal investigator with guidance on calculating these terms by detailing their definitions in equation form.

10.1 DATA PRECISION

If data precision is calculated from duplicate measurements, relative percent difference is the normal measure of precision:

$$RPD = \frac{(C_1 - C_2) \times 100\%}{\left(\frac{C_1 + C_2}{2}\right)} ,$$

where

RPD = relative percent difference,
C_1 = larger of the two observed values for the concentration or measurement,
C_2 = smaller of the two observed values for the concentration or measurement.

If data precision is calculated from three or more replicates, the relative standard deviation should be used in lieu of the relative percent difference:

$$RSD = (s/\bar{y}) \times 100\% ,$$

where

RSD = relative standard deviation,
s = standard deviation,
$\bar{y}$ = mean of replicate analyses.

Standard deviation is defined as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n - 1}} ,$$

where

s = standard deviation,
y_i = measured value of the ith replicate,
$\bar{y}$ = mean of replicate measurements,
n = number of replicates.

The calculation of the mean of replicate measurements is as follows:

$$\bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i ,$$

where

$\bar{x}$ = mean of replicate measurements,
n = number of measurements,
x_i = measured value of the ith replicate.

Another estimate of precision is given by the coefficient of variation, which is calculated as follows:

$$CV = 100 \times \frac{S}{\bar{x}} ,$$

where

CV = coefficient of variation,
$\bar{x}$ = mean of replicate measurements,
$\bar{S}$ = variance.

For measurements such as pH, where the absolute variation is more appropriate, precision is usually reported as the absolute range, D, of duplicate measurements:

$$D = |m_1 - m_2| ,$$

where

D = absolute range,
m_1 = first measurement,
m_2 = second measurement.

10.2 DATA ACCURACY

For measurements where matrix spikes are used, the percent recovery is calculated as follows:

$$\%R = 100\% \times \left[\frac{S - U}{C_{sa}}\right],$$

where

$\%R$ = percent recovery,
S = measured concentration in spiked aliquot,
U = measured concentration in unspiked aliquot,
C_{sa} = actual concentration of spike added.

10.3 COMPLETENESS

Completeness is defined as follows for all measurements:

$$\%C = 100\% \times \left[\frac{V}{n}\right],$$

where

$\%C$ = percent completeness,
V = number of measurements judged valid,
n = total number of measurements necessary to achieve a specified level of confidence in decision making.

10.4 METHOD DETECTION LIMIT (MDL)

MDL is defined as follows for all measurements:

$$MDL = t_{(n-1,\, 1-\alpha = 0.99)} \times s,$$

where

MDL = method detection limit,
s = standard deviation of the replicate analyses,
$t_{(n-1,\, 1-\alpha = 0.99)}$ = Student's t value for a one-sided 99% confidence level and a standard deviation estimate with $n - 1$ degrees of freedom (see Table 6).

Table 6. Student's t value for a one-sided 99% confidence level

Number of samples	t-statistic
4	4.541
5	3.747
6	3.365
7	3.143
8	2.998
9	2.896
10	2.821
11	2.764

Source: R. H. Perry and C. H. Chilton, *Chemical Engineers' Handbook*, 5th ed., McGraw-Hill, New York, 1973.

11. CORRECTIVE ACTION

Problem identification will be accomplished by audits, surveillances, self-assessments, managers "walking their space," section safety inspections, inspection by MMES support organizations, and observation by all project personnel. All problems identified will be reported to the project leader, who is responsible for initiating the appropriate corrective action. If a problem is identified that has the potential to seriously affect the quality of work or the health and safety of the worker or to adversely impact the environment, all project team members have the authority to immediately shut down operations.

11.1 CATEGORY 1, 2, OR 3 OCCURRENCE

If the project leader determines or suspects that the problem has the potential for meeting the criteria of a category 1, 2, or 3 occurrence, he will immediately inform the section head. The section head determines whether the situation meets the criteria for a category 1, 2, or 3 occurrence. If it meets these criteria, then the reporting and corrective action requirements of MMES procedure ES-OP-300, Occurrence Reporting System (ORS),[26] will be followed. The project leader will inform the program coordinator and program QAS of any problem meeting these criteria.

11.2 OTHER CORRECTIVE ACTIONS

11.2.1 Procedures for Corrective Action

All problems identified which do not meet the criteria in Sect. 11.1 will be subjected to corrective action as described in this section. Problems identified by personal observation may be communicated to the project leader orally and/or in writing (e.g., inspection reports). All problems identified in writing require a written response. Regardless of the method of identification, corrective action will take one of two forms depending on the seriousness of the problem. Problems identified which do not immediately jeopardize project quality and which can be corrected within 24 hours can be corrected by the project leader or his designee. Such problems are considered routine, and reporting of the corrective action is not required unless the problem has been identified in writing. Problems which have the potential to immediately jeopardize project quality or which cannot be corrected within 24 hours must be reported using the Corrective Action Report and Status (CARS) form, shown in Fig. 4. The project leader is responsible for supplying the following information on the CARS form: (1) problem description, (2) appropriate corrective action, (3) individual responsible for corrective action, and (4) schedule for completion of the corrective action. The section head and the program QAS are responsible for approving the corrective action and schedule. Verification of completed corrective action is performed by the program QAS and the section head or his designee. The section head will forward the CARS form to the CTD Office of Safety and Operational Readiness, which will document closure. The project leader will forward a copy of the closure documentation to the program records coordinator.

CORRECTIVE ACTION REPORT AND STATUS

CORRECTIVE ACTION SOURCE Initiation Date: _____

(Specify): _____ _____ _____

(Audit #, NRC #, Surveillance #, Occurence Report #, or Other) POC Risk Category

FINDINGS AND/OR RECOMMENDATION:

Applicable to Other Areas? (YorN) _____ _____ _____

Section Head QAS

CORRECTIVE ACTION OR ACTION TO PREVENT RECURRENCE:

RESPONSIBLE PERSON(S): _____ Distribution:

Sched. Completion Date: _____ Revised Completion Date: _____

Commit to Corrective Action: _____ _____

Division/Program Manager/Director Date

Accept and Understand Task: _____ _____

Responsible Person Date

Concurrence: _____ _____ _____ _____

Division/Program QAS Date Data entry Date Tracking Number

STATUS:

CORRECTIVE ACTION COMPLETED: _____ _____ Distribution:

Responsible Person Date

Verified ___ Acknowledged ___ _____ _____

QAS Date

Closed: _____ _____

Date Entered in Database By Whom

Fig. 4. Corrective Action Report and Status form.

The status of completion of corrective actions will be monitored and tracked by the program QAS in accordance with requirements specified in the QAPP.[7] The project leader will utilize the CTD Office of Safety and Operational Readiness for logging and tracking corrective action on the problem.

11.2.2 Nonconformances

A nonconformance is a special category of problem which is applicable to items or processes that do not conform to approved drawings or specified requirements. Nonconformances typically apply to items that are not yet installed or put into service, such as recently purchased equipment. The nonconforming item is to be identified through marking, labeling, or tagging and should be documented in a nonconformance report[27] on form UCN-11457. Nonconforming characteristics are to be reviewed and evaluated by the principal investigator or his designee. Any remedial or corrective actions required will be identified and approved by the project leader. In the context of this project, the most likely source of nonconformances is purchased items delivered damaged or in a nonoperating condition.

11.3 SUBCONTRACTOR CORRECTIVE ACTION PROCESS

Subcontractors performing activities associated with this project will be subject to corrective action processes as described in their approved QAPjP. Corrective actions for problems identified by a surveillance or audit conducted by MMES project personnel require approval by the project leader, the section head, and the program QAS in addition to that specified in the subcontractor's QAPjP.

12. QUALITY CONTROL REPORTS TO MANAGEMENT

The program QAS will report all quality-related activities to the program coordinator on a monthly basis. The project leader will provide input for this report. At a minimum, this input will include a copy of all inspection reports, corrective action forms, and the status of open corrective actions.

In addition, project reports will contain pertinent QA information relevant to the precision and accuracy of the contained data. These reports will be approved as described in Sect. 7.3.

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Appendix. IDENTIFICATION OF PROJECT DOCUMENTS AND QA RECORDS

Table A.1. Project documents and quality assurance records

Document	Retention period	Master file point	Duplicate file point	QA record[a]
Project QA plan	Lifetime[b]	Project leader	QAS[c]	Yes
Problem safety summary	Lifetime	Section office	Project leader	
Experiment plans and statistical designs	Lifetime	Principal investigator	Project leader	Yes
Procedures	Lifetime	Principal investigator	Project leader	Yes
Occurrence reports	Lifetime	CTD OSOR[d]		Yes
Audit and surveillance reports	Lifetime	Project leader		Yes
Nonconformance reports	Lifetime	Principal investigator	Project leader	Yes
Corrective Action Report and Status forms	Lifetime	CTD OSOR[d]		Yes
Index of procedures	Lifetime	Project leader		
Purchase requisition and receipt documentation	Warranty period[e]	Principal investigator		Yes[f]
Capital equipment requisition and receipt documentation	Lifetime	Project leader		Yes[f]
Laboratory notebooks	Lifetime	Principal investigator		Yes[g]
Experimental data	Lifetime	Principal investigator	Project leader[h]	
Index of calibration requirements	Lifetime	Project leader		
Calibration records	Lifetime	Principal investigator		Yes
Treatability study logs	3 years	Principal investigator	Project leader[i]	Yes
Training records	3 years	Section office		
Technical review comments on publications	Until published[j]	Principal investigator		
Statement of work	Lifetime	Project leader		Yes

[a]Document will be transferred to program records coordinator when it is no longer in active use.
[b]Lifetime of project.
[c]DDT&E program quality assurance specialist.
[d]Chemical Technology Division Office of Safety and Operational Readiness.
[e]Documentation will be maintained for the duration of the item's warranty period. For nonwarranty items, documentation will be maintained only until account cost information is verified.
[f]If equipment or services specify quality assurance requirements.
[g]When it is no longer in active use, the notebook will be transferred to Laboratory Records, not to the program records coordinator.
[h]Project leader will maintain only duplicates of electronic data which are sent to subcontractors.
[i]Project leader will maintain only duplicates of information for annual treatability report.
[j]Comments will be maintained until principal investigator initiates Document Clearance form, at which time the comments will be transferred to the section head.

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