QUALITY ASSURANCE CONSIDERATION FOR CEMENT-BASED GROUT TECHNOLOGY PROGRAMS AT OAK RIDGE NATIONAL LABORATORY

E. W. McDaniel, T. L. Sams, D. B. Delzer, O. K. Tallent

Oak Ridge National Laboratory*
Oak Ridge, Tennessee 37831

ABSTRACT

The Oak Ridge National Laboratory (ORNL) has developed and is continuing to refine a method for immobilizing low-level radioactive liquid wastes by mixing them with cementitious dry-solid blends. Currently, ORNL is investigating a grouting facility that will pump a liquid grout/waste mixture to a disposal site where the mixture will harden in situ, forming a grout monolith. A quality assurance (QA) program is vital to the project because Nuclear Regulatory Commission (NRC), Environmental Protection Agency (EPA), and state environmental regulations must be demonstrably met; that is, the work must be defensible in a court of law. The end result of QA is, by definition, a product of demonstrable quality. In the laboratory, this entails traceability, repeatability, and credibility. This paper describes the application of QA in grout technology development at ORNL.

INTRODUCTION

Quality assurance is defined as all those planned and systematic actions which are necessary to provide adequate confidence that a system will perform satisfactorily in service or, when the product is an investigation or study report, will provide confidence in the validity and integrity of the data, methods, and procedures and in the protection, retrievability, and replicability of the data. It must be a vital and integral part of any waste form development program, from inception, in order to ensure compliance with NRC, EPA, and state regulatory criteria and to merit public confidence. Activities associated with QA include quality control (QC), peer review (for experimental work), and auditing.

At ORNL, cement-based waste forms (grouts) have been used for a number of years to immobilize low-level waste (LLW) with little thought of using the experimental data for permitting purposes. Although ORNL claimed exclusion of regulatory agencies under the Atomic Energy Act of 1954, it did have a quality program that complied with Department of Energy (DOE) Order 5700.6A.(1)

In 1984, a federal court ruled that environmental jurisdiction of the Oak Ridge Reservation was the responsibility of the state of Tennessee. This court order then made it imperative that ORNL's quality programs satisfy not only the DOE requirements but also those of the NRC, the EPA, and the state of Tennessee.

To accomplish these goals, the Grout Technology Development Program at ORNL has structured its QA program around the following areas that are consistent with ANSI/ASME NQA-1:(2) 1) control of materials; 2) record keeping; 3) peer review, documentation, and reporting; 4) data generation, collection, and analysis; 5) training and certification of personnel; 6) instrument calibration; and 7) internal and external audits.

Although cement-based waste forms have been used for some time at ORNL, the disposal of LLW in grout monoliths is still in a developmental state. Technical standards and procedures have been evolving throughout the work as the data base enlarges and regulatory criteria change. In a case such as this, when technical criteria are being developed concurrently with technology, peer review is an absolutely essential part of QA. Research and development work contrasts with design and fabrication in that the results of the work are not controlled so much as they are discovered. Judgment must provide the review criteria; QA in research and development will often depend on the collective agreement of scientists/technical personnel.

The QA/QC inputs to analytical operations and procedures are more easily defined than the overall QA requirements for research and development, although continuing surveillance and peer review do have a proper place here, particularly in the identification and specification of criteria. Other aspects of QC, such as the application of technical standards and statistical control and measurement, are provided in the laboratory by training and certification of technical personnel, documentation of procedures, meticulous record keeping, and instrument calibration.

Using the above means, quality is achieved and maintained by those having responsibility for performing the work. Quality achievement is verified through audits performed by impartial personnel not directly involved with the work. Auditors may be from within the organization where the work is performed (internal audit) or from an outside organization (external audit).

CHAIN OF CUSTODY OF MATERIALS

Identification and traceability of raw material components are specific requirements of NQA-1. For traceability, a chain of custody should be established

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in which the materials source and container or locational transfers are documented and the records are cross-referenced. At a given time, any lot of material should be readily identifiable as to source, chemical components, and history.

Each raw material used in grout formulation work at ORNL is received with a chemical analysis being supplied by the vendor or supplier. It then undergoes an in-house chemical analysis for verification. If appropriate, this is performed by in-house CERCLA-approved laboratories. Each material item, as it is received, is given an identification or lot number and then logged into a numbered notebook with its chemical analyses. Separate notebooks are kept for each type of material. If the material is transferred to another container(s), the transfer is attended and verified by an impartial QA representative. Upon use of the material, it is again logged in by identification number.

As an example, let us consider the history of a shipment of fly ash for use in a grout dry-solids blend. The vendor, person receiving the shipment, date of receipt, and vendor-supplied analysis are logged in a notebook dedicated to fly ash, as are the results of the in-house analysis. After the fly ash has been blended with other dry components, its identification number is again logged in — this time in a "Blend" notebook. It becomes part of a uniquely numbered blend which will be referenced in technical notebooks after waste is incorporated and testing proceeds. Since the Blend notebook contains identification and amount (by weight) of all blend constituents, with cross-referenced information available in the records, any given blend can be quantified exactly. Chemical and physical properties of grouts documented in technical notebooks can be keyed to specific lots of fly ash and all other raw materials via the identification numbers.

RECORD KEEPING

Good record keeping provides documentary evidence of quality. It is probably the single most important aspect of QA in the laboratory, and one which is emphasized particularly in waste disposal work since protection of the public welfare and legal defensibility to state or federal agencies are imperatives. One aspect of record keeping, materials control, is described in the previous section. The other primary source of laboratory records is in the technical notebooks. These are kept in a manner prescribed by the Martin Marietta Energy Systems, Inc. (Energy Systems), Legal Department.(3)

The notebooks are registered and given a number by Laboratory Records (a division of Energy Systems' Information Resources Organization), making them permissible legal documents. All notebook entries are made in ink, with any errors crossed out using a single line and initialed. The notebooks are kept on a day-to-day basis as work progresses and are signed at the end of each work session. They are reviewed at random intervals by supervisory personnel and signed after being checked for procedural accuracy, legibility, upkeep, and accuracy of calculations. The signatures of the technician and the supervisor provide record authentication and validation. When completed, all laboratory notebooks are stored in fire-resistant or fireproof safes. In addition to a written record, all numerical data are entered and stored on computer files.

OTHER DOCUMENTATION

Although it is important to document all experimental results in a legally defensible manner, they are useful to others only when published in referenceable, peer-reviewed documents:

1) ORNL/TM reports, which contain preliminary data subject to revision;
2) ORNL reports, which include final data (this is a formal report not subject to revision);
3) symposia proceedings; and
4) open-literature, refereed journals.

At the end of each stage of work at ORNL, either an ORNL/TM or a final ORNL report is written for publication. These reports are published internally and may be referenced by interested persons across the country through technical information data bases. Before any report or paper is published, it proceeds through several steps designed to ensure the veracity of the finished work. A first draft is given at least two formal peer reviews before it is considered ready for formal editing. Subsequent reviews are performed by Energy Systems' legal and patent departments. The report or paper is published or submitted to a refereed journal only after all internal reviews and editing have been completed and any recommended changes have been made.

DATA GENERATION, COLLECTION, AND ANALYSIS

A rigorous approach to experimental design and to the analysis of experimental results is an integral part of QA in the Grout Technology Development Program at ORNL. The experimental approach is modeled after guidelines provided by the American Society for Quality Control for experiments involving mixtures where the independent variables represent proportionate amounts of the mixture rather than (as in factorial experiments) unrestrained amounts.(4) A matrix of experimental runs is set up so that a number of responses and variables may be studied. A large data base is required for accuracy of results; the experimental design includes generation of numerical data in triplicate for each combination of variables in the matrix. The data are recorded in technical notebooks and on computer files (see section on "record keeping") for subsequent analysis.

In addition to the standard methods of data analysis such as the t-test and hypotheses testing,(5,6) use is made of response surface methodology(4,7) and extreme vertices designs(8) in order to optimize the process with respect to the important variables.

Although the general direction of the experimental design is established by program personnel, who have extensive experience in the field and in the laboratory, this determines the experimental path only. Quality assurance in data generation, collection, and analysis for the Grout Technology Development Program is based not on experience, but on proven peer-reviewed and tested techniques.

ANALYTICAL PROCEDURES

At ORNL, all analytical processes are standard, peer-reviewed procedures which, in grout technology development, follow those prescribed by the American Society for Testing and Materials (ASTM), the American

Concrete Institute (ACI), and the American Petroleum Institute (API). In addition, grout technology development utilizes ANS 16.1 for radionuclide leaching tests and the Extraction Procedure Toxicity Characteristic, soon to be replaced by the Toxicity Characteristic Leaching Procedure (TCLP), for priority pollutant metals and organics leaching, reactivity, and corrosivity.(9,10) These analytical procedures are thoroughly documented in a manual for grout formulation and testing, which was prepared for use by technical personnel in standardizing their work. As would be expected for development work, the manual is subject to continuing review and to revisions when appropriate and as regulatory and performance criteria evolve.

Laboratory technicians in the Grout Development Program undergo a required five-step certification process that is outlined in a training manual:

1) read safety manuals, instrument operation and calibration instructions, pertinent ASTM standards, and the grout formulation and testing manual;
2) tour the laboratory facilities and receive oral instructions on equipment and laboratory operations, and emergency procedures;
3) participate in on-the-job training with an experienced and certified technician under the supervision of a graduate chemist or engineer for about six months, or until the supervisor is satisfied with the trainee's skill development;
4) demonstrate procedural techniques in all phases of grout formulation and testing under observation of the grout development supervisor and/or other designated individuals; and
5) pass an oral examination.

Personnel who are certified grout formulators in the Grout Technology Development Program are also required to attend an annual oral review for recertification.

INSTRUMENT CALIBRATION

Measuring and test equipment must be calibrated, adjusted, and maintained on a regular schedule to ensure the generation of valid, reproducible data. Depending on the equipment's stability characteristics, required accuracy, and intended use, calibration in the grout formulation laboratories is performed either by operating personnel or by technicians who have been certified by the National Bureau of Standards (NBS). For example, a mud balance for measuring density is properly calibrated against water at room temperature, and the Fann viscometer used for rheological measurements is calibrated against NBS-certified "dead" weights. Both of these calibrations are performed by within-group operating personnel. On the other hand, calibration of analytical balances and the compressive strength tester requires an NBS-certified technician. A label is placed on the instrument listing the calibration number, date, and initials of the technician who performed the calibration. All calibrations are documented in a log book.

INTERNAL AND EXTERNAL AUDITS

Quality achievement is continuously monitored by various levels of management reviews and revisions of procedures, as well as by formal internal and external audits. In addition to verifying and documenting quality achievement, an audit will assist project managers in identifying any nonconformance with QA standards. The Chemical Technology Division uses an NQA-1 checklist as well as an ORNL QA audit checklist for this purpose. The Quality Assurance Assessment form is considered to be a living document, which will be revised as experience dictates. Audits are performed by impartial personnel not directly involved with the activities under scrutiny. The result is a written report to be reviewed by responsible management. Management is expected to take follow-up action, where indicated by the report, and to verify such action in writing.

Formal internal audits take place regularly and are performed under the auspices of ORNL's Quality Assurance and Inspection Department. External reviews are done by the programmatic sponsors. These formal audits are made to verify compliance with NQA-1 requirements and all other aspects of the respective QA programs. If an area of noncompliance is identified, recommendations are put into writing and referred to the appropriate program manager. Immediate corrective action is taken whenever possible. Actions taken or reasons for temporary inaction are documented with letters to the appropriate auditing personnel.

CONCLUSIONS

Quality assurance/quality control is an important aspect of all work performed in the Grout Technology Program, as shown by control and documentation of methodology, ongoing surveillance and peer review, and regular auditing. This thorough review and documentation of the program ensures the integrity and availability of all data, thereby providing a sound basis for demonstration of product reliability and compliance with technical and regulatory criteria.

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