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MCC
Materials Characterization Center

Nuclear Waste Materials Characterization Center Semiannual Progress Report

April 1985 Through September 1985

J. E. Mendel

December 1985

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

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NUCLEAR WASTE MATERIALS CHARACTERIZATION CENTER SEMIANNUAL PROGRESS REPORT APRIL 1985 THROUGH SEPTEMBER 1985

Compiled by

J. E. Mendel, Manager
Materials Characterization Center

December 1985

Prepared for
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Richland, Washington

FOREWORD

This is the first in a new series of progress reports. Previously, Materials Characterization Center (MCC) progress was reported as one section of PNL-4250, a series of technical progress reports designed to report semiannually on nuclear waste management programs at the Pacific Northwest Laboratory (PNL) operated for the Department of Energy (DOE) by Battelle Memorial Institute. This report, PNL-5683, covers MCC progress from April through September of 1985. The next progress report on the MCC will be an annual report issued in late 1986.

ACRONYMS

AECL	Atomic Energy of Canada, Limited
ANL	Argonne National Laboratory
ANSI	American Standards Institute
ARM	Approved Reference Material
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATM	Approved Testing Material
BNL	Brookhaven National Laboratory
BWIP	Basalt Waste Isolation Project
BWR	Boiling Water Reactor
DWPF	Defense Waste Processing Facility
HEDL	Hanford Engineering Development Laboratory
HLW	High-Level Waste
HWVP	Hanford Waste Vitrification Project
LLNL	Lawrence Livermore National Laboratory
MCC	Materials Characterization Center
MIO	Materials Integration Office
MRB	Materials Review Board
MSC	Materials Steering Committee
NQA-1	Nuclear Quality Assurance-1
NRC	Nuclear Regulatory Commission
NWMH	Nuclear Waste Materials Handbook
ODW&BM	Office of Defense Waste and Byproducts Management
OGR	Office of Geologic Repositories
ONWI	Office of Nuclear Waste Isolation
PNL	Pacific Northwest Laboratory
PWR	Pressurized Water Reactor
QA	Quality Assurance
QE	Quality Engineer
SNL	Sandia National Laboratories
SRP	Salt Repository Project

TDM	Test Development Material
TTC	Transportation Technology Center
WAC	Waste Acceptance Committee
WADAP	Waste Acceptance Data Acquisition Plan
WCP	Waste-Form Compliance Plan
WINC	Westinghouse Idaho Nuclear Company
WVDP	West Valley Demonstration Project

SUMMARY

MCC Program Office Activities

Work continued on converting MCC Quality Assurance practices to comply with the national QA standard for nuclear facilities, ANSI/ASME NQA-1 (1983). In this reporting period activities included setting up record-keeping files, transferring records to the records center, conducting training sessions, identifying custodians and reviewers, and undergoing internal and sponsor surveillances and audits.

Two test methods were distributed to holders of the Nuclear Waste Materials Handbook as Revision Package No. 6. They are MCC-101 Unstressed, Static, Immersion, Corrosion Test Method and MCC-102 Unstressed, Flowing, Immersion, Corrosion Test Method.

The MCC continued to interact with ASTM for adoption of MCC test methods. MCC-11 Splitting Tensile Strength Test Method was adopted. One negative vote was cast on the revised Standard Test Method for Static Leaching of High-Level Waste Forms (the ASTM version of MCC-1 Static Leach Test Method). SRP-WPP-18/MCC-102.1, Determination of General Corrosion Rate of Candidate Structural Materials in Brines and Brine/Salt Mixtures, was submitted for the first time.

Support to the Office of Geologic Repositories

Additional emphasis is being placed on acquiring and characterizing spent fuel Approved Testing Materials (ATMs). Arrangements were made for four additional spent fuel ATMs: ATM-103, ATM-104, ATM-105, and ATM-106. Characterized material from these ATMs is expected to be available during the second half of FY 1986. Special equipment for spent fuel handling, storage, and characterization was designed and is being procured or fabricated and installed. The final pieces of equipment will be installed in the first part of FY 1986. Characteriation of spent fuel ATM-101 was completed, and the first revision of the characterization report (Barner 1985) was issued.

Reports were prepared describing three ATM glasses: ATM-1, ATM-8, and ATM-12. These are the first reports issued in a series on ATM glasses in the MCC's inventory.

Efforts continue in the transfer of two bars of ATM-5 glass to Whiteshell Laboratory of Atomic Energy of Canada, Limited. The transfer is currently awaiting DOE written approvals.

Discussions were held with potential suppliers of a shielded Auger analyzer for characterizing radioactive materials. Formal bids will be evaluated in October.

Support to the Salt Repository Project

Work continued on the task to submit key test methods for the repository sites to the MRB. Of twelve test methods identified for the Salt Repository Project (SRP), ten were prepared as submittal packages by the end of September. The remaining two methods will be submitted to SRP in FY 1986. Several of the methods will be submitted to the Materials Review Board (MRB) in FY 1986.

The MCC developed a technique for sampling brine solutions at test temperature and pressure, thus avoiding precipitation of salts caused by quenching to room temperature.

Support to the Basalt Waste Isolation Project

TDM-1, a crushed and sized, depleted UO_2 powder was characterized and delivered to BWIP for use as a Test Development Material.

Development continued on BWIP/MCC-14.4 test method, designed to test for compliance of a loaded waste form with radionuclide release specifications. Testing was completed using ATM-3 glass specimens.

The draft report on 30-day and 120-day benchmark tests with BWIP/MCC-105.1 Basalt Static Corrosion Test Method was approved by BWIP and submitted to the MRB. Other benchmark tests with the method and using radiation were initiated.

The MCC initiated benchmark testing using BWIP/MCC-105.4 Basalt Flowby Test and began construction of equipment for the BWIP/MCC-105.5 Air/Steam Test.

Support to the Office of Defense Waste and Byproducts Management

The Waste Acceptance Requirements Data Aquisition Plan, prepared last reporting period, was presented to waste-form producers, repository projects, and DOE headquarters. A DOE committee was formed to prepare a single set of specifications for the waste form. The MCC document will be revised based on these specifications.

The MCC-7 Method for Preparation of Isothermally Heat-Treated Specimens was revised and submitted to the MRB for provisional approval. Also, a new draft of the MCC-17 Method for Chemical and Radiochemical Analysis of Waste Forms was prepared.

A workshop was held on the need for long-term testing of the chemical durability of waste forms. The test matrix presented at the meeting will be revised and presented to waste-form producers for concurrence.

Leach tests on the MCC reference glass, ARM-1, were conducted with MCC-1, MCC-2, and MCC-3 test methods. MCC-3 tests were completed and a data report (MCC-D6) was submitted to the MRB. MCC-1 one-year tests were completed. MCC-2 tests will be completed in December.

Support to Hanford Programs

An examination of existing tests and test conditions led to the conclusion that a test needs to be developed to assess the effectiveness of grouts in containing radionuclides. An MCC grout-release test will be developed to meet this need.

Support to the Transportation Technology Center

The MCC-15 Waste/Canister Accident Analysis Test Method was revised and submitted to the Transportation Technology Center (TTC). A videotape on this impact test was also prepared. Six half-scale Defense Waste Processing Facility (DWPF) canisters were fabricated, filled and sent to TTC for impact testing.

Support to the West Valley Demonstration Project

A plan was developed for West Valley Demonstration Project (WVDP) to acquire data to meet preliminary waste acceptance specifications. A detailed test program was developed for use in qualifying the leach behavior of WVDP reference glass. An estimate of the number of feed samples needed to characterize each batch of WVDP feed was provided.

CONTENTS

NUCLEAR WASTE MATERIALS CHARACTERIZATION CENTER (MCC) SEMIANNUAL PROGRESS REPORT - APRIL 1985 THROUGH SEPTEMBER 1985

1.0 INTRODUCTION

The purpose of nuclear waste management is to provide systems of natural and man-made barriers that prevent radioactive wastes from harming man and his environment, now and in the future. Consequently, the United States Department of Energy (DOE) is supporting research and development programs at many different laboratories to develop suitable geologic repositories, compatible waste forms and other man-made barriers. Because of the wide range of these activities and the diversity of contributors, a program is needed to ensure that the waste forms produced plus the structural barriers and backfill materials used will be well characterized by approved methods.

The Nuclear Waste Materials Characterization Center (MCC) was created by DOE at the Pacific Northwest Laboratory (PNL) in FY 1980 to support waste-form producers and repository projects in developing a defensible materials property data base. Such a data base must be supported by well-documented test methods, statistics, and quality assurance, so that it can be used as a recognized, authoritative source of data for design, integration, and licensing of waste management systems. Development of these systems requires a long-term commitment; it will be approximately 15 years before the first geologic repository for commercial waste is in operation in the U.S. Thus, a lasting compilation of authoritative nuclear waste materials test methods and reference data must be consolidated in one place, conveniently accessible to scientists, engineers, and administrators. The U.S. DOE Nuclear Waste Materials Handbook (DOE/TIC-1140), published by the MCC, helps to meet this need.

The Materials Integration Office (MIO) at the DOE Chicago Operations Office has programmatic responsibility for the activities of the MCC and of the

Materials Review Board (MRB), an independent peer-review panel that was created at the same time as the MCC to review and approve test methods and data before publication in the Handbook.

The objective of the MCC is to assist DOE's waste-form producing and repository development projects by:

- providing reference and testing materials
- standardizing test methods and facilitating MRB review
- establishing confidence limits on test data through application of statistics and confirmatory testing
- publishing the Nuclear Waste Materials Handbook.

The MCC provides support to all high-level waste-form producers and repository development projects; thus it is a multifunded program. Currently, the MCC funding comes from these sponsors:

The Office of Geologic Repositories (OGR)

The Salt Repository Project (SRP)

The Basalt Waste Isolation Project (BWIP)

The Office of Defense Waste and Byproducts Management (ODW&BM)

Hanford Programs

The Transportation Technology Center (TTC)

The West Valley Demonstration Project (WVDP).

Following this introduction, selected MCC Program Office activities are discussed. The remainder of the progress report is organized according to the above list of sponsors.

2.0 MCC PROGRAM OFFICE ACTIVITIES

2.1 Quality Assurance

Major Contributor: S. L. Sutter, MCC QA Coordinator

A new Quality Assurance (QA) system, which adapts ANSI/ASME NQA-1 (1983) requirements for nuclear facilities to an R&D laboratory was installed for license-related programs at PNL in FY 1985. This was a tremendous undertaking, and work continues on converting the MCC QA practices to the NQA-1 standard. A brief review of major landmarks in installing the system includes:

- PNL-MA-60, "Quality Assurance Manual for License-Related Programs," and two volumes of "Procedures for License-Related Programs" were issued and are being implemented. A synopsis was distributed as a guide to the QA process.
- An MCC QA plan, NWTP-1, that is keyed to the QA documents was issued.
- A centralized PNL Records Center was established for storage of license-related records, and the MCC began the transfer of records to the center.
- Staff members were trained in QA procedures and the training was documented. Custodians and reviewers were assigned as required.
- The process of checking for compliance with the QA plan began. Surveillances were conducted by the MCC Quality Engineer and by BWIP. An internal audit was conducted and a report is being prepared.

The MCC QA coordinator interfaces with the MCC Quality Engineer to help assure that the MCC's implementation of the new QA system goes as smoothly as possible.

2.2 Revision Package to the Nuclear Waste Materials Handbook

Major Contributors: J. E. Mendel and M. D. Merz

Revision package number six was distributed to holders of the Handbook (which includes two volumes of DOE/TIC 11400 and two volumes of PNL-3990). The revision package contained revised versions of two test methods, MCC-101S Static, Immersion Corrosion Test Method, and MCC-102S Unstressed, Flowing, Immersion Corrosion Test Method for insertion in PNL-3990. Publication completes planned MCC activity on these methods.

2.3 ASTM Interactions

Major Contributor: M. D. Merz

The MCC continued to interact with ASTM to obtain ASTM adoption of test methods for waste forms and container materials. MCC-11 Splitting Tensile Strength Test Method was adopted and will be published in the 1986 Volume 12 of the ASTM Standards.

MCC-1 Static Leach Test Method was revised by the C26.07 Nuclear Waste Subcommittee and was balloted at the subcommittee/committee level. One negative vote was cast based on further qualifying the pH measurement in brines and identifying the source of the reference brine. Another recommendation to revise the Precision and Bias section necessitated changes before moving to society ballot. The test technique has not been substantively changed during the ASTM review process, but additional conditions for applying and interpretating data obtained by the method were incorporated.

SRP-WPP-18/MCC-102.1 Test Method for Determination of General Corrosion Rate of Candidate Structural Materials in Brine and Brine/Salt Mixtures was submitted to the C26.07 subcommittee for review at the next meeting in January 1986. This test method was prepared by the MCC for SRP and will also be reviewed by the MRB for publication in the Nuclear Waste Materials Handbook.

3.0 SUPPORT TO THE OFFICE OF GEOLOGIC REPOSITORIES

3.1 Acquisition and Characterization of Spent Fuel Approved Testing Materials

Major Contributors: J. O. Barner, C. A. Knox, R. E. Thornhill, D. N. Berger, and L. J. Parchen

As presented in the last semiannual report (McElroy and Powell 1985), the MCC has embarked on a long-range plan to acquire and supply spent fuel Approved Testing Material (ATMs). This plan involves the use of ATM-101 for near-term scoping experiments and the acquisition of larger quantities of commercial spent fuel, that can be used over a longer period, for repository licensing studies. The latter will improve reliability and traceability of the data generated during increasingly complex experiments. While implementing this plan, the acquisition of ATM-102 (12 experimental BWR rodlets one-fourth full-length) was cancelled because of a lack of interest in this limited quantity of BWR fuel.

Arrangements were made to acquire four additional spent fuel ATMs during the reporting period as follows:

- ATM-103 - A moderate-burnup (~30 MWd/kgM) low-fission-gas-releasing (in-reactor) PWR fuel from the Calvert Cliffs reactor. The 176 rods (a full assembly) that comprise this ATM were delivered to Hanford in September 1985.
- ATM-104 - A high-burnup (~45 MWd/kgM), low-fission-gas-releasing (in-reactor) PWR fuel from the Calvert Cliffs reactor. The 135 rods (a partial assembly) that comprise this ATM were delivered in November 1985.
- ATM-105 - A moderate burnup (~26 MWd/kgM) low-fission-gas-releasing (in-reactor) BWR fuel from the Cooper reactor. The 98 fuel rods (two fuel bundles) that comprise this ATM are scheduled for delivery to Hanford in mid-January 1986.

- ATM-106 - A high-burnup (~45 MWd/kgM), high (>10%)-fission-gas-releasing (in-reactor) PWR fuel from the Calvert Cliffs reactor. The 20 rods that comprise this group were delivered to Hanford with ATM-104 in November 1985.

Subcontracting for the acquisition of ATM-103, -104, and -106 spent fuels from Baltimore Gas and Electric Company was conducted with Combustion Engineering, Inc., the fuel vendor. Subcontracting for the acquisition of ATM-105 from Nebraska Public Power district is being conducted with General Electric Company, also the fuel vendor. Characterized material from these four spent fuel ATMs is expected to be available for distribution to repository experimenters during the second half of FY 1986.

3.1.1 Characterization Equipment for Spent Fuel

The MCC plan for characterization and distribution of spent fuel ATMs calls for 1) the commercial fuel bundles to be warehoused in a central storage area, 2) small groups of rods to be periodically removed from the bundles, characterized, and stored in an easily accessible area (a miniwarehouse), and 3) the ATMs to be distributed in the form requested by the repository experimenters on a nearly "on-call" basis. To accomplish this, several pieces of equipment are required to store, handle, characterize, and dismantle the fuel assemblies/bundles and the 13-foot-long individual fuel rods. Equipment was designed (or modified) and is now being procured or assembled for the following:

- Assembly/Bundle Storage - Existing fuel storage canisters are being modified to store and to maintain the identity of MCC spent fuel ATMs.
- Rod Retrieval - Existing equipment is being modified to permit removal of individual fuel rods from the fuel assemblies/bundles.
- Rod Storage - A container from which individual fuel rods can be easily removed was designed; two containers were fabricated. Each container will accommodate 24 fuel rods. A support stand was fabricated to hold the storage container; it is shielded to prevent the radiation field from the fuel rods from interfering with the gamma

scanner. This container is also used to transport the fuel rods from the assembly/bundle storage area to the miniwarehouse.

- Gamma Scanner - A computer-controlled gamma scanner consisting of a detector, multichannel analyzer, a fuel rod positioner, counting equipment, and a collimator in a hot-cell wall-plug was designed. Most of the components have been received. The gamma scanner will permit, in combination with limited chemical analyses, the determination of radionuclide inventories in samples and specimens distributed to repository experimenters.

- Fission Gas Sampler and Rewelder - A laser-operated fission gas sampler was designed and most of the components procured. After backfilling fission-gas-sampled rods with helium, the laser will be defocused to reseal the fuel rods. This will permit return of characterized fuel rods to a miniwarehouse in a condition that will prevent degradation.

- Fuel Rod Strongback - A strongback device was designed and fabricated to permit the handling of individual fuel rods.

- Sample Transfers - An obsolete shielded glovebox that was attached to the hot cell was removed and a spent-fuel sample-transfer port installed. This will ease transfer of individual characterization samples to the examination facilities because there will be lower levels of swipable radioactive debris within the shipping cask that could contaminate transfer apparatus at the receiving location.

During the first quarter of FY 1986, the final two pieces of equipment required for characterizing and distributing the spent fuel ATMs (a cut-off saw for sectioning fuel rods and an archive storage system) will be designed, fabricated and installed.

3.1.2 Spent Fuel ATM Characterization

The characterization of spent fuel ATM-101 was completed, except for the gamma scanning of a few segments of fuel rods that will be distributed to repository experimenters during FY 1986. The characterization plan was revised at mid-year to include additional chemical analyses for critical radionuclides that were recommended by repository experimenters. Chemical characterization now includes nine transuranic radionuclides (^{235}U, ^{236}U, ^{238}U, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{237}Np, and ^{241}Am), six fission products (^{137}Cs, ^{90}Sr, ^{99}Tc, ^{79}Se, ^{126}Sn, and ^{129}I), and the activation product ^{14}C. The first revision of the characterization report was issued in June (Barner 1985); however, the bulk of the additional characterization data was not available at the time of report preparation. This information will be included in Revision 2 (the final version) of the characterization report that will be issued in mid-FY 1986.

3.2 Approved Testing Materials (ATM) Waste Glass Fabrication Reports

Major Contributors: J. W. Wald and J. L. Daniel

Reports were prepared describing the fabrication and characterization of three of the MCC Approved Testing Materials glasses, ATM-1, ATM-8 and ATM-12 (Wald 1985a, b, and c). These are the first three in a series of reports on ATM glasses currently in the MCC's inventory. The reports provide details on the source materials and fabrication procedures used, and the best available information regarding chemical and physical characteristics. A similar report for ATM-9 is scheduled for completion in November 1985, and for ATM-11 upon completion of characterization analyses, tentatively in January 1986; the other ATM reports will follow.

3.3 Transfer of ATM-5 Glass to Whiteshell

Major Contributor: J. L. Daniel

Efforts began in January 1985 to arrange for transfer of two bars (about 350 g) of ATM-5 (fully loaded waste form glass based on 76-68 composition) from MCC to the Whiteshell Laboratory of Atomic Energy of Canada, Limited (AECL). AECL has an ongoing materials test program in their "Immobilized Fuel Test Facility," in which the behavior of several radioactive waste forms including ATM-5 glass will undergo behavior characterization relative to long-term storage. Test results and data will be reported to MCC by AECL upon completion of the tests.

Progress has been made in obtaining transfer approvals by U.S. and Canada. DOE terms and conditions for the transfer have been approved by AECL. Alternative shipping containers for shipment of the glass were identified, and the necessary procedures for documentation and shipping are continuing. The initial shipment target date was September 30, 1985, but delays in receiving written DOE approvals have forced a delay.

3.4 Shielded Auger Analyzer Procurement Planning

Major Contributors: J. L. Daniel and M. T. Thomas

The MCC is participating in the cooperative procurement of a research-grade Auger analyzer that will be shielded by PNL to allow application to highly radioactive materials in connection with nuclear waste materials characterization. Detailed discussions were conducted with two potential suppliers. The basis for the discussions was the successful design criteria applied previously in shielding the MCC's scanning electron microscope. Similar conditions of operation and maintenance pertain to the Auger, but the higher vacuum used in the Auger imposes constraints on the possible methods of shielding and sample introduction. Two manufacturers visited PNL to present their proposed plans for instrument modifications to permit operation, and one manufacturer decided to withdraw his initial proposal. Evaluation of formal bids will be conducted after all responses have been received.

3.5 Service Laboratory Procedures

Major Contributors: J. L. Daniel and J. W. Thielman

MCC continues to coordinate the preparation and controlled distribution of written procedures related to the PNL service analytical laboratories that are conducting work for the OGR programs in compliance with NQA-1 standard requirements. Some PNL service laboratory procedures were issued, and others are in process. Some of the procedures have similar subject matter but focus on different PNL service laboratories. Table 1 lists the MCC-coordinated service laboratory procedures.

TABLE 1. MCC Coordinated Service Laboratory Procedures

Title	Author	Reviewer(s)	Status as of 9-30-85[a]
PNL-SP-1 Metallography of Mild Steel	R. H. Beauchamp	H. C. Bowen	Issued
PNL-SP-2 X-ray Diffraction Analysis	H. E. Kissinger	E. D. Jenson R. D. Allen	Issued
PNL-SP-3 Scanning Electron Microscopy/Energy Dispersive Spectrometry	H. E. Kjarmo	B. Mastel	Issued
PNL-SP-4 Operation of Electron Microprobe	F. N. Hodges	E. D. Jensen	Issued
PNL-SP-5 X-ray Photoelectron Spectroscopy (XPS) and Auger Electron Spectroscopy (AES)	L. R. Pederson	E. D. Jenson	Issued
PNL-SP-6 ICP Analysis Using the Jarrel-Ash Atom Comp Model (3720 Building)	F. T. Hara	Roy Ko	Issued
PNL-SP-7 ICP Analysis (325 Building)	F. T. Hara	Roy Ko	Issued
PNL-SP-8 ICP Analysis (PSL Building)	A. W. Lautensleger	Roy Ko	Issued
PNL-SP-9 Energy Dispersive X-ray Fluorescence Analysis (EDXRF) Using the Tracor Spectrace 440 System	F. T. Hara	J. C. Evans Fred Scott	Original version approved by SRP - 3-6-85; PNL is revising before issuing; not yet resubmitted
PNL-SP-10 Inorganic Anion Determination Using the Dionex Model 12 Ion Chromatograph (IC)	F. T. Hara	A. W. Lautensleger	Issued
PNL-SP-11 Atomic Absorption Spectrophotometry Using the Perkin-Elmer Model 5000 Spectrophotometer and Graphite Furnace	F. T. Hara	J. C. Evans	Issued
PNL-SP-12 Potassium Hydroxide Fusion	F. T. Hara	G. M. Richardson	Issued
PNL-SP-13 AA-1/1 Atomic Absorption Spectrometry (Flame Source)	F. T. Hara	Bob Stromatt	Issued[b]
PNL-SP-14 IC Analyses Using the Dionex 12 S Autoion Chromatograph	A. W. Lautensleger	Bob Stromatt	In process
PNL-SP-15 Automated Acid/Base Titrations	A. W. Lautensleger	F. T. Hara	In process
PNL-SP-16 Metallography of Ceramic, Glassy & Cement Specimens	N. Saenz	B. Mastel	In process
PNL-SP-17 Metallography of Ferrous and Nonferrous Metals and Alloys	N. Saenz	B. Mastel	In process
PNL-SP-18 Instrumental Neutron Activation Analysis of Groundwater and Solid Samples by Gamma Spectrometry	J. C. Laul	R. W. Sanders	In process
PNL-SP-19 Procedures and Quality Control for Energy Dispersive X-ray Fluorescence Spectrometry	R. W. Sanders	J. W. Shade	In process
PNL-SP-20 Nonradioactive Solid Sample Preparation	J. S. Allen	B. Mastel	In process

(a) Those procedures listed as "Issued" have SRP approval. The BWIP is currently reviewing these procedures.
(b) Procedure PNL-SP-13 is issued with PNL approval and is being reviewed by SRP and BWIP.

4.0 SUPPORT TO THE SALT REPOSITORY PROJECT

4.1 Test Method Submissions

Major Contributor: R. D. Allen

The goal of this task is to select key methods for use in testing on the Salt Repository Project (SRP) and prepare them for submission to the MRB and publication in the Nuclear Waste Materials Handbook. In FY 1985, twelve test methods were selected, and MRB submission packages were prepared on ten of the twelve methods. The remaining two packages will be completed in FY 1986. The submission packages were sent to SRP for their acceptance before beginning the MRB review process.

To prepare a submission package, each SRP test procedure was rewritten according to the ASTM Standard Test Method format. A "Perspective" section was added to summarize the need for the method and show how it meets MRB criteria. A review of available data is required to prepare a precision and bias statement for each method. Where sufficient data were available a report on test experience was prepared to support the acceptance of the method. Table 2 shows the status of the twelve MRB submission packages. The purpose of the individual methods prepared in FY 1985 is outlined below.

Test methods SRP-WPP-18/MCC-102.1 and SRP-WPP-35/MCC-105.6 address the general and localized corrosion of candidate container materials in brines and brine/salt mixtures. The methods will be useful in testing candidate materials and developing models to predict the long-term corrosion of container materials in a salt repository. SRP-WPP-41/MCC-14.7 examines spent fuel leach testing. SRP-WPP-48/MCC-14.8 addresses interactive leaching of nuclear waste glass in brines. The behavior of these radioactive materials in brine relates to the transport and fixation of waste products. SRP-WPP-43/MCC-201 presents a test method for determining pH in brines; this capability is significant because pH is one of the most important parameters influencing corrosion rates.

Effects of irradiation and elevated temperature on rock salt and brine may influence the corrosiveness of the immediate environment. SRP-WPP-40/MCC-202 studies the radiolysis of salt brines exposed to gamma and alpha sources. Ionizing radiation affects the chemistry of aqueous solutions through the

TABLE 2. Preparation of MRB Submission Packages for SRP in FY 1985

Title	MCC Writer	Reviewer(s)[a]	Submitted to SRP
SRP-WPP-18/MCC-102.1 Determination of General Corrosion Rate of Candidate Structural Materials in Brines and Brine/Salt Mixtures	M. D. Merz	R. E. Westerman D. J. Bradley	July 1985
SRP-WPP-31/MCC-203 Determination of Different Oxidation States of Plutonium and Neptunium Using Solvent Extraction Techniques	R. M. Fruland	D. Rai	In process
SRP-WPP-35/MCC-105.6 General Corrosion Rate of Candidate Structural Materials in Brine/Salt Mixtures Under Excess Salt Conditions	R. D. Allen	M. D. Merz J. H. Haberman	September 1985
SRP-WPP-39/MCC-205 Preparation and Analysis of Simulated Permian Basin Brines and Dry Salts	P. E. Ruffin R. D. Allen	K. H. Pool	In process
SRP-WPP-40/MCC-202 Radiolysis of Salt Brines	P. E. Ruffin J. C. Evans	D. J. Bradley W. J. Gray	June 1985
SRP-WPP-41/MCC-14.7 Spent Fuel Leach Testing	P. E. Ruffin W. Ross	W. J. Gray J. H. Westsik	September 1985
SRP-WPP-43/MCC-201 Measurements of pH in Brines	J. C. Evans K. H. Pool	L. L. Burger L. R. Pedersen	September 1985
SRP-WPP-44/MCC-204 Total Base Determination in Solution of Irradiated Salts	J. C. Evans	L. L. Burger	July 1985
SRP-WPP-45/MCC-206 Hypochlorite ion Determination in Solutions of Irradiated Salts	K. D. Wiemers	L. R. Pedersen P. E. Ruffin	September 1985
SRP-WPP-48/MCC-14.8 Waste Glass Interaction Testing	R. M. Fruland G. L. McVay	B. P. McGrail D. J. Bradley	September 1985
SRP-WPP-53/MCC-104.1 Static-Load Crack Growth Testing and Modified Wedge Opening Load	M. D. Merz	S. G. Pitman W. E. Anderson	September 1985
SRP-WPP-57/MCC-103.1 U-Bend Testing of Metallic Specimens	M. D. Merz	S. G. Pitman	September 1985

(a) Each procedure was also reviewed by selected MCC staff members.

generation of free-radical, ionic, and molecular products including hydrogen gas. Transient basic conditions may be generated within rock salt in a repository because elevated temperature and irradiation cause partial decomposition of sodium chloride, and resultant reactions of sodium with water form sodium hydroxide and hydrogen. Water and chlorine compounds may react to form hypochlorite ions and other chlorine-containing species. Generation of these bases can temporarily affect the pH and composition of brines. Long-term effects, including decomposition of ions in solution, are also being investigated. SRP-WPP-44/MCC-204 describes total base determination for irradiated sodium chloride plus aqueous fluids whereas SRP-WPP-45/MCC-206 addresses the determination of hypochlorite ion concentration in solutions of irradiated salts.

Because the container will most likely experience stresses caused by lithostatic pressure, residual stresses from fabrication, and stresses due to thermal effects, it will be necessary to assess the potential for container degradation by stress-corrosion cracking. The SRP has identified two test methods to provide data on stress-corrosion cracking in simulated repository environments: 1) SRP-WPP-57/MCC-103.1, for testing materials and developing suitable welding and metallurgical treatments for container materials, and 2) SRP-WPP-53/MCC-104.1, for obtaining the static crack-growth threshold. Stress-corrosion cracking is not detectable below this threshold stress intensity.

4.2 Brine Sampling and Measurement Techniques

Major Contributor: R. L. Erikson

The MCC assisted the SRP in the development of a technique for obtaining multiple samples of concentrated brine solutions from Dickson autoclaves (Seyfried, Gordon, and Dickson 1979) at elevated in situ temperatures and pressures. In situ brine sampling techniques are required to avoid changes in brine compositions caused by quenching the solution to room temperature. A Dickson autoclave was modified to include an in-line, small-volume sampling vessel. Temperature gradients between the autoclave and sampling vessel were minimized by wrapping sampling vessel, tubing, and valves with heater tape or resistance windings and insulation. This design circumvents the problems reported in previous studies (e.g., Sourirajan and Kennedy 1962) where salts that have large solubilities at high temperatures precipitate as the brine cools in the sampling tubes.

The sampling technique was verified (i.e., proof tested) by determining the solubility of halite (NaCl) at 200°C and 25 MPa. The sampling technique was demonstrated by extracting three samples of saturated NaCl brine at 200°C and 25 MPa at run durations of 1, 3, and 7 days. The compositions of the brines were determined gravimetrically, and by direct determination of the Na and Cl concentrations of diluted samples using inductively coupled plasma emission spectrometry (ICP) and ion chromatography (IC), respectively. The data determined in this study for the solubility of NaCl in H_2O at 200°C and 25 MPa are compared in Figure 1 to other determinations of the solubility of NaCl on the solid-liquid-vapor curve.

The average compositions based on triplicate analyses of the brines sampled at 1, 3, and 7 days determined from gravimetric analyses were 8.255, 8.195, and 8.056 molal, respectively. These compositions are approximately 1 to 4% greater than the value (7.973 molal) reported by Liu and Lindsay (1971) for the solubility of NaCl in water at 200°C and the vapor pressure of the solution. Further experiments are necessary to determine if the slight over-saturation observed in our measurements relative to the data of Liu and Lindsay (1971) could be reduced by 1) improved temperature control of the Dickson autoclave/external sampling system, and 2) high-temperature ultrafiltration of the saturated brine samples for removal of suspended solids. However, the intended objective of developing a satisfactory sampling technique has been realized by the results obtained to date.

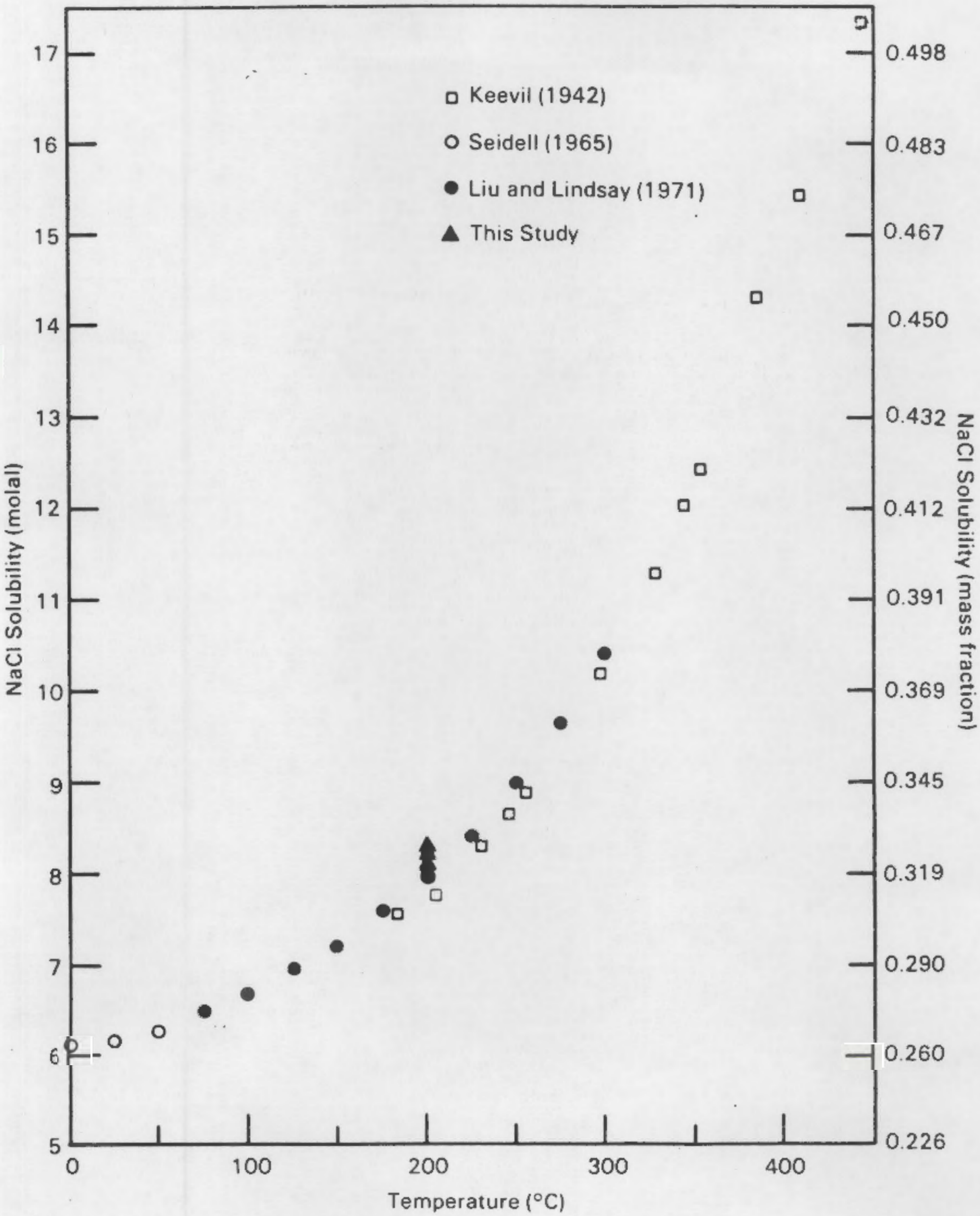

FIGURE 1. Molal Solubility of NaCl in Water Along the Three-Phase Solid-Liquid-Vapor Curve in the System $NaCl-H_2O$

5.0 SUPPORT TO THE BASALT WASTE ISOLATION PROJECT

5.1 Approved Reference and Testing Materials - UO_2 Powder

Major Contributors: G. D. White, J. W. Wald, J. L. Daniel

In response to a BWIP request, the MCC prepared and delivered a small quantity of crushed and sieve-sized depleted UO_2 powder for test development purposes. For starting material, 1 kg of well-documented uranium dioxide pellets was obtained from material available from the Hanford Engineering and Development Laboratory (HEDL), and a preparation run plan was developed and submitted to BWIP for concurrence. The crushed and sized material delivered to the sponsor was officially designated TDM-1 (Test Development Material). Characterization showed that it meets all specifications except for the expected particle size range. Scanning electron microscopy showed that a significant quantity of particles smaller than the specified 125 μm minimum was retained, apparently as a result of the agglomeration of small particles during sieving. Use of the material as produced was approved by the BWIP.

5.2 BWIP/MCC-14.4 Waste-Form Compliance Test Method Development

Major Contributors: R. L. Erikson, K. M. Krupka, and R. W. Fulton

The MCC continued development and benchmark testing of BWIP/MCC-14.4, the BWIP Compliance Test on Radionuclide Release for Determining Acceptance of a Loaded Waste Form. The test's purpose is to measure steady-state radionuclide concentrations in solutions after hydrothermal interactions of a synthetic basalt groundwater, basalt rock, and a radioactive waste form material. The results obtained during this reporting period include completion of the BWIP/MCC-14.4 test using ATM-3 (Np-doped MCC 76-68 glass waste form) with basalt and synthetic basalt groundwater.

The test conditions for the BWIP/MCC-14.4 tests using ATM-3 are listed in Table 3. The ATM-3 experiment was performed under run conditions identical to those used for the BWIP/MCC-14.4 test involving ATM-4 glass (Pu-doped MCC 76-68

TABLE 3. Conditions of BWIP/MCC-14.4 Test Used for ATM-3 Experiments

Temperature	150°C
Pressure	Vapor Pressure
Glass Particle Size	60 to 120 Mesh
Basalt Particle Size	120 to 230 Mesh
Mass Ratio of Solids	1:1
Solution/Solid Ratio	10:1 (mL/g)
Run Duration	3, 6, 9, 12, 27[a] Weeks
Test Replication	2 Vessels

(a) Sampling of single vessel at 27 weeks was used to verify the results obtained for the first four sampling periods. Sampling at 27 weeks is not required by the BWIP/MCC-14.4 test method.

glass waste form).[a] The experimental equipment, starting materials, and testing procedures are described in Strickert, Erikson, and Shade (1985). In the BWIP/MCC-14.4 test method, equal masses (1:1 by mass) of crushed waste form (-60 +120 mesh) and basalt (-120 +230 mesh) are added to synthetic GR-4 basalt groundwater at a solution-to-solids ratio of 10 mL/g. Leachant chemical compositions are determined for solution aliquots sampled from duplicate reaction vessels at each specified period of run duration. Aliquots of each solution are then filtered using both 0.45 µm and 0.0018-µm pore-sized membranes. The solution analyses include 1) pH and Eh for unfiltered fractions; 2) radionuclide concentrations for unfiltered, 0.45-µm, and 0.0018-µm fractions; 3) concentrations of nonradioactive cationic constituents for both 0.45-µm and 0.0018-µm fractions; and 4) concentrations of nonradioactive anionic constituents for only the 0.45-µm fractions. Radio-counting techniques, inductively coupled

(a) Results for BWIP/MCC-14.4 test using ATM-4 glass were described previously (McElroy and Powell 1985).

plasma spectroscopy (ICP), and ion chromatography (IC) are used to determine the total solution concentrations of individual radionuclide, cationic, and anionic constituents, respectively.

Sampling and chemical analysis of solutions from reaction vessels at 9-, 12-, and 27-weeks duration were completed during the report period for the BWIP/MCC-14.4 test using ATM-3 glass. The concentrations of dissolved boron and molybdenum had not reached steady state conditions, indicating that the ATM-3 glass is still dissolving after 27 weeks.

5.3 Corrosion Research Support to BWIP

Major Contributors: M. D. Merz, R. W. Wang, and W. E. Anderson

5.3.1 BWIP/MCC-105.1 Basalt Static Test

Benchmark tests at 100°C for 30-d, 120-d and 300-d test periods according to this test method were completed during the previous reporting period. During this reporting period, BWIP/MCC-105.1 Benchmark Test Report for 30-d and 120-d tests was completed and was submitted to the MRB after BWIP approval. BWIP/MCC-105.1 benchmark tests, conducted for 120-d at 200° under ^{60}Co radiation at 10^4 R/hr, were completed and specimen analysis began. Specimens and test materials were prepared for longer duration, 300-d tests under radiation, which began in October.

5.3.2 BWIP/MCC-105.4 Basalt Flowby Test

The MCC completed initial tests for 30 d and 120 d at 100°C and initiated 30-, 120- and 300-d benchmark tests. The 30-d benchmark tests were completed, the 120-d benchmark tests are being completed in early October, and a BWIP/MCC-105.4 Benchmark Test Report based on these two tests will be prepared to support the submission of BWIP/MCC-105.4 test method to the MRB. The test method was revised after completion of initial 30-d and 120-d tests and was submitted to the BWIP before submission to the MRB. Minor changes recommended by the BWIP in a Review Comment Record were made and the method will be submitted to the MRB in October after final BWIP approval.

5.3.3 BWIP/MCC-105.5 - Basalt Air/Steam Test

Investigators for the BWIP have requested MCC to conduct benchmark testing and codify a test method wherein the corrosion specimens are placed in a slowly refreshed water vapor environment to simulate conditions in the operational period of the basalt repository. HEDL investigators began BWIP/MCC-105.5-type testing, and the MCC followed the progress of their testing program in FY 1985. The MCC started constructing a test apparatus to begin benchmark testing in FY 1986 and will submit the test method to the MRB in FY 1986.

6.0 SUPPORT TO THE OFFICE OF DEFENSE WASTE AND BYPRODUCTS MANAGEMENT

6.1 Waste Acceptance Requirements Data Acquisition Plan

Major Contributors: G. B. Mellinger and D. H. Mitchell

The purpose of this plan is to identify the information that waste-form producers must supply to a repository to show compliance with acceptance requirements, and to show how this information might be obtained. Development of this plan was initiated in the first half of FY 1985. The draft waste acceptance requirements proposed by each of the three repository sites were combined into a single set of specifications. A means of obtaining compliance information with each requirement was suggested, and applicable test methods were identified.

During the second half of FY 1985, the content of the plan was presented to the waste-form producers, the repository projects, and DOE-HQ. Presentations were made to the West Valley Demonstration Project, Savannah River Laboratory, Westinghouse Idaho Nuclear Company, the Hanford Waste Vitrification Project, the Basalt Waste Isolation Project, the Nevada Nuclear Waste Site Investigations, the Salt Repository Project, and Mark Frei of DOE. The plan was revised based on comments received during these presentations. A comment draft was prepared and was forwarded to the persons who attended the presentations.

Subsequent to the preparation of this plan, the DOE's Office of Geologic Repositories (OGR) Waste Acceptance Process (WAP) was formally instituted. As part of this process, the Waste Acceptance Committee was formed, with members representing each waste producer and repository project contractors, as well as the MCC and OGR. A major responsibility of this committee is to propose to OGR a single set of Waste Acceptance Preliminary Specifications (WAPS) for each waste producer. The specifications contained in these documents will be somewhat different from, and will supersede those in the previously issued draft repository requirements documents. As a result, work on the MCC Waste Acceptance Requirements Data Acquisition Plan was suspended until the OGR preliminary specifications are near completion. The MCC document, to be reissued in

FY 1986, will take the form of a generic Waste-Form Compliance Plan (WCP). The plan, as defined by the acceptance process, shows how a producer will comply with the specifications applicable to his facility. The MCC document will suggest where testing appears necessary to gather data and which test methods should be used when such testing is required.

6.2 MCC-7 Method for Preparing Isothermally Heat-Treated Waste Forms

Major Contributor: W. J. Weber

This test method was revised and resubmitted to the MRB for provisional approval. The revised version more clearly separates heat treatment, which is the sole function of MCC-7, from the preparation of samples to be treated, the characterization of heat-treated samples, and the treatment of this characterization data. In the revised version, it is the responsibility of the requester of the treatment to specify treatment times and temperatures that will yield appropriate samples. The treatments that would be specified for determining time-temperature-transformation (TTT) curves would not be necessary if one were, for example, investigating the effect of a specific heat treatment on radionuclide release. As a result, references to TTT curves and test matrices designed to provide samples for such determinations have been eliminated. The method deals strictly with the conditions and considerations necessary for making reproducibly heat-treated materials.

6.3 Long-Term Chemical Durability Testing Needs

Major Contributor: K. H. Abel

Plans were made to begin long-term testing of waste forms using the Catholic University pulsed-flow test. This test simulates the condition of slow-flow by periodically removing part of the leachate and replacing it with fresh leachant. This long-term testing would continue for periods exceeding one to two years. Such testing would not be intended to predict the repository behavior of waste forms. However, it may be useful to waste form producers to determine the leach rate of various waste forms under conditions that simulate long-term exposure to a slowly flowing leachant. A workshop was held in Seattle, August 19-20, 1985 at which long-term testing and a proposed test matrix were discussed. Individuals who attended represented Savannah River Laboratory, the West Valley Demonstration Project, the Hanford Waste Vitrification Project, Westinghouse Idaho Nuclear Company and Pacific Northwest Laboratory. Also present were T. H. Pigford from the University of California, and P. B. Macedo and A. Barkatt from the Catholic University of America. The consensus of the meeting was that long-term testing is a worthwhile undertaking. The test matrix outlined at the meeting will be revised and presented to waste-form producers for their concurrence.

6.4 Solubility-Controlled Concentration Data for Radioactive Elements in Nuclear Waste

Major Contributors: K. M. Krupka and R. W. Fulton

Accurate thermochemical data for aqueous complexation constants and solubility products are needed to predict the geochemical behavior of nuclear waste elements (e.g., actinides) in surface-water and ground-water systems. The solubility of U(VI) hydroxide is considered to have an important role in constraining the maximum possible concentrations of uranium dissolved in oxidizing to moderately reducing waters (e.g., see Allard 1982; Wood and Rai 1981; and Thompson, Dove, and Krupka 1984). Solid U(VI) hydroxide has also been identified as an oxidation product in dissolution studies of crystalline UO_2 (Wang 1981) and as a possible precipitate in leaching studies of irradiated UO_2 fuel in air-saturated aqueous solutions (Vandergraaf 1980).

The MCC-3S Agitated Powder Leach Test Method (MCC 1984) was used with minor modifications to investigate the solubility of solid U(VI) hydroxide.[a] The solubility of solid U(VI) hydroxide was determined from both undersaturation (dissolution) and oversaturation (precipitation) conditions at 25°, 70°, and 90°C in carbonate-free, oxidizing aqueous solutions in the pH range of 3 to 11. Solution samples were analyzed for pH and total concentration of dissolved uranium. Solid starting materials and precipitates that formed during the solubility experiments were characterized by x-ray diffraction analysis and

(a) At temperatures less than approximately 60°C, the mineral schoepite ($UO_3 \cdot 2H_2O$) is the most stable form of solid U(VI) hydroxide.

scanning electron microscopy. The experimental procedure and solubility measurements at 25°C for schoepite ($UO_3 \cdot 2H_2O$) are described in Krupka et al. (1985). Solubility measurements at 70° and 90° were completed during the report period.

The aqueous speciation and saturation indices for solution compositions determined from the solubility experiments at 25°, 70°, and 90°C were calculated using the MINTEQ geochemical reaction code (Felmy, Girvin, and Jenne 1984). The solubility of U(VI) hydroxide measured in our study is in good agreement with the solubility calculated for U(VI) hydroxide from the thermodynamic data tabulated in Sylva and Davidson (1979) and Lemire and Tremaine (1980). Our measurements confirm the amphoteric behavior of dissolved U(VI) and demonstrate the importance of including anionic U(VI) hydroxyl complexes [e.g., $(UO_2)_3(OH)_7^-$] in speciation/solubility calculations for U(VI) in carbonate-free waters under basic conditions. Our data also suggest that the solubility of U(VI) hydroxide is constant or decreases slightly between 25°C and 90°C at a constant pH.

6.5 ARM-1 Reference Glass Leach Data

Major Contributors: J. L. Daniel and K. M. Olson

Leach tests were completed for MCC-3 tests on ARM-1 glass for test periods up to 6 months. All test data and instrument calibration information underwent statistical analysis, and test procedures, materials, specimens, and results were documented in detail.

The report MCC-D6 Agitated Powder Leach Testing of ARM-1 Reference Glass Using the MCC-3S Test Method was submitted to the Materials Review Board. The D6 report consists of about 250 pages of detailed information and data describing the test and results, and detailed data analysis and evaluation of the significance of the test data. This is the first data set based on the ARM-1 glass, and the first dealing with the MCC-3 test method. The data are intended to provide the basis for MCC-certified standard reference values in applications of the MCC-3 test method in the field. Data are for tests of particles in the size range of -40+80 mesh; data for two other smaller size powders will be submitted as a supplement to D6 in FY 1986.

The one-year leaching tests on ARM-1 using the MCC-1 test method (to 90°C) were completed. The MCC-2 tests (to 150°C) will be completed in December 1985. The combined report on MCC-1 and -2 test results was started.

6.6 MCC-17 Method for Chemical and Radiochemical Analyses of Waste Forms

Major Contributor: F. T. Hara

The first draft of this method was reviewed and is being revised. This revision will include expanded descriptions of the required characteristics of analytical equipment that the method employs, such as the Induction-Coupled Plasma Spectrometry (ICP). It will also include data from the analysis of ARM-1 glass.

7.0 SUPPORT TO HANFORD PROGRAMS

7.1 Hanford Grout Testing

Major Contributors: R. J. Serne, R. L. Treat, and R. O. Lokken

A grout-release test is needed to assess the effectiveness of grouts in containing radionuclides under environmental conditions at Hanford. A number of existing tests and test conditions were examined to determine which would be most likely to simulate actual disposal conditions and/or yield results expected under actual disposal conditions. Testing was conducted using the ANS 16.1 intermittent solution-exchange test, static tests using monolithic and crushed material, flow-through-column tests using soil and grout both separately and in combination, and batch K_d adsorption tests. Based on this work, it was determined that none of these tests, by itself, was adequate to characterize grout's behavior. The MCC grout-release test, to be developed in FY 1986, will incorporate features of a number of these tests. The results of the FY 1985 work were documented in a report entitled "Development of a Technical Approach for Assessing Environmental Release and Migration Characteristics of Hanford Grout."

8.0 SUPPORT TO TRANSPORTATION TECHNOLOGY CENTER

8.1 Impact Test Video Tape

Major Contributors: S. C. Slate and A. H. McMakin

A video tape entitled "Impact-Testing of Canisters That Will Contain High-Level Nuclear Waste" was produced. This eight-minute presentation outlines the reasons for doing impact testing, demonstrates the steps involved in such testing, and shows the results obtained. It will be used by the Transportation Technology Center to help introduce this aspect of their program to develop a cask for transporting HLW.

8.2 MCC-15 Waste/Canister Accident Analysis Test Method

Major Contributors: P. A. Scott and R. K. Farnsworth

This method was revised based on a peer review that occurred in early FY 1985. The review comments were primarily of an editorial nature; no substantial changes were required. This method will be used by Sandia for impact testing of half-scale DWPF canisters supplied by the MCC.

8.3 Half-Scale DWPF Canisters

Major Contributor: P. A. Scott

Six half-scale DWPF canisters were fabricated and filled with simulated borosilicate waste glass, and shipped to Sandia National Laboratory for use in their transportation cask development program. The canisters were fabricated to NQA-1 standards. Their design will allow accelerometers to be attached to the canisters during impact testing. The accelerometers will measure and record the impact forces experienced by the canisters. A Hanford-type simulated waste glass with a composition similar to that of DWPF glass was cast in the canisters from a PNL ceramic melter. Two of the canisters will be returned to the MCC in FY 1986 for post-test analysis. The effects of the impact testing on canister integrity and glass particle-size distribution will be assessed.

9.0 SUPPORT TO WEST VALLEY DEMONSTRATION PROJECT

9.1 Waste Acceptance Data Acquisition Plan

Major Contributors: G. B. Mellinger and D. H. Mitchell

A plan was developed for the West Valley Demonstration Project (WVDP) that describes the waste-form testing that may be required for the DOE Waste Acceptance Process. The testing is part of an overall compliance strategy that the plan proposes. The acceptance requirements that the plan addresses are the DOE Waste Acceptance Preliminary Specifications. Compliance with some specifications can probably be assured through the use of existing open literature data. Such specifications deal with glass properties not particularly sensitive to compositional and microstructural variation. The majority of the testing called for in the plan is in the area of radionuclide release. A significant effort will be required also for the development of "process models" that will allow West Valley to predict the composition and microstructure of their glass from process data such as feed composition analyses.

9.2 WVDP Reference Glass Radionuclide-Release Test Program

Major Contributor: K. H. Abel

A radionuclide-release test program was developed that the WVDP may initiate as part of the process of qualifying its reference glass. The tests included in this program include the methods identified by the NNWSI and BWIP repository projects (MCC-1 and BWIP/MCC-14.4, respectively) and a standardized version of the Catholic University partial replacement test to meet the SRP's acceptance requirement. The statistically designed test program specifies the materials to be tested, including the glass reference material ARM-1, and the number of specimens of each material to be tested. A central feature of the test program is the use of three laboratories for generating benchmark data. Significantly better statistical information is obtained through use of multiple laboratories, and the precision with which true waste-form test behavior may be estimated from test results is substantially improved.

9.3 Melter Feed Sampling Strategy

Major Contributors: W. M. Bowen and P. A. Pulsipher

WVDP was provided with an estimate of the number of melter feed samples that they will need to take during radioactive operations in order to adequately characterize their feed. They will need to take approximately seven samples from the feed makeup tank for each new batch of feed. These samples should be taken from various locations in the tank, and one analysis should be performed on each sample. This sampling strategy is based in part on the analysis of samples obtained during a 20-day pilot-scale ceramic melter test, conducted at PNL in FY 1985, that processed WVDP feed. The results of a PNL study of feed tank mixing behavior were also used in developing this proposed strategy. WVDP was also furnished with a suggested melter feed tank sampling plan that, when implemented at WVDP, will allow them to determine whether the assumptions made in developing the proposed sampling strategy were valid.

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