MCC MATERIALS
CHARACTERIZATION CENTER

Fabrication and Characterization of MCC Approved Testing Material—ATM-1 Glass

J. W. Wald

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

Printed in the United States of America
Available from
National Technical Information Service
United States Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161

NTIS Price Codes
Microfiche A01

Printed Copy

Pages	Price Codes
001-025	A02
026-050	A03
051-075	A04
076-100	A05
101-125	A06
126-150	A07
151-175	A08
176-200	A09
201-225	A010
226-250	A011
251-275	A012
276-300	A013

FABRICATION AND CHARACTERIZATION OF
MCC APPROVED TESTING MATERIAL - ATM-1 GLASS

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Pacific Northwest Laboratory
Richland, Washington 99352

PREFACE

This report, prepared by the Materials Characterization Center, is one in a series of PNL-5577 reports on the MCC's nuclear waste glass Approved Testing Materials (ATMs). The suffix number shows the specific ATM described (e.g., ATM-1 is the subject of PNL-5577-1; ATMs -2, -3 and -4 are included in PNL-5577-2,3,4; etc.). Additional reports will be added to the series as the corresponding glass ATMs are produced by MCC. For a summary of existing ATMs, see the most recent issue of PNL-4955, Approved Reference and Testing Materials for Use in Nuclear Waste Management Research and Development Programs. PNL-4955-2 (Mellinger and Daniel) was issued in December 1984.

The series currently planned includes the following reports:

<u>Report No.</u>	<u>Subject Glass</u>
PNL-5577-1	ATM-1
PNL-5577-2,3,4	ATM-2, ATM-3, ATM-4
PNL-5577-5	ATM-5
PNL-5577-6	ATM-6
PNL-5577-8	ATM-8
PNL-5577-9	ATM-9
PNL-5577-10	ATM-10
PNL-5577-11	ATM-11
PNL-5577-12	ATM-12
PNL-5577-18	ATM-18

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1.0 SUMMARY

The Materials Characterization Center (MCC) Approved Testing Material ATM-1 is a borosilicate glass that incorporates nonradioactive constituents and uranium to represent high-level waste (HLW) resulting from the reprocessing of commercial nuclear reactor fuel. Its composition is based upon the simulated HLW glass type 76-68 (Mendel et al. 1977) to which depleted uranium has been added as UO_2 . Three separate lots of ATM-1 glass have been fabricated, designated ATM-1a, ATM-1b, and ATM-1c. These individual lots were made by the MCC at the Pacific Northwest Laboratory (PNL) operated for the Department of Energy (DOE) by Battelle Memorial Institute. The lots were made in the years 1982, 1983, and 1984, respectively. Limited analyses and microstructural evaluations were conducted on each type during 1984.

Each lot of ATM-1 glass was produced from a feedstock melted in an air atmosphere at between 1150-1200°C in platinum crucibles and cast into stress annealed rectangular bars. Bars of ATM-1a were nominally 1.3 x 1.3 x 7.6 cm (~36 g each), bars of ATM-1b were nominally 2 x 2.5 x 17.5 cm (~190 g each) and bars of ATM-1c were nominally 1.9 x 1.9 x 15 cm (~170 g each). Thirteen bars of ATM-1a, 14 bars of ATM-1b, and 6 bars of ATM-1c (used to produce special forms) were produced.

Table 1.1 lists the analyzed composition of 12 random samples from each of lots ATM-1a, ATM-1b, and ATM-1c. The concentrations (except for U and Cs) were obtained by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy (ICP) analysis. Cesium analysis was performed by Atomic Absorption Spectroscopy (AA), while uranium was analyzed by Pulsed Laser Fluorometry. X-ray diffraction (XRD) analysis of four samples randomly obtained from each lot indicated that lot ATM-1a had no detectable crystalline phases (<3 wt%), while ATM-1b and ATM-1c contained ~3-5 wt% (just above estimated detection level) iron-chrome spinel crystals. These concentrations of secondary spinel component are not considered uncommon in this particular borosilicate glass composition. Scanning electron microscopy (SEM) examination of fracture surfaces of random samples from each lot revealed only a random, apparently crystalline, second phase (1 to 10 μ m diameter) and a random distribution of small voids or bubbles (~1 μ m nominal diameter).

TABLE 1.1. Analyzed Composition of ATM-1 Glass^(a)

Oxide	ATM-1a		ATM-1b		ATM-1c	
	wt%	Standard Deviation	wt%	Standard Deviation	wt%	Standard Deviation
Ag ₂ O	--NA	NA--	NA	--		
Al ₂ O ₃	0.64	0.03	0.85	0.02	0.58	0.02
B ₂ O ₃	8.47	0.31	8.55	0.08	9.07	0.07
BaO	0.50	0.02	0.62	0.00	0.54	0.00
CaO	1.99	0.99	2.11	0.03	2.23	0.02
CdO	0.04	0.00	0.04	0.00	0.04	0.00
CeO ₂	0.88	0.03	0.97	0.09	0.90	0.03
Cr ₂ O ₃	0.39	0.02	0.45	0.04	0.42	0.01
Cs ₂ O	0.71	0.04	0.77	0.02	0.78	0.05
Dy ₂ O ₃	<DL, 0.01	--	<DL, 0.01	--	<DL, 0.01	--
Eu ₂ O ₃	NA	--	NA	--	NA	--
Fe ₂ O ₃	8.16	0.29	8.16	0.04	8.70	0.05
Gd ₂ O ₃	<DL, 0.03	--	<DL, 0.03	--	<DL, 0.03	--
K ₂ O	0.56	0.29	0.56	0.15	0.73	0.13
La ₂ O ₃	4.00	0.19	5.07	0.02	4.39	0.03
MgO	0.30	0.00	0.30	0.01	0.24	0.02
MnO ₂	0.03	0.00	0.03	0.00	0.03	0.00
MoO ₃	1.72	0.08	1.99	0.03	1.82	0.01
Na ₂ O	10.14	0.43	9.66	0.08	10.51	0.10
Nd ₂ O ₃	1.34	0.05	1.61	0.03	1.42	0.01
NiO	0.18	0.01	0.22	0.03	0.20	0.01
P ₂ O ₅	0.63	0.05	0.60	0.06	0.60	0.06
PdO	NA	--	NA	--	NA	--
Rh ₂ O ₃	NA	--	NA	--	NA	--
RuO ₂	NA	--	NA	--	NA	--
SiO ₂	38.73	1.35	40.50	0.28	41.2	0.03
Sm ₂ O ₃	NA	--	NA	--	NA	--
SrO	0.37	0.01	0.46	0.01	0.40	0.005
TeO ₂	NA	--	NA	--	NA	--
TiO ₂	2.62	0.09	2.60	0.01	2.82	0.02
UO ₂	3.98	0.11	4.16	0.08	4.02	0.08
Y ₂ O ₃	NA	--	NA	--	NA	--
ZnO	4.15	0.15	4.34	0.03	4.46	0.04
ZrO ₂	1.46	0.05	1.88	0.06	1.54	0.09
Total	91.99		96.50		97.64	

(a) Based on analysis of 12 random samples for each batch; standard deviations for an individual analysis.

NA = Not analyzed.

<DL = Below the detection limit indicated.

While the precision of the analyses shown in Table 1.1 is satisfactory, the accuracy of individual values is uncertain, since the summation is significantly less than 100%. Additional analyses by other laboratories and methods are in progress.

Material from the three lots of ATM-1 glass has been made available to several waste management programs. As of August 1985 the remaining quantities of material still available for distribution are: ATM-1a, 127 g; ATM-1b, 1679 g; ATM-1c, no material available. Requests for materials or services related to this glass should be directed to the Materials Characterization Center Program Office, PNL.

2.0 INTRODUCTION

The objective of the MCC is to assist the DOE projects responsible for nuclear waste form production, transportation, and disposal to assemble a materials database 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 purpose of this document is to provide the fabrication and characterization details of the Approved Test Material (ATM) ATM-1 glass, which was prepared under the MCC charter.

ATM-1 is a borosilicate glass that incorporates nonradioactive constituents and uranium to represent high-level waste (HLW) resulting from the reprocessing of commercial nuclear reactor fuels. Its composition is based upon the simulated HLW glass type 76-68 (Mendel et al. 1977) to which depleted uranium has been added. Three separate lots of ATM-1 were fabricated at different times; the lots are designated ATM-1a, ATM-1b, and ATM-1c in accordance with fabrication order. ATM-1a was prepared at the Pacific Northwest Laboratory (PNL) in December of 1982 at the request of the Salt Repository Investigations Program at PNL for use in testing at PNL in support of the Office of Nuclear Waste Isolation (ONWI). ATM-1b was prepared at PNL in January of 1983 at the request of the Tuff Repository Investigations Project at PNL for use in testing at PNL in support of the Nevada Nuclear Waste Storage Investigations (NNWSI). ATM-1c was produced at PNL in February of 1984 at the request of NNWSI staff at Lawrence Livermore National Laboratory, Livermore, California. Special castings and crushed material were produced for NNWSI from ATM-1c for use in their repository development testing program. Instructions and information on these special forms were provided to the MCC by NNWSI staff in private correspondence.

Section 3 of this report describes the fabrication of the three lots of ATM-1 glass, and Section 4 summarizes the analysis and characterization of the final glass product. The glass lots reported here were produced under the guidance of the MCC Program Quality Assurance (QA) Plan PGM-4, Rev. 2, and PNL

QA Plan PNL-MA-65, which were in effect during the course of this work. No written technical procedures were prepared specifically for the fabrication and characterization of these glass lots. The procedures followed were fully documented in official laboratory record books, as required in the previously listed QA plans.

3.0 FABRICATION^(a)

3.1 FABRICATION OF ATM-1a

The fabrication of ATM-1a glass began on December 20, 1982 and was completed by December 29, 1982. All work involved in the fabrication of this glass took place in Lab 208, 308 Building, 300 Area. ATM-1a was prepared from crushed type 76-68 glass to which depleted uranium dioxide was added.

3.1.1 Starting Materials

The glass which served as the feedstock for ATM-1a was MCC-76-68, Lot 3, bar numbers 265, 276, 281, 307 and 309. The MCC-76-68 glass was produced for PNL by Penberthy Electromelt of Seattle, Washington under purchase order number A2820/A2823AF and received at PNL in September 1980. The average analyzed composition of lot 3 of the MCC-76-68 glass used for ATM-1a is given in Table 3.1; no specific analyses were conducted on the 5 glass bars used to produce ATM-1a. Depleted uranium was added as a nitrate solution for the formulation of ATM-1a. The source of the UO_2 additive was from on-hand stock of 0.15% ^{235}U material. No other historical or analytical information is available for the UO_2 additive.

3.1.2 Batch and Glass Preparation

Table 3.2 lists the calculated oxide composition of ATM-1a glass based on a 3.98 wt% addition of UO_2 to the MCC-76-68, Lot 3 glass. The batching process was conducted as follows:

- Five bars of MCC-76-68, Lot 3 glass, were crushed, ground, and sieved to -100 mesh (-150 μm) using an agate chamber laboratory disc mill (Angstrom), and Newark stainless steel sieves. All material (-100 mesh) was combined into a single container and tumble blended to assure uniformity of mixture.

(a) The use of trade and manufacturers' names in this report is for documentation purposes only and does not imply endorsement by PNL.

TABLE 3.1. Analysis of MCC-76-6B Glass(a)

Oxide(d)	Lot 1 (Feedstock for Glass ATM-1b)		Lot 3 (Feedstock for Glass ATM-1a and ATM-1c)	
	Concentration, wt%(b)		Concentration, wt%(b)	
	wt%	Standard Deviation(c)	wt%	Standard Deviation(c)
Al ₂ O ₃	0.84	0.03	0.59	0.02
B ₂ O ₃	8.65	0.06	9.06	0.01
BaO	0.62	0.01	0.55	0.00
CaO	2.29	0.04	2.37	0.04
CdO	0.05	0.01	0.05	0.01
CeO ₂	<0.02	--	0.92	0.01
Cr ₂ O ₃	0.50	0.02	0.47	0.01
Cs ₂ O	1.21	0.08	1.11	0.06
Dy ₂ O ₃	0.01	0.00	0.01	0.00
Eu ₂ O ₃	0.01	0.00	0.01	0.00
Fe ₂ O ₃	9.08	0.12	9.56	0.13
Gd ₂ O ₃	0.03	0.01	0.02	0.00
K ₂ O	0.09	0.03	<0.06	--
La ₂ O ₃	4.89	0.06	4.16	0.05
MgO	0.18	0.01	0.16	0.01
MnO ₂	0.06	0.00	0.06	0.01
MoO ₃	2.17	0.01	1.97	0.03
Na ₂ O	11.60	0.95	12.77	1.21
Nd ₂ O ₃	1.65	0.01	1.41	0.02
NiO	0.25	0.03	0.23	0.01
P ₂ O ₅	0.56	0.08	0.80	0.16
SiO ₂	40.33	0.81	42.00	0.26
SrO	0.49	0.01	0.42	0.01
TiO ₂	2.86	0.04	3.08	0.05
ZnO	4.75	0.08	5.10	0.46
ZrO ₂	2.33	0.04	1.89	0.04
TOTAL	95.50		98.76	

(a) See Table 12, Mellinger and Daniel, 1984.

(b) Based on ICP analysis of three bars from the 100 bars comprising each lot.

(c) Standard Deviation of an individual analysis.

(d) Oxides listed were components of fabrication specifications for MCC-76-68. Other oxides of interest for ATM-1c glass, neither added to MCC-76-68 nor analyzed, include Ag₂O, PdO, Rh₂O₃, RuO₂, Sm₂O₃, TeO₂, Y₂O₃, and UO₂.

TABLE 3.2. Calculated Bulk Composition
of Glass ATM-1a^(a)

<u>Oxide</u>	<u>wt%</u>
Al ₂ O ₃	0.57
B ₂ O ₃	8.81
BaO	0.53
CaO	2.30
CdO	0.05
CeO ₂	0.89
Cr ₂ O ₃	0.46
Cs ₂ O	1.08
Dy ₂ O ₃	0.01
Eu ₂ O ₃	0.01
Fe ₂ O ₃	9.29
Gd ₂ O ₃	0.02
K ₂ O	<0.06
La ₂ O ₃	4.04
MgO	0.16
MnO ₂	0.06
MoO	1.92
Na ₂ O	12.42
Nd ₂ O ₃	1.37
NiO	0.22
P ₂ O ₅	0.78
SiO ₂	40.83
SrO	0.41
TiO ₂	2.99
UO ₂	3.98
ZnO	4.96
ZrO ₂	1.84
TOTAL	100.00

(a) Based on addition of uranium oxide to the oxide concentration values shown in Table 3.1 (Lot 3), and normalization to 100%. See Table 3.1, footnote d.

- 432.08 g of crushed glass was weighed out and mixed with a nitrate solution containing 17.92 g UO_2 powder dissolved in 30 mL of concentrated HNO_3 and diluted to a final solution volume of 120 mL using deionized water. A target ratio (dopant solution to glass powder) of 25-30 mL per 100 g was sought.
- All glass powder was wetted by the dopant uranium nitrate solution allowing a uniform blending of the slurry by hand stirring. Mix time was approximately 5 minutes. The slurry mixture was dried in a single batch contained in a Pyrex beaker on a conventional hot plate. Total drying time was approximately 4 hours at about 200°C.
- A hardened steel punch and die was used to crush and grind the doped and dried glass mixture by hand to a -20 mesh ($\sim 850 \mu\text{m}$) particle size prior to melting. The batch was melted in a single Pt crucible at 1200°C in an air atmosphere for 5-10 minutes prior to bar pouring. Each melt was mixed just prior to pouring by swirling and agitating the crucible. The molten glass batch was poured into a preheated ($\sim 500^\circ\text{C}$) steel bar mold (nominal dimensions 1.3 x 1.3 x 7.6 cm), allowed to cool enough to facilitate handling, and placed into an annealing furnace at 500°C, held for 2 hours and furnace cooled at $\sim 175^\circ\text{C/h}$ to 25°C. Three separate melting runs were required to produce the initial 13 bars that were formed.

3.1.3 Measurement and Testing Equipment

Fluke Digital Thermometer, Model No. 2190A/Y2001; CALIB. #364-79-06-007

Sartorius Balance, Model No. 202MP; CALIB. #364-06-01-011

Mettler Balance, Model No. HK-160; CALIB. #364-06-01-010

3.1.4 Other Equipment

Deltac DT-31 Furnace

Lindberg Lightweight Box furnace

Angstrom Laboratory Disc Mill, with agate grinding chamber

Corning PC-354 Hot Plate

3.1.5 Procedures

MCC-SP-1 (Draft): Glass Component Preparation

MCC-SP-2 (Draft): Glass Preparation

RWP-308-1, Rev. 14: Lab Specific, General Radiation Work Procedure

3.1.6 Identification System

Glass bars poured in this fabrication work were individually numbered, independent of the pouring order. Bars from this preparation were initially labeled U1 through U13 but have been relabeled ATM-1a-1 through ATM-1a-13. Thirteen bars total were produced with three sizes as follows: 10 each at 1.3 x 1.3 x 7.6 cm; 2 each at 1.3 x 1.3 x 5.1 cm; 1 at 1.3 x 1.3 x 2.5 cm. Production records for this glass are recorded on Page 14 of PNL Laboratory Record Book BNW-3660. Table 3.3 gives the initial and final ID numbers and mass of the bars.

3.1.7 Material Availability and Storage

As of August 1985, approximately 127 g of ATM-1a (bars -2, -3 and -4) are available for distribution. Archive material has been retained from some of the batch melting operations but no bar specific archive samples were retained. The glass and archive material is controlled by the MCC Program Material Custodian and is stored in the MCC assigned storage vault in Corridor 7, 308 Building, 300 Area.

TABLE 3.3. Product Summary for ATM-1a Glass

<u>Initial IO No.</u>	<u>ID No.</u>	<u>Mass, g</u>
U1	ATM-1a-1	38.47
U2	ATM-1a-2	39.87
U3	ATM-1a-3	39.96
U4	ATM-1a-4	25.49
U5	ATM-1a-5	28.59
U6	ATM-1a-6	36.69
U7	ATM-1a-7	30.55
U8	ATM-1a-8	34.58
U9	ATM-1a-9	37.83
U10	ATM-1a-10	36.76
U11	ATM-1a-11	37.91
U12	ATM-1a-12	40.18
U13	ATM-1a-13	12.40

3.2 FABRICATION OF ATM-1b

The fabrication of glass ATM-1b began on January 24, 1983 and was completed by January 28, 1983. All work involved in the fabrication of this glass took place in Lab 500, 325 Building, 300 Area. ATM-1b was prepared from crushed type 76-68 glass to which cerium nitrate and depleted uranium oxide powders were added prior to melting.

3.2.1 Starting Materials

The glass which served as the feedstock for ATM-1b was MCC-76-68, Lot 1, bar numbers 2, 3, 4, 5, 6, 7, and 8. The MCC-76-68 glass was produced for PNL by Penberthy Electromelt of Seattle, Washington under purchase order number A2820/A2823AF and received at PNL in September 1980. The average analyzed composition of Lot 1 of the MCC-76-68 glass used for ATM-1b is given in Table 3.1; no specific analyses were conducted on the glass bars actually used in this work. The additives to this base glass for the formulation of ATM-1b included cerium in the form of $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and depleted uranium in the form of UO_2 powder. Both additives were from on-hand stock; the depleted uranium was 0.15% ^{235}U material. No other historical or analytical information is available for these additives.

3.2.2 Batch Preparation

Table 3.4 lists the calculated oxide composition of ATM-1b glass. Calculations are based upon a 1 wt% addition of CeO_2 and a 4 wt% addition of UO_2 to glass MCC-76-68 Lot 1. The batching process was accomplished as follows:

- Seven bars of glass MCC-76-68, Lot 1, as listed previously, were crushed, ground and sieved to -60 mesh (-150 μm) using a tungsten carbide chamber laboratory disc mill (Angstrom), and Newark stainless steel sieves. All material (-60 mesh) was combined into a single container and stirred by hand to assure a uniform mixture.
- 3200.0 g of crushed glass were weighed out and blended with 85.3 g $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ and 134.7 g of UO_2 powder. This mixture was placed into a hardened porcelain ball mill with burundum grinding media and

TABLE 3.4. Calculated Bulk Composition
of Glass ATM-1b(a)

<u>Oxide</u>	<u>wt%</u>
Al ₂ O ₃	0.84
B ₂ O ₃	8.60
BaO	0.62
CaO	2.28
CdO	0.05
CeO ₂	1.00
Cr ₂ O ₃	0.50
Cs ₂ O	1.20
Dy ₂ O ₃	0.01
Eu ₂ O ₃	0.01
Fe ₂ O ₃	9.03
Gd ₂ O ₃	0.03
K ₂ O	0.09
La ₂ O ₃	4.86
MgO	0.18
MnO ₂	0.06
MoO ₃	2.16
Na ₂ O	11.53
Nd ₂ O ₃	1.64
NiO	0.25
P ₂ O ₅	0.56
SiO ₂	40.11
SrO	0.49
TiO ₂	2.85
UO ₂	4.00
ZnO	4.73
ZrO ₂	2.32
TOTAL	100.00

(a) Based on addition of uranium and cerium oxides to the oxide concentration values shown in Table 3.1 (Lot 1), and normalization to 100%. See Table 3.1, footnote d.

processed for 2 hours. At the completion of the milling period the material was removed from the mill and separated from the grinding media.

- The blended batch was divided into three roughly equal sub-batches for the melting operation. Each sub-batch was: 1) melted in a Pt crucible by placing about 900 g into the melting furnace with an air atmosphere which was preheated to 1100°C; 2) held for 10 minutes and recharged with about 300 g additional batch; 3) held at 1100°C for a total elapsed time of 1.5 hours; 4) held at an increased temperature of 1150°C for 0.5 hours. The crucible was then removed from the furnace, and the glass mixed by swirling and agitating the crucible. The crucible was returned to the furnace for an additional 10 minutes of soaking before pouring the bars.
- Three separate sub-batches were processed as described above to produce the required bars. Bars were formed by pouring molten glass batch from 1150°C into a heated steel bar mold nominally 2.0 x 2.5 x 17.5 cm, allowing each mold to cool enough to facilitate handling, and placing it into an annealing furnace at 500°C, where it was held for 2.5 hours and furnace cooled at ~175°/hr to 25°C. The three sub-batches produced a total of 14 bars.

3.2.3 Measurement and Testing Equipment

Fluke Digital Thermometer, Model No. 2100A, Calib. No. 362-76-06-005

Mettler Balance, Model No. PN5, Calib. No. 362-06-03-069

Mettler Balance, Model No. 323, Calib. No. 362-06-03-067

Mettler Balance, Model No. PC 400, Calib. No. 362-06-03-017

3.2.4 Other Equipment

Deltac DT-31-R-SS Furnace

Lindberg Model 51442 Furnace

Angstrom Laboratory Disc mill with Tungsten Carbide Grinding Chamber

Jar Mill Assembly, Hardened Porcelain

3.2.5 Procedures

MCC-SP-1 (Draft): Glass Component Preparation

MCC-SP-2 (Draft): Glass Preparation

RWS-325-10: Radiation Work Specification, General Facility

RCP-325-10: Radiation Control Protocol, Laboratory Specific

3.2.6 Identification System

Glass bars poured in this fabrication work were individually numbered in accordance with pour order. When the bars were removed from the annealing furnace they were mechanically scribed with a unique number relating to this run. Initially the bars were labeled GLM-1 through GLM-14, but have been relabeled ATM-1b-1 through ATM-1b-14. All 14 bars were nominally the same dimensional size with an average mass per bar of ~190 g. Production records for this glass are recorded on Pages 2-3 of PNL Laboratory Record Book BNW-5501. Table 3.5 gives the initial and final bar ID numbers along with the mass.

TABLE 3.5. Product Summary for ATM-1b Glass

<u>Initial ID No.</u>	<u>ID No.</u>	<u>Mass, g^(a)</u>
GLM-1	ATM-1b-1	159.6
GLM-2	ATM-1b-2	154.5
GLM-3	ATM-1b-3	181.6
GLM-4	ATM-1b-4	164.7
GLM-5	ATM-1b-5	171.6
GLM-6	ATM-1b-6	212.0
GLM-7	ATM-1b-7	186.4
GLM-8	ATM-1b-8	238.0
GLM-9	ATM-1b-9	160.7
GLM-10	ATM-1b-10	204.0
GLM-11	ATM-1b-11	193.0
GLM-12	ATM-1b-12	188.2
GLM-13	ATM-1b-13	177.9
GLM-14	ATM-1b-14	215.3

(a) Some masses are for bars after removal of archive and analytical samples.

3.2.7 Material Availability and Storage

As of August 1985 approximately 1679 g of ATM-1b are available for distribution in bar form. Archive material has been retained from some of the batch melting operations and a few of the bars have been distributed. The glass and archive material are controlled by the MCC Program Material Custodian and stored in the MCC assigned storage vault in Corridor 7, 308 Building, 300 Area.

3.3 FABRICATION OF ATM-1c

The fabrication of glass ATM-1c began on January 11, 1984 and was completed by February 13, 1984. All work involved in the fabrication of this glass took place in Lab 500, 325 Building, 300 Area. ATM-1c glass was prepared from crushed type 76-68 Glass to which depleted uranium oxide powder was added prior to melting.

3.3.1 Starting Materials

The glass which served as the feedstock for ATM-1c was MCC-76-68, Lot 3, bar numbers 249, 251, 261, 264, 275, and 315. The MCC-76-68 glass was produced for PNL by Penberthy Electromelt of Seattle, Washington under purchase order number A2820/A2823AF and received at PNL in September 1980. The average analyzed composition of Lot 3 of the MCC-76-68 Glass used for ATM-1c is given in Table 3.1; no specific analyses were conducted on the glass bars actually used in this work. For the formulation of ATM-1c, uranium was added in the form of UO_2 powder. The source of the UO_2 additive was on-hand stock of 0.15% ^{235}U material. No other historical or analytical information is available for the UO_2 additive.

3.3.2 Batch Preparation

Table 3.6 lists the requested oxide composition of ATM-1c as stated in the letter of request for NNWSI Glass Composition I (private correspondence, Ballou to Mendel, November 30, 1983). The batching process was accomplished as follows:

- Six bars of glass MCC-76-68, Lot 3, as listed above, were crushed, ground, and sieved to a -100 mesh (-150 μm) using a tungsten carbide disc mill.

TABLE 3.6. Requested Oxide Composition of Glass ATM-1c
(NNWSI Glass I)

Oxide	wt%
<u>Inerts</u>	
Al ₂ O ₃	0.49
B ₂ O ₃	9.17
CaO	2.30
Cr ₂ O ₃	0.34
Fe ₂ O ₃	8.95
K ₂ O	0.20
MnO ₂	0.08
Na ₂ O	12.9
NiO	0.20
P ₂ O ₅	0.68
SiO ₂	41.3
TiO ₂	2.95
ZnO	4.45
<u>Fission Products</u>	
Ag ₂ O	0.03
BaO	0.53
CdO	0.03
CeO ₂	1.00
Cs ₂ O	1.00
La ₂ O ₃	1.85
MoO ₂	1.78
Nd ₂ O ₃	1.30
PdO	0.57
Rh ₂ O ₃	0.17
RuO ₂	1.00
Sm ₂ O ₃	0.12
SrO	0.40
TeO ₂	0.28
Y ₂ O ₃	0.21
ZrO ₂	1.62
<u>Actinides</u>	
UO ₂	4.10
TOTAL	100.00

- 2880.6 g of the crushed glass were weighed out and blended with 119.4 g of UO_2 powder. This mixture was then placed into a porcelain ball mill with burundum grinding cylinders and milled for 6 hours. Four random samples were taken from the mill and submitted for compositional analysis after the milling operation was completed.
- The average UO_2 analysis of the resulting batch was 4.2 wt%, slightly higher than the intended concentration. Additional crushed glass from the initial batching step was added to the batch and ball milled for 3 hours. The average UO_2 concentration of four random samples from this mixture was 3.98 wt%.
- The blended material was melted in a Pt crucible in 2 increments of ~1500 g each, on 2 different days. The melting was conducted at 1150°C in an air atmosphere for 0.5 hour at which time it was removed from the furnace, swirled and agitated for stirring and returned to the furnace for an additional 2.5 hours. Six bars were initially poured directly from this batch. The molten glass was poured into a steel mold nominally 1.9 x 1.9 x 15 cm, allowed to cool enough to facilitate handling, and placed into an annealing furnace at 500°C for 2 hours minimum and furnace cooled at ~50°/hr to 25°C.
- The glass bars produced in the previous step were brown-colored rather than the expected black so the bars were reprocessed as follows. The six bars were coarsely ground and remelted as previously described. Bars cast from this remelting were annealed at 480°C for 2 hours and cooled as described previously. The bars from this melting were the expected black color.

3.3.2.1 Preparation of Special Forms

Six special forms were requested for delivery to NNWSI (Table 3.7): crushed and sized powder, discs and cylinders with and without stainless steel casings, and tapered cast cylinders. The production of these special forms from the glass fabricated above is described in the following sections.

3.3.2.1.1 Crushed and Sized Powder. Crushed glass was produced from a portion of a cast bar by crushing and grinding in a laboratory disc mill using

TABLE 3.7. Special Form Requirements Requested by NNWSI for
MCC ATM-1c Glass

Form	Physical Description	Quantity Requested
A	Crushed glass, -40 to +80 mesh size	50 g
B	Discs, 1-cm dia. x 3 mm-thick	80 discs
C	Discs, dimensions as in "B" but with 304L SS rims	30 discs
D	Cylinder, 1-cm dia. x 1- cm long	10 cylinders
E	Cylinders, dimensions as in "D" but in 304L SS casing	10 cylinders
F	Tapered cast cylinders (1° Taper), 1.6-cm dia. x 2-cm long	20 cylinders

a tungsten carbide grinding chamber. Material was recovered from the size fraction -40 to +80 mesh (U.S. sieve designation) and packaged for shipment. Sizing was performed by manually working the crushed material through the stainless steel sieves with brushing and tapping actions.

3.3.2.1.2 Uncased Discs and Cylinders. Cast bars were cut into ~5 cm lengths in preparation for core drilling. Cylindrical cores were obtained from the cast bars by diamond core drilling with a water cooled Starlite Industries core drill assembly. These cores were then cut to size using a Buehler Isomet low speed saw with a low concentration diamond wafering blade and water as the cutting media. Discs were nominally 3-mm thick by 1-cm diameter; whole cylinders were 1-cm long by 1-cm diameter. Eighty discs and 10 cylinders were produced.

3.3.2.1.3 Stainless-Steel Clad Discs and Cylinders. Scraps and pieces from the production of core-drilled samples and cutting remains were used for an additional melt to cast glass inside 304L stainless-steel tubing. Molds were fabricated by cutting 5-cm long sections from seamless 304L stainless-steel tubing 1 cm in outside diameter. The nominal wall thickness of this tubing was 0.9 mm. The glass material was melted at 1250°C for approximately 30 minutes and poured into the tubular molds which had been preheated to 480°C. After casting was complete the tubes were annealed at 480°C for a minimum of 2 hours and furnace cooled to 25°C. The tubular castings were cut to size using a Buehler Isomet low speed saw with a low concentration diamond

wafering blade and water as the cutting medium. Discs were nominally 3-mm thick by 1-cm diameter, while cylinders were 1-cm long by 1-cm diameter. Thirty discs and 10 cylinders were produced.

3.3.2.1.4 Tapered Cast Cylinders. The following fabrication outline lists the steps that were followed to produce the tapered cast cylinders (Form F) of ATM-1c glass. The required cast glass dimensions were specified by NNWSI (private correspondence, Bates to Mellinger, May 17, 1984). The casting crucibles were specified by NNWSI as "a Pt 5% Au mold, ...a modified J. Lawrence Smith crucible from Englehard Industries, per ANL purchase order 055090." The crucibles for this work were obtained from Johnson Matthey Co. on PNL P.O. J 7160 ARO using the same specification as above with the concurrence of NNWSI staff.

- Previously melted glass bars were remelted to form the feed material for the present castings. Melting was conducted in a Pt crucible at 1150-1200°C in an air atmosphere, a temperature sufficient to produce satisfactory casting behavior. Residence time at temperature was approximately 30 minutes.
- Molten glass was poured into the casting crucible mold which had been preheated to 500°C; the mold was completely enclosed in a mold/firebrick assembly. The assembly was transferred to an annealing furnace and held at 500°C for 30 minutes; cooled at approximately 100°C/h to 350°C; removed from the firebrick and allowed to cool to room temperature.
- The glass casting was removed from the crucible by inverting the crucible and gently shaking and tapping the bottom. Twisting of the glass casting in the crucible was avoided to minimize glass surface scratching.
- Finished samples were a tapered right-circular cylinder cut to a length of 2.00 ± 0.1 cm with parallel cut ends normal to the cylinder axis. Cutting to dimensions was performed on a Buehler low speed cut-off saw using a low concentration diamond blade (No. 11-4254), at a speed setting of "6" and water as the cutting fluid. A specially

padding double jaw chuck was used to hold the samples for the entire cutting operation. A ring gauge was used to establish the first cut, and a dimensional shift of the chuck and sample was made to position the sample for the second cut.

3.3.3 Measurement and Testing Equipment

Fluke Digital Thermometer, Model No. 2100A, Calib. #362-79-06-005

Mettler Balance, Model No. PC 400, Calib. #362-06-01-017

3.3.4 Other Equipment

Deitec DT-31-1255 Furnace

Norton Jar Mill

Angstrom Disc Mill w/Tungsten Carbide Chamber

Cut-off Saw, Buehler Isomet

Core Drill, Starlite Industries

3.3.5 Procedures

MCC-SP-1 (Draft): Glass component Preparation

MCC-SP-2 (Draft): Glass Preparation

RWS-325-10: Radiation Work Specification, General Facility

RCP-325-10: Radiation Control Protocol, Laboratory

3.3.6 Identification System

Glass bars produced in this fabrication work were not individually numbered or otherwise identified individually as they were poured. Bars were subsequently numbered in random order for purposes of identification. No other similar activities occurred in the lab during this period, and the identity of the material was maintained by collection and storage in a singular location or container. This container was marked "MCC:ATM-1c GLASS." Fourteen glass bars were produced from the batch of ATM-1c material, some of which were subsequently core drilled to produce cylinders and discs, crushed to produce powdered material, and remelted to produce special castings. Production records for this glass are recorded on Pages 9-11 of PNL Laboratory Record Book BNW-5501.

3.3.7 Material Availability and Storage

All ATM-1c glass was distributed to NNWSI, and no material is available for general distribution. Archive samples remaining from the initial as-cast bars are controlled by the MCC Program Material custodian and are stored in the MCC assigned storage vault in Corridor 7, 308 Building, 300 Area. No archive samples were retained for the special NNWSI samples.

4.0 CHARACTERIZATION

Characterization of the three lots of ATM-1 glass consisted of bulk physical measurements, chemical analysis, scanning electron microscopy (SEM), and x-ray diffraction (XRD) analysis. The results of these analyses on ATM-1a, -1b, and -1c are detailed in the following sections according to analytical procedure.

4.1 BULK DENSITY MEASUREMENTS

Bulk density was determined on three samples taken from each glass lot by ASTM procedure C 693-74, "Standard Method for Density of Glass by Buoyancy." The technique was modified by using 2-g samples instead of the 20-g samples specified. Density samples were prepared by removing a section from the end of randomly selected bars and trimming off all cast surfaces using a Buehler Isomet saw, using a low concentration diamond wafering blade and water coolant. Before measurement, all samples were cleaned twice in acetone in an ultrasonic cleaner and dried at approximately 100°C. Table 4.1 summarizes the density measurements.

TABLE 4.1. Measured Bulk Density of ATM-1 Glass

<u>Glass Lot</u>	<u>Identity of Bars Used for Specimens</u>	<u>Average Measured Density, g/cm³</u>
ATM-1a	3, 4, 5	3.025 ± 0.002
ATM-1b	1, 9, 14	3.045 ± 0.003
ATM-1c	5, 7, 8	3.017 ± 0.003

4.2 CHEMICAL ANALYSIS

Chemical analysis was performed by Inductively-Coupled Argon Plasma Atomic Emission Spectroscopy (ICP) for the majority of the elements. Uranium was analyzed by pulsed laser fluorometry and cesium by Atomic Absorption Spectroscopy. ICP analysis was performed on an ARL-3650 spectrometer, laser fluorometry was performed on a Scintrex Model 3-A Uranium Analyzer and AA was performed on a Perkin-Elmer Model 305 spectrometer. Solid analytical samples were ground to -100 mesh (-150 μm) duplicate samples fused with KOH and with Na_2O_2 , and dissolved in dilute HCl to a known volume for analysis.

The results of the chemical analysis of 12 samples from each glass batch are given in Table 4.2. Analysis of ATM-1a glass was conducted on randomly selected remnants of the bars used to prepare test specimens. For ATM-1b glass, 12 samples for analysis were taken from individual bars (number 1, 2, 3, 4, 5, 7, 9, 12, and 13). Similarly, 9 of the unnumbered ATM-1c bars were used to provide 12 analytical samples for analysis.

It should be noted that the satisfactory precision shown for individual element analyses indicates that all 3 glass lots are homogeneous, but the summation of concentrations is significantly less than 100%, even considering probable concentrations of elements not analyzed. Therefore, the accuracy of individual values is uncertain, and simple normalization of all data equally to achieve 100% is not justified. Additional analyses by independent laboratories and methods are in progress.

4.3 MICROSCOPIC EXAMINATION

Scanning electron microscopy was conducted on four fracture samples of each of the glass lots prepared from sections removed from the ends of three of the bars (one end from two bars, two ends from one bar) used for chemical analysis. Figure 4.1 illustrates typical microstructures observed in ATM-1 glass. The microscopy showed a random distribution of small voids or bubbles (~1 μm nominal diameter) as well as a minor microstructural features, apparently crystalline and high in Fe, probably a spinel. SEM examination was performed on a JEOL Model JSM 35C Scanning Electron Microscope with a Tracor Northeqn Model TN-2000 Energy Dispersive Spectrometer.

TABLE 4.2. Analyzed Composition of ATM-1 Glass(a)

Oxide	ATM-1a		ATM-1b		ATM-1c	
	wt%	Standard Deviation	wt%	Standard Deviation	wt%	Standard Deviation
Ag ₂ O	NA	--	NA	--	NA	--
Al ₂ O ₃	0.64	0.03	0.85	0.02	0.58	0.02
B ₂ O ₃	8.47	0.31	8.55	0.08	9.07	0.07
BaO	0.50	0.02	0.62	0.00	0.54	0.00
CaO	1.99	0.99	2.11	0.03	2.23	0.02
CdO	0.04	0.00	0.04	0.00	0.04	0.00
CeO ₂	0.88	0.03	0.97	0.09	0.90	0.03
Cr ₂ O ₃	0.39	0.02	0.45	0.04	0.42	0.01
Cs ₂ O	0.71	0.04	0.77	0.02	0.78	0.05
Dy ₂ O ₃	<DL, 0.01	--	<DL, 0.01	--	<DL, 0.01	--
Eu ₂ O ₃	NA	--	NA	--	NA	--
Fe ₂ O ₃	8.16	0.29	8.16	0.04	8.70	0.05
Gd ₂ O ₃	<DL, 0.03	--	<DL, 0.03	--	<DL, 0.03	--
K ₂ O	0.56	0.29	0.56	0.15	0.73	0.13
La ₂ O ₃	4.00	0.19	5.07	0.02	4.39	0.03
MgO	0.30	0.00	0.30	0.01	0.24	0.02
MnO ₂	0.03	0.00	0.03	0.00	0.03	0.00
MoO ₃	1.72	0.08	1.99	0.03	1.82	0.01
Na ₂ O	10.14	0.43	9.66	0.08	10.51	0.10
Nd ₂ O ₃	1.34	0.05	1.61	0.03	1.42	0.01
NiO	0.18	0.01	0.22	0.03	0.20	0.01
P ₂ O ₅	0.63	0.05	0.60	0.06	0.60	0.06
PdO	NA	--	NA	--	NA	--
Rh ₂ O ₃	NA	--	NA	--	NA	--
RuO ₂	NA	--	NA	--	NA	--
SiO ₂	38.73	1.35	40.50	0.28	41.2	0.03
Sm ₂ O ₃	NA	--	NA	--	NA	--
SrO	0.37	0.01	0.46	0.01	0.40	0.005
TeO ₂	NA	--	NA	--	NA	--
TiO ₂	2.62	0.09	2.60	0.01	2.82	0.02
UO ₂	3.98	0.11	4.16	0.08	4.02	0.08
Y ₂ O ₃	NA	--	NA	--	NA	--
ZnO	4.15	0.15	4.34	0.03	4.46	0.04
ZrO ₂	1.46	0.05	1.88	0.06	1.54	0.09
Total	91.99		96.50		97.64	

(a) Based on analysis of 12 random samples for each batch; standard deviations for an individual analysis.

NA = Not analyzed.

<DL = Below the detection limit indicated.

ATM-1a

ATM-1b

ATM-1c

FIGURE 4.1. Typical Microstructure of Glass ATM-1 (scanning electron micrographs of fracture surfaces; bar at the bottom of photo represents 100 μm for ATM-1a and 10 μm for ATM-1b and 1c)

4.4 X-RAY DIFFRACTION ANALYSIS

Four samples of each glass batch were prepared for standard powder x-ray diffraction (XRD) analysis. As before, two samples were obtained from a single end of two bars, while two more samples were obtained from both ends of one bar. Solid samples were ground and sieved to -200 mesh ($-75\ \mu\text{m}$) and analyzed on a Phillips Model WCDU X-Ray Diffractometer. No crystalline features were observed in ATM-1a, while two samples of ATM-1b showed trace levels of an iron-chrome spinel phase. ATM-1c also showed the spinel phase present in all the samples analyzed. Concentration of the spinel in ATM-1b and ATM-1c were estimated at $\sim 3\text{-}5\ \text{wt}\%$; the detection limit of this phase in a glassy matrix using XRD is estimated to be about $3\ \text{wt}\%$. Table 4.3 is a summary of the XRD observations.

TABLE 4.3. Summary of X-ray Diffraction Analysis of ATM-1

Conditions Used: Instrument - Philips Model WCDU
Radiation - $\text{Cu K}\alpha$
Scan Range - 10 to $60^\circ\ 2\theta$ @ $1/4^\circ/\text{min}$

<u>Glass Type</u>	<u>Observations^(a)</u>
ATM-1a	No crystalline phases evident in 4 samples
ATM-1b	1 sample: spinel peaks (220) and (311); sharp, well defined, weak 1 sample: trace of (311) spinel; broad, poorly defined 2 samples: no crystalline phases
ATM-1c	2 samples: resolvable spinel peaks (220) and (311) 1 sample: spinel peaks appear to be present but not reproducible 1 sample: trace of (311) peak only

(a) All spectra were superimposed on the high background typical of glass x-ray spectra.

5.0 REFERENCES

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

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