
MCC

Materials Characterization Center

**Approved Reference and
Testing Materials for Use in
Nuclear Waste Management
Research and Development
Programs**

G. B. Mellinger

J. L. Daniel

December 1983

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.



PNL-4955-1

Legacy - ns

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

MCC

Materials Characterization Center

**Approved Reference and
Testing Materials for Use in
Nuclear Waste Management
Research and Development
Programs**

G. B. Mellinger

J. L. Daniel

December 1983

**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**

CONTENTS

I. SUMMARY	1
II. INTRODUCTION	3
The Objective of the MCC	3
The Nuclear Waste Materials Characterization Organization	3
This Document	3
Classifications of Standard Materials	3
Approved Reference Materials	4
Approved Testing Materials	4
Archiving and Distribution of Standard Materials	4
III. SUMMARY OF STANDARD MATERIALS	5
Inventory of Available Standard Materials	5
Materials Policies	5
IV. STANDARD MATERIALS DESCRIPTIONS	7
ARM-1; Reference Waste Glass, Nonradioactive	8
ATM-1, 2, 3 and 4; 76-68 Type Glass, Actinide Dopants	10
ATM-5; 76-68 Type Glass, Fully Radioactive	12
ATM-6; Barnwell Type Glass, Radioactive Dopants	14
ATM-101; H. B. Robinson Spent Fuel, Typical PWR	16
MCC 76-68; 76-68 Type Glass, Used for Selected Applications	16
Planned Acquisitions; Reference Leachates, Basalt, Bentonite, Glass ATMs, Spent Fuel	18
V. REFERENCES	19

I. SUMMARY

This document, addressed to members of the waste management research and development community, summarizes reference and testing materials now available from the Materials Characterization Center (MCC). These materials are furnished under the MCC's charter to distribute reference materials essential for quantitative evaluation of nuclear waste package materials under development in the U.S.

Reference materials with known behavior in various standard waste management related tests are needed to assure that individual testing programs are correctly performing those tests. Approved testing materials are provided to assist the projects in assembling a materials data base of defensible accuracy and precision.

This is the first issue of this publication. Future issues will provide updates of the characterization status of the materials presented in this issue, and information about new standard materials as they are acquired.

II. INTRODUCTION

In this section, the objectives of the Materials Characterization Center (MCC), and the structure and functions of the Nuclear Waste Materials Characterization Organization (MCO), (of which the MCC is a part), are briefly outlined. The purpose for and information contained in this listing of approved reference and testing materials are described. Two classifications of standard materials available from the MCC are defined, and the archiving and distribution of the materials are discussed.

The Objective of the MCC

The objective of the MCC is to assist the DOE projects for nuclear waste form production, transportation and disposal to assemble a materials data base of defensible precision and accuracy. A major responsibility of the MCC is to arrange for the formulation, acquisition and distribution of reference and testing materials essential for quantitative evaluation of nuclear waste forms and repository systems under development in the U.S.

The Nuclear Waste Materials Characterization Organization

The MCC is part of the Nuclear Waste Materials Characterization Organization (MCO). The MCO, created by the Department of Energy (DOE), coordinates the collection of a defensible materials property data base. This data base can be used as a recognized, referenceable source of data for waste management systems design, integration, and licensing.

The MCO is coordinated by the Materials Steering Committee (MSC) and it consists of the Materials Integration Office (MIO), the MCC, and the Materials Review Board (MRB). The MCO is structurally arranged as shown in Figure 1. The direction of the MCO is determined by means of quarterly meetings of the MSC in which issues affecting the overall program objectives are addressed and resolved. The MIO functions to integrate the activities of the MCC and the MRB to assure that the test methods and materials data needed by the waste management community are available when required.

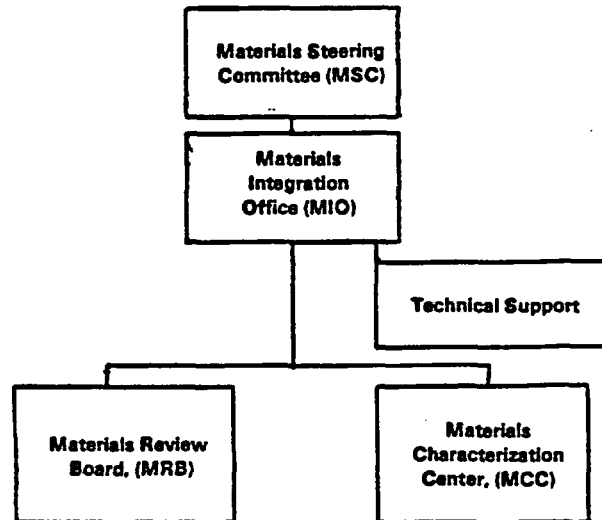


Figure 1. The Structure of the Materials Characterization Organization

This Document

The purpose of this document, which will be updated periodically, is to inform members of the nuclear waste management research and development community about the reference and testing materials available from the MCC. The fabrication and compositions of these materials are reported. Also reported are summaries of any completed characterization and the intended uses for these materials. New materials which are being acquired or are under consideration are also described.

Classifications of Standard Materials

MCC standard materials are intended to provide a basis for comparison of data quality obtained in repository development work by the DOE projects, and to assist in verifying the accuracy and precision of the data obtained. For that purpose, two classifications of standard materials are available from the MCC. These are Approved Reference Materials (ARMs) and Approved Testing Materials (ATMs). These two types of materials are described in this section.

Approved Reference Materials

The primary use of ARMs is as a standard reference and calibration material, similar to that of the SRMs of the United States National Bureau of Standards (NBS). The characterization of each ARM (composition, homogeneity, behavior under specific MCC test conditions) is conducted by MCC and supporting laboratories such as NBS to a high level of accuracy and precision. The ARMs are intended especially for field calibration of MCC test methods, and for verification by test method users that they are conducting the test procedure in a controlled reproducible manner. Therefore, the primary requirement for each ARM is a well-defined uniform composition representing a class of waste form, rather than a specific formulation.

Only one ARM is currently available. The MCC has no present plans for other ARMs.

Approved Testing Materials

The purpose of ATMs is to serve as common testing materials, officially endorsed by DOE, for use in the DOE nuclear waste repository development projects. ATMs represent specific formulations of typical waste forms, or of other components of proposed repository systems. Each ATM will be characterized appropriately by MCC for composition, homogeneity and behavior under specified test conditions. Accuracy and precision of data regarding performance under test conditions will be determined from consensus-average data obtained by both the MCC and DOE projects. The primary objective is to develop a defensible data base of known accuracy and precision using these commonly accepted testing materials of recognized quality.

ATMs will be sufficiently numerous to allow controlled testing under all reasonable conditions required by the DOE repository development projects. The ATMs may be produced by the MCC or by the waste-form producer. In every case the MCC will conduct sufficient characterization to validate the use of the material as an ATM.

Archiving and Distribution of Standard Materials

An important function of MCC is the maintenance of a complete archive of all MCC reference and testing materials used in the nuclear repository development programs, and of the characterization documentation for those materials. Sufficient quantities of these materials will be stored by MCC at PNL to assure their future availability in reasonable quantities for additional testing or resolution of important questions raised in scheduled tests and experiments.

The MCC will be responsive to the needs of the waste management community for high quality reference and testing materials. Readers interested in obtaining any of the items listed in this document or additional materials not listed, should communicate their needs to the MCC. Address requests for existing or new standard materials to:

J. E. Mendel, Manager
Materials Characterization Center
Pacific Northwest Laboratory
Richland, Washington 99352

III. SUMMARY OF STANDARD MATERIALS

In this section, the materials available from the MCC are summarized in tabular form. The MCC's policies for acquiring, characterizing and distributing the materials are outlined.

Inventory of Available Standard Materials

Table 1 provides a summary of the reference and testing materials available from the MCC. The MCC plans the use of its manpower and funding carefully to maximize the number and quality of reference and testing materials for application to the U.S. nuclear waste R&D programs. In addition, funding is allotted to a careful characterization of each material consistent with its intended use. This characterization is an essential step in certifying with confidence that the material is suitable for use as a standard to verify test performance, or as a reference point to compare materials or test conditions.

Further acquisitions are planned. Users are encouraged to communicate their needs to the MCC.

Materials Policies

The MCC acquires and distributes reference and testing materials which are of wide use to the nuclear waste management research and development community. These materials are provided without charge, in reasonable amounts, to DOE programs. Others, such as Nuclear Regulatory Commission (NRC) users, will pay pro-rated production costs. The materials will normally be supplied in the form in which they were produced (e.g., glass will be furnished as cast bars). Some laboratory test programs require use of the materials in a special form (e.g., sized blended powders, specially shaped monoliths, unique containment), or with special characterization. Usually the MCC can supply the materials in the form required, providing there is sufficient prior notice.

The MCC will characterize all reference and testing materials and provide characterization results to the users. As a minimum, the composition and homogeneity will be determined. Statistical

TABLE 1. MCC Reference and Testing Materials

Designation	Type	Purpose	Quantity Fabricated
ARM-1	Reference Waste Glass, nonradioactive	Waste Form Reference	255 kg
ATM-1	76-68 glass, U-doped	Repository Development Tests	4 kg
ATM-2	76-68 glass, Am-doped	Repository Development Tests	0.45 kg
ATM-3	76-68 glass, Np-doped	Repository Development Tests	0.45 kg
ATM-4	76-68 glass, Pu-doped	Repository Development Tests	0.45 kg
ATM-5	76-68 glass, fully radioactive	Repository Development Tests	3 kg
ATM-6	Barnwell glass, radioactive dopants	Repository Development Tests	3 kg
ATM-101	Spent Fuel, Typical PWR	Repository Development Tests	9 pins
MCC 76-68	76-68 glass, nonradioactive	Selected Applications	200 kg

methods will be applied to sampling procedures and evaluation of uncertainty in the analytical data. Additional characterization will generally be applied consistent with the material's intended use. Reference waste form materials will be subjected to MCC leach tests to provide reference data for verification of users' test performance. Microstructure and phase analyses will be conducted on solid materials.

Documentation reports will be available for all reference and testing materials supplied by the MCC. These reports will include details of materials fabrication and the results of its characterization. Also

included, when available, will be a statistical analysis of the uniformity of the material's composition, phases, etc. These reports will be provided to the requestors of a reference or testing material, or to a program that needs to examine such a report prior to a material request.

In order to provide materials as quickly as possible, and because of the time required for adequate characterization, materials are being made available before all characterization has been completed and compiled into a formal documentation. Reports of additional characterization will be distributed as the information becomes available.

IV. STANDARD MATERIALS DESCRIPTIONS

In the following section, the presently available reference and testing materials are described. In addition to information on composition, the methods of fabrication, characterization and reporting of the materials are outlined. Detailed characterization of these materials is in progress. The compositional information listed for each material is based on nominal values or initial analyses. This information will be updated as characterization work progresses.

The homogeneity of standard materials is of particular importance, and at the same time is difficult to achieve with low concentrations of dopants added to bulk quantities of glass. In addition to the problem of thorough uniform mixing of additives, some components of ATM glasses tend to segregate, precipitate or crystallize under glass forming conditions. Characterization in progress on the ATMs includes a careful determination of material homogeneity. Where such information is already available it is included here in the narrative description of the standard material.

ARM-1

Reference Waste Glass, Nonradioactive

This material represents a generic borosilicate reference waste glass. It is nonradioactive. It was produced by Schott Optical Glass of Duryea, PA in 1983, in the form of 6 x 7 x 50 cm bars.

Its composition is shown in Table 2, based on analyses by the National Bureau of Standards. Flame atomic absorption spectrometry and direct-current plasma emission spectrometry were used for the analyses. Each value is based on the analysis of ten statistically random samples of the material. Each sample was analyzed twice. Refractive index measurements confirmed the high homogeneity of the glass.

Additional characterization of the glass will include analyses of the residual strain and numbers and types of inclusions. The glass behavior in various MCC tests will also be determined.

Current plans call for a report to be completed by March of 1984 which will cover ARM-1's fabrication, characterization and behavior in the 28-day MCC-1 leach test. Additional test behavior information will be added to this document as it becomes available.

TABLE 2. Composition of ARM-1(a)

Oxide	Wt%	Standard Deviation
Al ₂ O ₃	5.59	0.06
B ₂ O ₃	11.3	0.25
BaO	0.658	0.008
CaO	2.24	0.03
CeO ₂	1.51	0.04
Cs ₂ O	1.17	0.03
Li ₂ O	5.08	0.13
MoO ₃	1.66	0.03
Na ₂ O	9.66	0.11
Nd ₂ O ₃	5.96	0.20
P ₂ O ₅	0.65	0.03
SiO ₂	46.5	0.9
SrO ₂	0.453	0.005
TiO ₂	3.21	0.05
ZnO	1.46	0.03
ZrO ₂	1.80	0.03
TOTAL	99.90	

(a) Based on analysis of ten random samples,
each sample analyzed twice.

**ATM-1, 2, 3 and 4
76-68 Type Glass,
Actinide Dopants**

These materials represent borosilicate glasses which incorporate high-level wastes resulting from the reprocessing of commercial nuclear reactor fuel. Their compositions are based on 76-68 glass (Mendel, 1977). Each contains a single actinide dopant, either natural U, Am-241, Np-237 or Pu-239; no other radioactive species are present. Glass compositions are given in Tables 3 and 4.

These glasses were prepared at PNL in 1983 from crushed MCC 76-68 glass to which dopants were added. Each batch was melted in platinum crucibles and bars were cast and annealed. The bars of ATM-1b are ~2 x 2.5 x 17.5 cm and weigh ~275 g each. The bars of ATM-1a, 2, 3 and 4 are 1.3 x 1.3 x 7.6 cm and weigh ~36 g.

TABLE 3. Composition of ATM-1 (U-doped 76-68 glass)

Oxide	ATM-1a(a)		ATM-1b(b)	
	Wt%	Standard Deviation	Wt%	Standard Deviation
Al ₂ O ₃	0.59	0.02	0.80	0.02
²⁴¹ Am ₂ O ₃	—	—	—	—
B ₂ O ₃	9.06	0.01	11.3	0.25
BaO	0.55	0.00	0.66	0.01
CaO	2.37	0.04	2.30	0.01
CeO ₂	0.92	0.01	1.13	0.12
Cr ₂ O ₃	0.47	0.01	0.48	0.02
Cs ₂ O	1.11	0.06	1.07(b)	—
Fe ₂ O ₃	9.56	0.13	8.7	0.06
La ₂ O ₃	4.16	0.05	4.73	0.02
MoO ₃	1.97	0.03	2.05	0.01
Na ₂ O	12.77	1.21	12.0	0.13
Nd ₂ O ₃	1.41	0.02	1.65	0.01
NiO	0.23	0.01	0.24	0.01
²³⁷ NpO ₂	—	—	—	—
P ₂ O ₅	0.80	0.16	0.70	0.06
²³⁹ PuO ₂	—	—	—	—
SiO ₂	42.00	0.26	39.9	0.18
SrO	0.42	0.01	0.48	0.00
TiO ₂	3.08	0.05	2.79	0.01
UO ₂	3.98(c)		3.75	0.13
ZnO	5.10	0.46	4.41	0.06
ZrO ₂	1.89	0.04	1.98	0.07

(a) Prepared from MCC-76-68 glass lot 3 (see Table 7); composition listed is that of the base glass except for UO₂.

(b) Prepared from MCC 76-68 glass lot 1 (see Table 7); composition listed is the initial analysis of the ATM.

(c) Wt% UO₂ added to the base glass; not yet analyzed in the ATM.

Alpha autoradiography has been performed on cut bar sections of ATM-2, 3 and 4. The Pu in ATM-4 was fairly uniformly distributed within the bar. There was some layering of Np within the ATM-3. This appears to have occurred during bar pouring. The Am in ATM-2 is very uniform in the center of the bar, but has a lower concentration around the bar's exterior surface.

Additional characterization of these glasses will include determining the actual compositions of ATM-1a, 2, 3 and 4. Also, the crystallinity, presence of phase separation and homogeneity of ATM-1 through 4 will be analyzed.

TABLE 4. Composition of ATM 2, 3 and 4 (Transuranium-doped 76-68 glass)

Oxide	ATM-2(a)		ATM-3(a)		ATM-4(a)	
	Wt%	Standard Deviation	Wt%	Standard Deviation	Wt%	Standard Deviation
Al ₂ O ₃	0.59	0.02	0.59	0.02	0.59	0.02
²⁴¹ Am ₂ O ₃	0.060(b)	—	—	—	—	—
B ₂ O ₃	9.06	0.01	9.06	0.01	9.06	0.01
BaO	0.55	0.00	0.55	0.00	0.55	0.00
CaO	2.37	0.04	2.37	0.04	2.37	0.04
CeO ₂	0.92	0.01	0.92	0.01	0.92	0.01
Cr ₂ O ₃	0.47	0.01	0.47	0.01	0.47	0.01
Cs ₂ O	1.11	0.06	1.11	0.06	1.11	0.06
Fe ₂ O ₃	9.56	0.13	9.56	0.13	9.56	0.13
La ₂ O ₃	4.16	0.05	4.16	0.05	4.16	0.05
MoO ₃	1.97	0.03	1.97	0.03	1.97	0.03
Na ₂ O	12.77	1.21	12.77	1.21	12.77	1.21
Nd ₂ O ₃	1.41	0.02	1.41	0.02	1.41	0.02
NiO	0.23	0.01	0.23	0.01	0.23	0.01
²³⁷ NpO ₂	—	—	0.310(b)	—	—	—
P ₂ O ₅	0.80	0.16	0.80	0.16	0.80	0.16
²³⁹ PuO ₂	—	—	—	—	0.062(b)	—
SiO ₂	42.00	0.26	42.00	0.26	42.00	0.26
SrO	0.42	0.01	0.42	0.01	0.42	0.01
TiO ₂	3.08	0.05	3.08	0.05	3.08	0.05
UO ₂	—	—	—	—	—	—
ZnO	5.10	0.46	5.10	0.46	5.10	0.46
ZrO ₂	1.89	0.04	1.89	0.04	1.89	0.04

(a) Prepared from MCC 76-68 glass lot 3; composition listed is that of the base glass except for the transuranium oxide.

(b) Based on a single measurement in the ATM.

**ATM-5
76-68 Type Glass,
Fully Radioactive**

This material represents a borosilicate glass which incorporates the high-level waste resulting from the reprocessing of commercial nuclear fuel. Its composition is based on 76-68 glass (Mendel, 1977). ATM-5 is fully loaded with actual waste from the reprocessing of commercial nuclear fuel. This waste was generated during the Nuclear Waste Vitrification Project (NWVP) at PNL (Wheelwright, 1979).

ATM-5 was fabricated at PNL in 1983. The nonradioactive ingredients were combined, melted and crushed to form a base glass. All subsequent processing was performed in a hot cell. Dried and powdered NWVP waste was added to the base glass. The powdered waste was made by heating NWVP waste solution to drive off water and NO_x. Oxides of Np, Pu and U were added to the batch to bring their levels to those in the 76-68 composition. The glass was produced in the form of cast, annealed bars ~2 x 2 x 18 cm, weighing ~180 g.

The anticipated composition of ATM-5 is shown in Table 5. This is based on analyses of the powdered waste and nonradioactive base glass. The actual glass composition, crystallinity, homogeneity and phases will be determined in early 1984.

A report is presently available which covers the fabrication of ATM-5. A supplement to this report covering characterization will be prepared by late 1984.

TABLE 5. Anticipated Composition of ATM-5(a)

Oxide	Anticipated Composition, wt%
Glass Additives	
B ₂ O ₃	9.50
CaO	2.00
Na ₂ O	13.01
SiO ₂	40.00
TiO ₂	3.00
ZnO	5.00
Waste Components	
Ag ₂ O	0.03(b)
BaO	0.84
CdO	0.03(b)
CeO ₂	1.03
Cr ₂ O ₃	0.54
Cs ₂ O	1.02(b)
Eu ₂ O ₃	0.07(b)
Fe ₂ O ₃	7.56
Gd ₂ O ₃	0.05(b)
La ₂ O ₃	0.54
MoO ₃	1.15
Na ₂ O	5.51
Nd ₂ O ₃	1.81
NiO	0.29
P ₂ O ₅	0.48
PdO	0.53(b)
Pm ₂ O ₃	0.04(b)
Pr ₆ O ₁₁	0.53(b)
Rb ₂ O	0.13(b)
Rh ₂ O ₃	0.18
RuO ₂	0.63
Sm ₂ O ₃	0.33(b)
SrO	0.34
Tc ₂ O ₇	0.46(b)
TeO ₂	0.19
Y ₂ O ₃	0.21(b)
ZrO ₂	1.75(b)
Other	1.20(c)
Actinides	
Am ₂ O ₃	0.16
Cm ₂ O ₃	0.01
NpO ₂	0.31
PuO ₂	0.06
U ₃ O ₈	4.17

(a) Based on analyses of batch components; analyses of ATM not yet completed.

(b) Component of NWVP Waste which has not yet been analyzed. Anticipated amount in ATM-5 based on composition of PW-8 Waste (Mendel 1977).

(c) Minor impurities in NWVP Waste; Al₂O₃, B₂O₃, CaO, MgO, MnO₂, SiO₂, TiO₂, BnO.

ATM-6 Barnwell Type Glass, Radioactive Dopants

This material represents a borosilicate glass which incorporates the anticipated high-level waste from the Barnwell Nuclear Fuel Plant (BNFP) (Anderson, 1982). The glass was requested by the Basalt Waste Isolation Project (BWIP) for use in their repository interactions testing programs. The types and levels of radioactive dopants which it contains were those specified by the BWIP. The composition of the glass former mix (type 83-B-15) to which the simulated waste was added was developed by the Commercial Waste Treatment Program at PNL.

ATM-6 was fabricated at PNL in 1983. It was produced in a hot cell. Radioactive dopants were mixed with a pre-melted and crushed glass which contained the nonradioactive components. The dopants used included depleted U, Cm-244, Th-232, Np-237, Pu-238, 239 and 240, Tc-99, Cs-137 and Se-75. The glass was produced as cast, annealed bars ~2 x 2 x 18 cm, weighing ~180 g each. A portion of this glass was crushed into two size fractions, -60+115 and -115+250 Tyler mesh, using a motor-driven Al₂O₃ mortar and pestle. A few grams of this crushed material are still available.

Table 6 compares the compositions of the bar and crushed materials. It can be seen that except for Na₂O and Tc₂O₇ there is no significant difference between them. This indicates that no impurity was introduced by the crushing operation, nor did any significant segregation occur during sieving. The reason for the Na₂O and Tc₂O₇ difference is being investigated.

ATM-6 has been examined using an SEM/microprobe. Overall, the material was uniform in appearance and composition. No evidence of crystallinity was seen. One small bubble, ~300μ in diameter was found in the specimens examined; flakes of Ru were found in its interior surface. This was the only evidence of inhomogeneity in the material.

Additional characterization of this material will include a crystallinity determination by X-ray diffraction and a homogeneity determination based on compositional analyses.

A report is currently available for ATM-6 which covers its fabrication and characterization. An analysis of the homogeneity of ATM-6 will be available as a supplement in early 1984.

TABLE 6. Composition of ATM-6

Oxide	Approximate Compositions, wt%(a)				
	Bars		Crushed		
	Average(b)	Standard Deviation	Tyler mesh size		
			-60+115	-115+250	-250
Glass Additives					
Al ₂ O ₃	5.64	0.21	5.42	5.54	5.52
B ₂ O ₃	9.19	0.41	9.09	9.36	8.72
Li ₂ O	4.38	0.17	4.21	4.38	4.24
Na ₂ O(c)	14.4	1.3	17.8	17.9	14.6
SiO ₂	49.0	2.4	46.7	47.8	46.1
Waste Components					
Ag ₂ O		—			
BaO		—			
CaO	0.40	0.08	0.63	0.56	0.38
CdO		—			
CeO ₂	1.06	0.07	0.51	0.77	0.84
Cr ₂ O ₃	0.11	0.01	0.11	0.11	0.10
Cs ₂ O	0.74	0.04			
Fe ₂ O ₃	0.53	0.03	0.55	0.66	0.48
Gd ₂ O ₃		—			
MnO ₂	0.12	0.02	0.15	0.15	0.12
MgO	0.22	0.05	0.35	0.33	0.23
MoO ₃	1.54	0.07	1.48	1.50	1.42
Na ₂ O	(Included above)				
NaCl		—			
Na ₂ SO ₄		—			
Nd ₂ O ₃	1.62	0.06	1.26	1.27	1.44
NiO	nd	—	nd	nd	nd
P ₂ O ₅		—			
PbO		—			
PdO		—			
Rb ₂ O		—			
RuO ₂	0.27	0.15	0.29	0.33	0.34
SeO ₂		—			
Sm ₂ O ₃		—			
SnO ₂		—			
SrO	0.29	0.02	0.28	0.23	0.27
TeO ₂	0.13	0.01	0.14	0.12	0.13
TiO ₂	0.02	0.01	nd	nd	nd
Y ₂ O ₃		—			
ZnO	0.09	0.09	0.10	0.10	0.10
ZrO ₂	0.64	0.36	0.15	0.15	0.15
Radioactive Dopants					
²⁴⁴ Cm ₂ O ₃	6.5E-5	2E-6	5.6E-5	6.4E-5	6.3E-5
¹³⁷ Cs ₂ O	3.0E-3	3E-4	3.0E-3	3.3E-3	3.1E-3
²³⁷ NpO ₂	0.358	0.025	0.312	0.368	0.360
²³⁸ PuO ₂	2.5E-3	1E-4	2.5E-3	2.7E-3	2.5E-3
²³⁹⁺²⁴⁰ PuO ₂	2.0E-2	1E-3	2.0E-2	2.2E-2	2.0E-2
⁷⁵ SeO ₂	1.9E-7	6E-9	1.6E-7	1.6E-7	1.3E-7
⁹⁹ Tc ₂ O ₇ (c)	0.128	0.008	0.308	0.305	0.289
U ₃ O ₈	2.4	0.1			
²³² ThO ₂	1.1E-2	4E-3			

(a) Based on initial incomplete analyses. Data omissions in the table indicate that the component is present but analyses are not yet completed.

(b) Average of three samples taken during bar pouring.

(c) Analytical discrepancy between bars and powder is under investigation.

nd = Not detected.

ATM-101
H. B. Robinson Spent Fuel,
Typical PWR

This material represents the class of spent commercial nuclear reactor fuel characterized by low burnup and high in-reactor fission gas release. This fuel came originally from the H.B. Robinson PWR. The fuel's nominal burnup is 30 MWd/KgM. It was removed from the reactor in 1973. It was received by PNL from EG&G, Idaho in November 1983.

Prior to shipment of the rods to PNL, the rods' fission gases were analyzed by EG&G, Idaho and Exxon Nuclear Idaho Company. Analyses included: rod void volume, gas pressure and gas composition. Results of these analyses will be received by the MCC early in 1984. The rods are undergoing additional characterization at PNL. This is expected to include transverse and longitudinal ceramography, gross gamma scanning, inventory calculation and analytical spot-check of inventory (selected transuranics and fission products), and a radial distribution calculation and spot-check by SEM/microprobe or EDX.

Current scheduling calls for a report to be issued in June 1984 covering material characterization and sample preparation.

MCC 76-68
76-68 Type Glass, Used
for Selected Applications

This material represents a borosilicate glass which incorporates the high level waste resulting from reprocessing of commercial nuclear reactor fuel. This glass is nonradioactive. Its composition is based on that of 76-68 glass (Mendel, 1977).

MCC 76-68 was fabricated by Penberthy Electromelt of Seattle, Washington, in 1980. It was supplied in the form of cast, annealed bars, 2.5 x 2.5 x 30 cm. Three lots were produced; the composition of these lots, as determined by Inductively Coupled Plasma spectrometry (ICP) is given in Table 7. The observed variation in composition of random specimens of this material is as much as 10% for some elements. MCC recommends that use of this material be limited to application in which this inhomogeneity is acceptable. One use of this material is as a feed stock for the production of doped ATMs based on 76-68 glass.

TABLE 7. Composition of MCC-76-68 Glass

Oxide	Compositions, wt%(a)					
	Lot 1		Lot 2		Lot 3	
	Wt%	Standard Deviation(b)	Wt%	Standard Deviation(b)	Wt%	Standard Deviation(b)
Al ₂ O ₃	0.84	0.03	0.63	0.03	0.59	0.02
B ₂ O ₃	8.65	0.06	8.99	0.15	9.06	0.01
BaO	0.62	0.01	0.54	0.02	0.55	0.00
CaO	2.29	0.04	2.22	0.11	2.37	0.04
CdO	0.05	0.01	0.05	0.01	0.05	0.01
CeO ₂	<0.02	—	0.82	0.11	0.92	0.01
Cr ₂ O ₃	0.50	0.02	0.47	0.01	0.47	0.01
Cs ₂ O	1.21	0.08	1.14	0.01	1.11	0.06
Dy ₂ O ₃	0.01	0.00	0.01	0.00	0.01	0.00
Eu ₂ O ₃	0.01	0.00	0.01	0.00	0.01	0.00
Fe ₂ O ₃	9.08	0.12	9.52	0.17	9.56	0.13
Gd ₂ O ₃	0.03	0.01	0.02	0.00	0.02	0.00
K ₂ O	0.09	0.03	0.27	0.06	<0.06	—
La ₂ O ₃	4.89	0.06	4.33	0.24	4.16	0.05
MgO	0.18	0.01	0.14	0.01	0.16	0.01
MnO ₂	0.06	0.00	0.06	0.01	0.06	0.01
MoO ₃	2.17	0.01	1.96	0.05	1.97	0.03
Na ₂ O	11.60	0.95	11.30	0.20	12.77	1.21
Nd ₂ O ₃	1.65	0.01	1.47	0.08	1.41	0.02
NiO	0.25	0.03	0.22	0.02	0.23	0.01
P ₂ O ₅	0.56	0.08	0.66	0.11	0.80	0.16
SiO ₂	40.33	0.81	40.73	0.32	42.00	0.26
SrO ₂	0.49	0.01	0.42	0.01	0.42	0.01
TiO ₂	2.86	0.04	3.04	0.03	3.08	0.05
ZnO	4.75	0.08	4.76	0.05	5.10	0.46
ZrO ₂	2.33	0.04	1.79	0.07	1.89	0.04
TOTAL	95.50		95.57		98.76	

(a) Based on analysis of three bars for each lot.

(b) Standard deviation of a single analysis.

Planned Acquisitions

- a) Reference Leachates
- b) A27 Cast Steel,
Basalt, Bentonite
- c) Glass ATMs
- d) Spent Fuel

Additional standard materials will be acquired during 1984:

- a) A series of **reference leachates** will be made available which will approximate the leachates resulting from various MCC test procedures. These materials will be used to confirm that leachate analysis is being performed correctly.
- b) The BWIP has provided the MCC with samples of the **A27 cast steel, basalt and bentonite** used in their testing program. These will be used as reference materials in repository interactions tests.

V. REFERENCES

Anderson, K. J., et al. 1982. *Barnwell Nuclear Fuel Plant (BNFP) Waste Characterization and Waste Treatment*. PNL-3136/Fr-01. Pacific Northwest Laboratory, Richland, Washington.

Mendel, J. E., et al. 1977. *Annual Report of the Characteristics of High-Level Waste Glasses*. BNWL-2252. Battelle, Pacific Northwest Laboratory, Richland, Washington.

Wheelwright, E. J., et al. 1979 *Technical Summary: Nuclear Waste Vitrification Project*. PNL-3038, Pacific Northwest Laboratory, Richland, Washington.

Commercial Waste Treatment Program, Monthly Report. March 1983. Pacific Northwest Laboratory, Richland, Washington.

DISTRIBUTION

<u>No. of Copies</u>		<u>No. of Copies</u>	
OFFSITE			
	DOE Office of Defense Waste & Byproducts Management Washington, DC 20545 ATTN: R. D. Walton, Jr. DP123	2	National Bureau of Standards Washington, DC 20234 ATTN: H. P. R. Frederikse W. K. Haller
2	DOE Office of Geologic Repository Deployment Washington, DC 20545 ATTN: M. W. Frei W. K. Eister	3	Argonne National Laboratory 9700 South Cass Avenue Argonne, IL 60439 ATTN: M. J. Steindler W. B. Seefeldt C. C. McPheeters
	DOE Office of Terminal Waste Disposal and Remedial Action Washington, DC 20545 ATTN: J. A. Coleman NE-25 J. A. Turi NE-25		Brookhaven National Laboratory Upton NY 11973 ATTN: D. G. Schweitzer
2	DOE Chicago Operations Office 9800 South Cass Avenue Argonne, IL 60439 ATTN: S. A. Mann J. C. Haugen	4	Lawrence Livermore National Laboratory P. O. Box 808 Livermore, CA 94550 ATTN: J. Harrar D. Isherwood V. Oversby L. Ballou
2	DOE Savannah River Operations Office P. O. Box A Aiken, SC 29801 ATTN: T. B. Hindman W. J. Brumley	2	Oak Ridge National Laboratory P. O. Box Y Oak Ridge, TN 37830 ATTN: L. R. Dole H. Godbee
2	DOE Albuquerque Operations Office P. O. Box 5400 Albuquerque, NM 87115 ATTN: M. McFadden K. Golliher	2	Sandia National Laboratories P. O. Box 5800 Albuquerque, NM 87107 ATTN: R. B. Diegel M. A. Molecke
2	DOE National Waste Program Office 505 King Avenue Columbus OH 43201 ATTN: R. Wunderlich J. Neff	5	E. I. duPont deNemours Savannah River Laboratory Aiken, SC 29808 ATTN: P. L. Gray M. J. Plodinec J. A. Stone G. G. Wicks E. J. Hennelly
	DOE Nevada Operations Office P. O. Box 14100 Las Vegas, NV 89114 ATTN: D. L. Vieth	3	Catholic University of America Vitreous State Laboratory Keane Hall Washington, DC 20064 ATTN: P. B. Macedo A. Barkatt C. J. Montrose
2	Technical Information Center		
3	Nuclear Regulatory Commission Washington, DC 20555 ATTN: M. J. Bell K. S. Kim E. Wicks		

**No. of
Copies**

- Massachusetts Institute of Technology
Building 13, Room 4062
77 Massachusetts Avenue
Cambridge, MA 02139
ATTN: R. L. Coble
- Pennsylvania State University
Materials Research Laboratory
University Park, PA 16802
ATTN: W. B. White
- University of Colorado
Department of Geological Sciences
Campus Box 250
Boulder, CO 80309
ATTN: D. D. Runnells
- 2 University of Florida
Department of Materials Science
and Engineering
Gainesville, FL 32611
ATTN: L. L. Hench
D. E. Clark
- University of Illinois
Department of Metallurgy and
Mining Engineering
University of Illinois
Urbana, IL 61801
ATTN: H. Birnbaum
- University of Minnesota
Dept. of Chemical Engrg.
Material Science
141 Amundson Hall
Minneapolis, MN 55455
ATTN: W. W. Gerberich
- Alcoa Laboratories
Alcoa Center, PA 14069
ATTN: G. Mac Zura
- 4 Office of Nuclear Waste Isolation
Battelle Project Management Div.
P. O. Box 16594
Columbus, OH 43216-6594
ATTN: Mgr. Quality Assurance Office
D. P. Moak
D. E. Clark
R. W. Cote
- 3 Battelle Memorial Institute
Battelle Columbus Laboratory
505 King Avenue
Columbus, OH 43201
ATTN: N. E. Miller
D. Stahl
J. Means

**No. of
Copies**

- Office of Crystalline Repository
Development
Battelle Project Management Division
P. O. Box 16595
Columbus, OH 43216-6595
ATTN: A. A. Bauer
- J. E. Burke
33 Forest Rd.
Burnt Hills, NY 12027
- 4 EG&G Idaho, Inc.
P. O. Box 1625
Idaho Falls, ID 83415
ATTN: G. Gibson
J. Hinckley
R. Tallman
R. M. Nielson, Jr.
- Enrico Fermi Institute
5640 S. Ellis
Chicago, IL 60647
ATTN: R. Clayton
- Exxon Nuclear Co., Inc.
Idaho Corporation
P. O. Box 2800
Idaho Falls, ID 83401
ATTN: J. R. Berreth
- Mound Facility
P. O. Box 32
Miamisburg, OH 43242
ATTN: E. L. Lewis
- Rockwell International Science Center
1049 Camino Dos Rios
P. O. Box 1085
Thousand Oaks, CA 91360
ATTN: A. B. Harker
- U. S. Steel Corporation
5733 Northumberland St.
Pittsburgh, PA 15217
ATTN: S. K. Coburn
- 2 West Valley Nuclear Services, Inc.
P. O. Box 191
West Valley, NY 14171-0191
ATTN: J. L. Knabenschuh
J. M. Pope

**No. of
Copies**

ONSITE

- 6 DOE Richland Operations Office**
- 3 E. A. Bracken**
P. A. Craig
L. Olson
H. E. Ransom
- 4 Rockwell Hanford Operations**
W. J. Anderson
M. J. Smith
E. H. Randklev
P. J. Salter
- 3 Westinghouse Hanford Company**
B. Einziger
R. L. Knecht
J. J. McCown

**No. of
Copies**

27 Pacific Northwest Laboratory

- W. M. Bowen**
- D. J. Bradley**
- G. L. Bryan**
- T. D. Chikalla**
- J. L. Daniel**
- R. C. Liikala**
- J. E. Mendel**
- M. D. Merz**
- J. L. McElroy**
- G. B. Mellinger**
- D. Rai**
- G. R. Robinson**
- W. A. Ross**
- R. J. Serne**
- S. C. Slate**
- D. M. Strachan**
- R. G. Strickert**
- R. P. Turcotte**
- R. E. Westerman**
- J. F. Williford, Jr.**
- Publishing Coordination**
- Technical Information (2)**