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

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**QUALITY ASSURANCE CONSIDERATION FOR CEMENT-BASED GROUT TECHNOLOGY  
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**ABSTRACT**

Oak Ridge National Laboratory has developed and is continuing to refine a method of immobilizing low-level radioactive liquid wastes by mixing them with cementitious dry-solid blends. A quality assurance program is vital to the project because Nuclear Regulatory Commission (NRC), Environmental Protection Agency (EPA) and state environmental regulations must be demonstrably met (the work must be defensible in a court of law). The end result of quality assurance (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 Oak Ridge National Laboratory.

**INTRODUCTION**

Quality assurance (QA) must be a vital and integral part of any waste form development program, from inception, in order to ensure compliance with Nuclear Regulatory Commission (NRC), Environmental Protection Agency, (EPA) and state regulatory criteria and to merit public confidence. QA is defined as all those planned and systematic actions 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. QA activities include quality control (QC), peer review (for experimental work), and auditing.

At Oak Ridge National Laboratory (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 experimental data for permitting purposes. ORNL claimed exclusion of regulatory agencies under the Atomic

Energy Act of 1954 but did have a quality program that complied with DOE Order 5700.6A.<sup>1</sup>

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 made it imperative that ORNL's quality programs satisfy not only the Department of Energy 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 which are consistent with ANSI/ASME NQA-1:<sup>2</sup> (1) control of materials, (2) record keeping, (3) peer review, documentation, and reporting, (4) training and certification of personnel, (5) instrument calibration, and (6) 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. Judgement must provide the review criteria; QA in research and development will often depend upon the collective agreement of scientists/technical personnel.

QA/QC inputs to analytical operations and procedures are more easily defined although continuing surveillance and peer review do have a proper place here, particularly in 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 is a specific requirement of NQA-1. For traceability, a chain of custody should be established documenting materials source and container or locational transfers, with the records being cross-referenced. At any time, any lot of material should be readily identifiable as to source, chemical components, and history.

Raw materials used in grout formulation work at ORNL are received with a chemical analysis supplied by the vendor or supplier. They then undergo an in-house chemical analysis, for verification. If appropriate, this is performed by in-house CERCLA approved labs. Each material item, as it is received, is given an identification or lot number and is 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 grout dry-blend. The vendor, person receiving, date of receipt, and vendor-supplied analysis are logged in a notebook for fly ash only, as is the in-house analysis when it has been completed. When the fly ash is 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. Grout chemical and physical properties 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 arguably 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.<sup>3</sup>

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 initialled. 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. These signatures of technician and supervisor provide record authentication and validation.

When completed, all laboratory notebooks are stored in fire-resistant or -proof safes. In addition to a written record of data, all data is entered and stored on computer files.

## OTHER DOCUMENTATION

Although it is certainly important to document all experimental data in a legally defensible manner, in order to be of use to others it must be published in referenceable, peer reviewed documents:

- (1) An ORNL/TM report which contains 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 referred journals.

At the end of each stage of work at ORNL, a report is written for publication, either as an ORNL/TM or a final ORNL report. These reports are published internally and are referenceable by interested persons across the country through technical information data bases. Before any report or paper is published, it goes 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 rewritten and sent for editing. Subsequent reviews are performed by Energy Systems' legal and patent departments. Only after all reviews and editing have been completed and recommended changes have been made is the report or paper published.

#### ANALYTICAL PROCEDURES

At ORNL, all analytical processes are standard, peer-reviewed procedures which, in grout technology development, follow procedures prescribed by the American Society of Testing Materials (ASTM), American Concrete Institute (ACI), and American Petroleum Institute (API). In addition, grout technology development utilizes ANS 16.1 for radionuclide leaching tests and the EP Toxicity extraction procedure for priority pollutant metals.<sup>4</sup> These analytical procedures are thoroughly documented in a procedures manual for grout formulation and testing, 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 five-step certification process, outlined in a training manual and requiring that he/she:

- (1) Read safety manuals, instrument operating and calibration instructions, pertinent ASTM standards, and the grout formulation and testing manual,
- (2) Tour the laboratory facilities and receive oral instruction on laboratory operations and emergency procedures,
- (3) Participate in on-the-job training with an experienced and certified technician, under supervision of a graduate chemist or

engineer, for about six months or until the supervisor is satisfied with the trainee's progress.

- (4) Demonstrate procedural technique in all phases of grout formulation and testing, under observation of the grout development supervisor and/or other designated individuals, and
- (5) Take an oral examination.

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

#### INSTRUMENT CALIBRATION

For generation of valid, reproducible data, measuring and test equipment must be calibrated, adjusted, and maintained on a regular schedule. Depending on equipment stability characteristics, required accuracy, and intended use, calibration in the grout formulation labs is performed either by operating personnel or by National Bureau of Standards (NBS) certified technicians. 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 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 calibration number, date, and initials of calibrating technician. Within-group calibrations are documented in a calibration log.

#### 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. This is accomplished by formal internal and external audits as well as by various levels of management reviews and revisions of procedures and QA requirements. In ORNL's Chemical Technology Division an NOA-1 checklist is used as well as an ORNL QA audit checklist. The Quality Assurance Assessment form is considered to be a living document,

to be revised as experience dictates. Audits are performed by impartial personnel not directly involved with the activities being audited. 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 ONRL's Quality Assurance and Inspection Department. External reviews are done by the programmatic sponsors. These formal audits are to verify compliance with NQA-1 requirements and all other aspects of the respective QA programs. If an area of non-compliance 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

QA/QC 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, providing a sound basis for demonstration of product reliability and compliance with technical and regulatory criteria.



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