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THE PURPOSE OF THE MATERIALS
CHARACTERIZATION CENTER

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THE PURPOSE OF THE MATERIALS CHARACTERIZATION CENTER

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Abstract

The Materials Characterization Center (MCC) at the Pacific Northwest Laboratory is the experimental arm of the Materials Characterization Organization (MCO), which was established by the U.S. Department of Energy (DOE) in FY 1980 to ensure high quality characterization and qualification of waste package materials essential to the reliable performance of DOE nuclear waste management programs. The MCC is responsible for publishing key test methods and data in the Nuclear Waste Materials Handbook (DOE/TIC-11400) after the methods and data have been reviewed and approved by the Materials Review Board (MRB), which is the other major operating arm of the MCO.

The MCC is carrying out its responsibilities in several ways. It sponsors a continuing series of workshops that address materials characterization test method issues. It supplies well-characterized reference and testing materials for use by the DOE nuclear waste management programs. It develops generic test methods and supports the repository waste package projects in developing selected site-specific test methods and performing confirmatory testing of these methods. When these test methods are approved by the MRB they constitute the formal tests to be used by laboratories to test and qualify materials, evaluate waste package components, and assure compliance with standards and/or specifications for the final product.

Introduction

The Materials Characterization Organization (MCO) was established to ensure that coordination of all waste package materials characterization for U.S. Department of Energy (DOE) nuclear waste management programs (i.e., the waste producers and repository projects) is provided and that efficient utilization of resources and high quality performance is available in support of those programs. It is administered by the Materials Integration Office (MIO) at DOE-Chicago and consists of the Materials Characterization Center (MCC) located at DOE's Pacific Northwest Laboratory and the Materials Review Board (MRB), a DOE-funded peer review group under the chairmanship of Dr. M. J. Steindler of Argonne National Laboratory. A series of papers, of which this is the first, is being presented at this symposium to describe the status of MCC and MRB activities.

The MCC is working in cooperation with other DOE-contractors, to ensure high quality characterization and qualification of waste package materials essential to the reliable performance of DOE nuclear waste management programs by providing the following services:

- reference and testing materials
- standardized test methods
- documentation of data reliability
- publishing the Nuclear Waste Materials Handbook.

The MCC has a staff of about 12 scientists and 10 support staff organized as shown in Figure 1.

Figure 1. Organization of the Materials Characterization Center

The MCC/MRB provides specialized services to DOE nuclear waste management programs of the type that are provided more broadly to industry by the American Society for Testing and Materials (ASTM) and the National Bureau of Standards. In order for the services provided by the MCC/MRB to be most useful it is important that they meet the evolving needs of the waste form producers and the repository development programs. To assure the relevancy of its activities, much of the MCC's funding comes from the individual repository projects and waste form producers. A particular valuable service will be performed at the interface between waste form producer and waste form receiver. Standard tests are needed to demonstrate conformance to acceptance specifications. Reference materials are needed to serve as a frame of reference for the acceptance specifications.

The MCC's major activities are described briefly in the following four sections. More details will be found in the series of papers that follow in this symposium.

Reference and Testing Materials

We have made a distinction between reference and testing materials [1]. The use of reference materials to calibrate operator performance is mandated in some MCC test methods, particularly the leach test methods; the provision of testing materials is a convenient service provided by the MCC so that the repository projects can develop an authoritative data base on independently characterized waste package materials.

The MCC's principal reference material, Approved Reference Material No. 1 (ARM-1), is a generic simulated borosilicate waste glass, manufactured for the MCC by Schott Optical Glass, Duryea, Pennsylvania, and certified to meet homogeneity specifications by the National Bureau of Standards. The MCC is presently developing certified leach data for ARM-1 using the MCC-1, -2 and -3 leach tests. The ARM-1 glass is available from the MCC on request, free of charge, accompanied by the current MCC leach data.

Most of the testing materials, designated Approved Testing Materials (ATMs), supplied to the repository projects by the MCC to date have been radioactive waste forms, either doped glasses [2] or spent fuel [3]. These materials are being carefully characterized. Important parts of this characterization are done in fully-shielded, research-quality SEM, XRD and ICP instruments operated by the MCC.

Standardized Test Methods

A major impetus for creation of the MCC was the lack of comparability in testing methods being used to obtain nuclear waste data in different laboratories. The MCC is working with the waste form producers and repository projects to standardize test methods and determine which test methods should be peer reviewed by the MRB.

A complete test method contains many elements in addition to detailed "how to" instructions for the test operator. The detailed instructions help assure that the test results are reproducible when performed in different laboratories by different operators. But other elements are needed to assure a clear understanding of the purpose of each test method, its limitations, the use of reference materials and the information that should be contained in the report of test results. To assure that each test method contains all of the required elements, the MCC has adopted a standard format, generally patterned after that used by the ASTM [4], with the following major sections.

Title, Scope and Summary. These elements in combination identify the test, its purpose, the materials to which it is applicable, and describe briefly its distinctive features.

Uses and Limitations. This very important section explicitly bounds the applications of the test method. The applications are circumscribed either by known limitations (e.g., dose at which radiation damage to Teflon leach containers becomes limiting), or in some cases by lack of knowledge (e.g., the interpretation of a platinum electrode determination of Eh). The limitation statements clearly foreclose some applications of the test; for some other applications, the limitation statements serve only to warn the operator of unexplored or unsolved aspects of the tests.

Equipment, Safety Precautions, Analytical and Calibration Requirements. The equipment to be used is defined with sufficient detail to assure that results can be duplicated in different laboratories. In some cases special equipment, preparation or cleaning is required. This is specified. When possible, examples of suitable equipment are given to assist new test operators in preparing to do the test. Applicable safety precautions are highlighted. Analytical requirements are defined in terms of results to be achieved, without specifying what analytical technique is to be used to obtain the results. The equipment or devices requiring calibration are specified, along with the frequency and method of calibration.

In addition to the normal equipment calibration, the MCC is providing Approved Reference Materials (ARMs), Approved Testing Materials (ATMs) and reference Simulated Leachates (SLs) for use in leach tests and interactions tests [1].

Reagent and Test Specimen Preparation. Detailed instructions are given for preparation and storage of reagents and test specimens. All characterization of the test specimens that must be done prior to conducting the test is defined.

Procedure. This section contains step-by-step directions for using the equipment, test specimens, etc., described in previous sections to perform the actual test. The instructions are as concise and unequivocal as possible. Clear statements on the use of replicates, blanks, and reference materials are included. The procedure also defines all follow-on analyses that are required after termination of the test.

Calculations. Applicable mathematical operations to be used in processing the experimental data are given.

Precision and Bias. This very important entry tells the potential user what kind of data variability can be expected when the test method is applied in reasonably competent laboratories. Statistical information of this kind has been notably lacking in most nuclear waste management data. A major goal of the MCC is to promote improved practices in the application of statistics to test data, as described in the next section, and in the paper at this symposium by Bowen [5].

Report. Detailed directions for reporting test results are given. This amounts to a check list that assures results are reported in sufficient detail so that someone, for example on the staff of a regulatory agency, can assess the quality of the data at a time which may be many years after the data were collected.

The applications of the MCC test methods are shown in Table 1 and described in more detail in the papers that follow in this symposium. The test methods are designed to obtain the key materials data needed to assure safe operation of the high-level nuclear waste management system. If the need for new or different test methods becomes apparent, they will be added to the list. The test methods in Table 1 are all being codified by the MCC. This year, we are also working with investigators in the Office of Nuclear Waste Isolation (ONWI) project to codify test methods used in their testing program that are specific to salt repositories and present them to the MRB for peer review.

Table 1. Purpose of MCC Test Methods

<u>Purpose</u>	<u>Test Method(s)^a</u>
<u>Waste Form</u>	
Chemical durability	MCC-1, 2, 3, 4, 5
Aging effects (thermal and radiation)	MCC-6, 7, 12, 13
Volatility	MCC-8, 9, 16
Physical Strength	MCC-10, 11, 15
<u>Canister/Container</u>	
Corrosion resistance	MCC-101, 102, 103, 104
<u>Repository Interactions</u>	
Canister/container corrosion	MCC-105 ^b
Waste form durability	MCC-14 ^b

^aMore information on individual test methods may be found in the papers that follow in this symposium.

^bThe repository interactions tests are divided into site-specific sub-categories, i.e., MCC-105.1 (basalt), etc.

Documentation of Data Reliability

Two of the paramount requirements for reliable data have been discussed above, the use of well-characterized testing materials and well-documented test methods. But it is also necessary that reproducibility of the data be well-documented, i.e., would another investigator have obtained the same results? The answer to that question must be based on statistics, and statistics requires data, thus the MCC is working to promote the replicate and interlaboratory testing that will produce the needed data. The ultimate goal is to have a defensible statement on precision and bias in each key test method.

Interlaboratory data is of special importance - it is also a relatively rare commodity within the waste management community today. Interlaboratory data can be obtained by conducting round robins. The MCC conducted a round robin with the MCC-1 Static Leach Test Method in which 26 laboratories participated [6], and the Council of European Communities recently completed a round robin with the MCC-2 High-Temperature Static Leach Test Method, in which 12 laboratories participated. Obviously, it is not practical to conduct round robins of this type for all test methods that are used to obtain nuclear waste management materials data. Therefore, the MCC is promoting a concept of benchmark testing in which two, or more, laboratories perform the test at pre-defined bench mark conditions as a minimum documentation of interlaboratory reproducibility. In many cases, one of the laboratories may be the MCC, but of course, it does not have to be.

The Nuclear Waste Materials Handbook

Currently, the Nuclear Waste Materials Handbook, which is published by the MCC, consists of four partially-filled loose-leaf volumes. Two of the volumes (gray covers, DOE/TIC-11400) contain test methods and data that have been approved by the MRB; two (blue covers, PNL-3990) contain test methods and data that have been submitted to the MRB, but not yet approved. The MRB approval process is described in the paper by Steindler and Seefeldt [7].

The Handbook has a controlled distribution so that additions and revisions can be made as new test methods and data become ready for publishing. The Handbook is made available by DOE free of charge to anyone having a need for the information it contains.

Requests should be sent to:

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