PNNL-11098
UC-810
Project Technical Information

Comparison of Simulants to Actual Neutralized Current Acid Waste: Process and Product Testing of Three NCAW Core Samples from Tanks 101-AZ and 102-AZ

E. V. Morrey
J. M. Tingey

April 1996

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

Pacific Northwest National Laboratory
Richland, Washington 99352

Battelle

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

PNNL-11098
UC-810
Project Technical Information

Comparison of Simulants to Actual Neutralized Current Acid Waste: Process and Product Testing of Three NCAW Core Samples from Tanks 101-AZ and 102-AZ

EV Morrey
JM Tingey

April 1996

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

Pacific Northwest National Laboratory
Richland, Washington 99352

Reprint of historical document PVTD-C95-02.03E, dated September 1995. Data, formatting, and other conventions reflect standards at the original date of printing. Technical peer reviews and editorial reviews may not have been performed.

PACIFIC NORTHWEST NATIONAL 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 to DOE and DOE contractors from the
Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831;
prices available from (615) 576-8401.

Available to the public from the National Technical Information Service,
U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161

The document was printed on recycled paper.

Acknowledgements

The process and product testing of the core samples required contributions from a large group of scientists, engineers, and technicians. Garry Richardson was responsible for processing the waste samples through frit addition and drying; he also performed physical and rheological characterization along each step of the process. Ron Holeman, Jim Dunn, and Dewayne Smith were responsible for calcining and vitrifying the waste/frit mixtures. Ron Holeman, Jim Dunn, Dewayne Smith, and Tim Reining performed sample preparation and durability testing of the resulting glasses. Chemical and radiochemical characterizations were provided by Dale Archer, Rebecca Barrington, Simon Barsoum, Jeff Creium, John Ennen, Larry Greenwood, Clark Harvey, Shawn Homi, Vaughn Hoopes, Evan Jenson, Lloyd Kellogg, Anne Kozelisky, Pat Raney, John Rau, Don Rinehart, Danny Sanders, Sally Slate, Monte Smith, Rick Steele, Shannon Thompson, Mike Urie, Jerry Wagner, and Eric Wyse. Assistance with statistical treatment of the data was provided by Greg Piepel and Trish Redgate. Assistance with rheological data reduction was provided by Lynette Jagoda. Technical editing support was provided by Ann Fillion. The authors wish to thank these Pacific Northwest Laboratory staff members and others who contributed to this study.

Summary

A vitrification plant is planned to process the high-level waste (HLW) solids from Hanford Site tanks into canistered glass logs for disposal in a national repository. Programs have been established within the Pacific Northwest Laboratory Vitrification Technology Development (PVTD) Project to test and model simulated waste to support design, feed processibility, operations, permitting, safety, and waste-form qualification. Parallel testing with actual radioactive waste is being performed on a laboratory-scale to confirm the validity of using simulants and glass property models developed from simulants.

Laboratory-scale testing has been completed on three radioactive core samples from tanks 101-AZ and 102-AZ containing neutralized current acid waste (NCAW), which is one of the first waste types to be processed in the high-level waste vitrification plant under a privatization scenario. Properties of the radioactive waste measured during process and product testing were compared to simulant properties and model predictions to confirm the validity of simulant and glass property models work. This report includes results from the three NCAW core samples, comparable results from slurry and glass simulants, and comparisons to glass property model predictions.

Experimental Approach

The three NCAW samples were retrieved from the tanks in cylindrical segments 1 inch in diameter and 19 inches long. Several segments representing a complete vertical sample of the settled solids in the tank were combined and blended to make up a core sample. Solids from each core sample were pretreated using a water wash/settle/decant process, including a ferric-nitrate flocculent additive, settle/decant, and two water washes (3 volumes deionized water to 1 volume solids). The washed solids were then characterized chemically, radiochemically, physically, and rheologically (101-AZ Core 1 only). After adjusting the samples to 125 g waste oxide/L, the waste was treated with formic acid to adjust the feed rheology and to reduce the redox-sensitive species for introduction into the melter. Off-gas analysis during formic acid addition was performed on 102-AZ Core 2 and is described in a separate report.(a) The formated slurry samples were characterized chemically, physically, and rheologically. Frit was added to each of the formated slurries and samples were characterized chemically, radiochemically, physically, and rheologically. The frit/slurry mixtures were dried and melted at 1150°C in crucibles; resulting glass was characterized with respect to chemical and radiochemical composition, durability (Product Consistency Test [PCT] and Materials Characterization Center [MCC-1]), crystallinity, redox, and density.

Two types of simulants were prepared and tested for comparison with the actual waste glass. Process-based slurry simulants were used to develop and test hot-cell procedures and to provide a direct comparison with the core sample feed chemistry and rheology. Major and minor insoluble components were co-precipitated with NaOH from nitrate solutions and washed to remove the sodium

---

(a) Langowski, M.H., E.V. Morrey, J.M. Tingey, and M.R. Beckette. 1993. *Offgas Characterization from the Radioactive NCAW Core Sample (102-AZ-C1) and Simulant During HWVP Feed Preparation Testing.* Letter Report for U.S. Department of Energy. Pacific Northwest Laboratory. Richland, Washington.

and nitrate. Soluble minor components were added separately, following washing. Glass simulants, similar to those used to develop glass property models, were prepared to provide a direct comparison with the actual waste-glass product quality. Three simulant glasses were prepared to match the chemical composition of the three actual waste glasses by batching and melting appropriate amounts of dry chemicals. The simulants were characterized the same as the actual waste samples, excluding radiochemical, to provide a direct comparison of simulant and actual waste.

Process and product behaviors of the actual waste were compared to simulant behavior, glass property models, slurry property correlations, and simulant behavior from other studies and larger scale tests. Statistical comparisons of simulant waste-glass durabilities and model predictions to actual waste-glass durabilities were made based on 95% confidence t-tests. Other comparisons are primarily nonstatistical.

Results and Conclusions

Slurry Chemical Characterization. The chemical composition of the three NCAW core samples and simulants were similar. The major components in all three samples are Fe, Al, and Na as OH^-, CO_3^{2-}, NO_3^-, and NO_2^-. The pH of the washed solids were approximately 12.7 for the core samples from tank 101-AZ, 10.2 for tank 102-AZ, and 10.4 for the simulants. High washing efficiencies of the major cations as measured by the percentage of the analyte remaining in the washed solids slurries compared to the prewashed solids were only observed for Na (30%) and Cr (60%), but significant quantities of Al, Cr, K, Na, F^-, Cl^-, NO_3^-, NO_2^-, and SO_4^{2-} were removed from the sludge in the washing steps. Phosphate is the only measured anion in which a significant percentage remained in the sample. Comparison of the concentration of Na in the washed solids, the sludge prior to washing, and the reference nominal value for the previously planned HWVP indicates that acceptable washing efficiencies are being achieved on the laboratory-scale processes. A comparison of chemical composition of simulant 102-AZ Core 1 and the corresponding core sample indicates that accurate chemical simulants can be prepared.

During the formating process CO_3^{2-}, NO_3^-, and NO_2^- react to produce gas, and the concentrations of these anions in the sample decrease. Slurry chemistry and offgas generation reactions are similar between the core sample and simulants. Observed offgas differences between simulant and core sample could be explained by differences in testing conditions and slurry chemical composition.

Formated slurry was combined with frit to achieve melter feeds with waste oxide loadings of 25 to 28 percent; therefore, the major constituents in the melter feeds are the frit components. These major components include Si, Na, B, and Li. The frit components were added as the oxides; therefore, the majority of the elements in the melter feeds are as oxides. Other anions which are present in significant quantities are NO_3^-, NO_2^-, Cl^-, F^-, and SO_4^{2-}. The supernatant from the melter feeds contained only three cations in significant quantities (Na, K, and Li). The major anions associated with the supernatant are similar to those observed in the slurries with the exception of the oxides which are not soluble.

Slurry Radiochemical Characterization. Handling and disposal of chemical simulants is much more cost effective than radiochemical simulants; therefore, no radiochemical simulants were prepared in these studies. The major radionuclides present in the core samples are ^{137}Cs, ^{90}Sr, ^{144}Ce, and ^{106}Ru. All of the slurry samples are transuranic (contain > 100 nCi/g transuranic isotopes). The majority of the transuranic activity is due to americium and plutonium. None of the supernatant or wash solutions were transuranic, and ^{137}Cs is the primary radionuclide in the supernatant. The only radionuclides affected by the washing steps were ^{137}Cs and ^{125}Sb. The only radionuclide which may have been affected by the formating steps was ^{129}I. The data is not definitive, but it appears that some of the iodine may have been lost during the formating process. Measured radionuclide activities were within the previous HWVP specifications with the exception of ^{129}I, ^{90}Sr, and ^{60}Co.

Slurry Physical Characterization. The density of the washed solids from the core samples and simulants ranged from 1.04 to 1.14 g/ml. The density of the formated slurries was similar to that of the washed solids. As expected the density of the samples increased with increasing solids concentrations. A correlation between the density of the formated slurries and the solids concentration is observed, and simulants are representative of actual waste with respect to this correlation. The density of the melter feeds (1.28 to 1.47 g/ml) increases significantly from the formated slurry and washed solids density. This trend is also observed for the centrifuged solids density. The simulants have a significantly lower centrifuged solids density than is observed in the core samples. The density of the centrifuged liquid (1.02 ± 0.03) was similar for all of the slurries and is comparable to the density of water.

The majority of the settling of the washed-solids and formated slurries occurs in the initial 10 hours of settling. The rate of settling increases with each ensuing wash. The settling behavior of the simulants did not match the behavior of the core samples. This is also observed in the volume percent settled solids. The core samples settled much faster and achieved a significantly higher settled solids packing than was observed in the simulants. The settling behavior of the melter feed from NCAW simulant and the 101-AZ core samples are comparable, but the settling behavior of the 102-AZ Core 1 melter feed and the corresponding simulant did not match. The 102-AZ Core 1 simulant did not settle. Previous studies indicate that the frit blended with this simulant tends to gel. This is consistent with the behavior observed for this simulant. The 102-AZ Core 1 melter feed settled much quicker than was observed in the other core samples and simulants. The washed solids from this core dried prior to processing the sample. Previous results indicate that when some tank waste samples are allowed evaporate to dryness, the settling and rheological behavior of the sample are irreversibly altered.

The mean particle diameters of the washed solids, formated slurries, and melter feeds based on volume distribution and population distribution are similar for the three core samples. The particle size of the solids in the core samples did not change significantly as the samples were processed. The majority of the particles were less than 5 microns in diameter with a significant number of the particles less than 1 micron in diameter. The analysis of 101-AZ Core 1 was performed with water as a diluent, and a large portion of the solids may have gone into solution; therefore, the most representative sample for actual particle size in tank 101-AZ is Core 2.

Slurry Rheological Characterization. Actual formated waste slurries exhibited lower yield stresses and apparent viscosities than simulant-formated waste slurries, which was attributed to differences in microstructure. Rheological behavior of the radioactive and simulant-formatted slurries was best represented as yield pseudoplastic with slight shear-thinning and hysteresis. Yield stresses of the radioactive formated slurry ranged from 0.085 Pa to 0.23 Pa, compared to simulant-formated slurry yield stresses of 1.2 Pa. Apparent viscosities of the actual waste samples at $50s^{-1}$ ranged from 3.8 cP to 7.2 cP compared to simulant viscosities of 32 cP. The rheology of both the formated waste samples and simulants were well below the design limit for the prior-planned Hanford Waste and nitrate. Soluble minor components were added separately, following washing. Glass simulants,

Vitrification Plant (HWVP). After accounting for differences in concentration, the simulated formated waste exhibited yield stresses and apparent viscosities roughly two times greater than those for actual formated waste. Actual formated waste samples exhibited greater initial settling rates, greater degrees of settling, and denser centrifuged solids than simulant formated samples, indicating a difference in microstructure. A comparison with rheological data from full-scale formated simulant tests showed essentially identical results with laboratory-scale formated simulant data from this study. A comparison with historical NCAW formated simulant data dating back to 1985 showed actual formated waste results to be equal to or lower than the weakest (i.e., lowest shear stress and apparent viscosity for given concentrations) simulants reported.

For melter feed samples, actual waste exhibited lower yield stresses and apparent viscosities than simulated waste, which again is attributed to differences in microstructure. Rheological behavior at the radioactive and simulant melter feed slurries was best represented as thixotropic, yield pseudoplastic with varying degrees of hysteresis. Yield stresses of the radioactive melter feed ranged from 1.4 Pa to 10.3 Pa compared to simulant melter feed yield stresses of 2.2 Pa and 12.4 Pa. Apparent viscosities of the actual waste samples at $50s^{-1}$ ranged from 38 cP to 260 cP compared to simulant viscosities of 58 cP and 365 cP.

Glass Characterization. Initial chemical characterizations of the glass were inadequate, using the standard KOH/Na_2O_2 preparation methods for inductively coupled argon plasma/atomic emission spectrometry (ICP/AES) analysis. Analysis of the first two radioactive glasses accounted for only 91%-93% of the mass of the glass. Additional analysis using an HF preparation and comparable standard glasses were required to arrive at a reasonable glass composition. Subsequent procedural improvements to the KOH/Na_2O_2 preparation methods resulted in satisfactory results for the third core-sample glass. For all three radioactive glasses, the measured major analytes were generally within 10% of calculated values, which were determined from washed solids composition, frit compositions, and assumed waste loadings. Achieved waste loadings were slightly greater than targeted (i.e., 2% to 5%), because of accuracy limitations on slurry sampling or total oxides analysis.

Glass redox for the actual waste glasses as measured by Fe^{+2}/Fe^{+3} ratio ranged between 0.026 to 0.085 compared to a simulant redox of 0.005. Each of the glasses measured was within the design limit for the prior planned HWVP plant. Glass redox of the actual waste compared well with historical simulant data correlating glass redox to formic acid added and initial nitrite and nitrate compositions.

Radioactive glass samples were analyzed by X-ray diffraction to determine the extent and composition of crystalline phases. As predicted by models and simulant experience, no substantial crystallinity was found (i.e., likely under 1%).

Density of the three radioactive glasses was measured to be 2.56 g/cc for 101-AZ Core 1, 2.67 g/cc for 101-AZ Core 2, and 2.54 g/cc for 102-AZ Core 1 at room temperature, values typical of simulant glass densities.

Glass Durability. Each of the three radioactive and simulant glass formulations produced highly durable glasses in all cases at least 20 times more durable than the Savannah River Environmental Assessment (EA) glass as measured by the PCT. Seven-day PCT boron releases for the radioactive glasses ranged from 0.13 to 0.21 g/m^2 compared to simulant boron releases of 0.20 to 0.34 g/m^2. The magnitude of the increase from radioactive to simulant releases ranged from 28 to

67%. In each case the differences in boron release were found to be statistically significant to a 95% confidence. Model predictions for each of the three glass formulations were greater than both actual and simulant waste glass releases. Over the limited amount of tests performed, the actual and simulant waste glass releases fell within the 95% prediction interval for the model 56% and 89% of the time, respectively. Twenty-eight-day MCC-1 results for actual and simulant glasses showed similar results, however the differences were greater. An indeterminate portion of the difference was attributed to differences in leach containers used for these tests.

Radiation dose has been shown to have a significant effect on glass corrosion in aqueous leach tests; however, the prediction and explanation of the radiolytic effects are complex. The durability differences between actual and simulated waste glasses reported in this study were equal to or lower than differences observed by others, which was consistent with removing part of the radiolytic effect (i.e., tests performed in Ar atmosphere). To the extent Ar backfilling of the leach containers was effective, the effect of radiolytic generation of nitric acid was eliminated. Based on the type of test performed and the relative durability of the glasses in this study, the dominant corrosion mechanism is expected to be network hydrolysis, which is favored under higher pHs. Had the leach containers contained air, one would have predicted the radiolytic effect to be decreased pH and glass corrosion. With the absence of air in the system, it was not clear whether radiolytic affects should increase or decrease corrosion.

Seven-day PCT and 28-day MCC-1 radionuclide releases were measured, calculated, and compared to results from prior studies. As with prior studies, Am had normalized releases significantly lower than B, ranging from 0.1% to 6% of B. Also consistent with prior studies, Tc, U, Np, and Cs were generally more soluble than Am (i.e., ≥ 10% of B-normalized release). Not consistent with prior studies, Pu exhibited significant normalized releases near B. Strontium was relatively soluble in MCC-1 tests and insoluble in PCT.

Contents

Figures

Appendix Figures

Tables

Appendix Tables

Abbreviations

CAW	Current Acid Waste
CI	Confidence Interval
CVS	Composition Variability Study

PHTD	PNL HWVP Technology Development
PNL	Pacific Northwest Laboratory
PUREX	Plutonium and Uranium Extraction
PVTD	PNL Vitrification Technology Development
QA	Quality Assurance
RSD	Relative Standard Deviation
TC	Total Carbon
TDP	Technical Data Package
TEM	Transmission Electron Microscopy
TIC	Total Inorganic Carbon
TOC	Total Organic Carbon
TRU	Transuranic
WAPS	Waste Acceptance Preliminary Specifications
WQR	Waste Form Qualification Report
WS	Washed Solids
XRD	X-Ray Diffraction

1.0 Introduction

A High-Level Waste (HLW) Vitrification Plant is being planned to process Hanford high-level and transuranic (TRU) tank waste into canistered glass logs for disposal in a national repository. The Pacific Northwest Laboratory (PNL)[a] Vitrification Technology Development (PVTD) testing programs have been established to develop and verify process technology using simulated waste. A parallel testing program with radioactive waste is being performed to confirm the validity of using simulants and glass property models for waste form qualification and process testing.

The type of HLW used in this study is pretreated neutralized current acid waste (NCAW). The NCAW is a neutralized HLW stream generated from the reprocessing of irradiated nuclear fuel in the Plutonium and Uranium Extraction (PUREX) Plant at Hanford. As part of the fuel reprocessing, the high-level waste generated in PUREX was denitrated with sugar to form current acid waste (CAW). Sodium hydroxide and sodium nitrite were added to the CAW to minimize corrosion in the tanks, thus yielding neutralized CAW. The NCAW contains small amounts of plutonium, fission products from the irradiated fuel, stainless steel corrosion products, and iron and sulfate from the ferrous sulfamate reductant used in the PUREX process.

The total inventory of NCAW is contained in two one-million-gallon double-shell tanks (DSTs). Three core samples from the two tanks have been characterized, pretreated, vitrified, and leach-tested. Properties of the radioactive waste measured during laboratory process and product testing have been compared to simulant properties and model predictions to confirm the validity of simulants and glass property models.

1.1 Objectives

The radioactive process/product laboratory testing provides confirmation of the adequacy of nonradioactive feed simulants used in laboratory and pilot scale testing to support feed processability assessments, vitrification process development, and glass property model development. Small-scale process/product testing is being conducted in the hot cell using limited quantities (100-200ml at 125 g of equivalent oxides/L) of actual tank waste materials derived from core samples and compare results to those obtained from similar testing using simulants.

1.2 Background

Radioactive waste samples and waste simulants are being tested to support the design, operation, and permitting of the HLW Vitrification Plant. The relationships between these test programs and the plant design efforts are described in this section.

The laboratory-scale radioactive testing directly supports two of PNL's simulant-based, laboratory-scale testing programs. These programs, in turn, support the bench- and pilot-scale PNL test programs by determining appropriate simulant compositions and test conditions. The testing

---

(a) Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

programs combine to support various aspects of the HLW Vitrification Plant design and, to some extent, the permitting requirements.

The flow of information between the various activities of the HLW vitrification project is shown schematically in Figure 1.1. The nonradioactive laboratory-scale through pilot-scale testing relies on waste simulants. The data from these tests are used in conjunction with waste characterization information to develop the plant design and information flowsheet. Thus, it is essential that the simulant's chemical and physical behavior be representative of the actual radioactive waste.

The simulant development efforts form the foundation of the HLW vitrification design/verification testing. Simulant development is, in turn, supported directly by a combination of process knowledge, characterization, estimates of feed compositions, and core sample testing. Testing radioactive core samples provides the most defensible and direct link between the tank waste and the waste simulants used for testing. Without radioactive sample testing, the use of simulants are not sufficiently defensible for process/equipment/product testing.

The simulant-based, laboratory-scale testing focuses on two different aspects of the HLW Vitrification Plant flowsheet. One focus area is the chemical and rheological characteristics of the plant feed as it is processed through the feed treatment portion of the flowsheet. This includes feed concentration and the addition of reductant and glass formers. The second focus area is the processability and product quality of the vitrification portion of the flowsheet. The chemical and physical properties of the molten waste glass as well as the cooled glass product are of primary interest. Both focus areas are supported through radioactive sample testing.

The first of the two laboratory-scale test programs supported by radioactive core sample testing is the feed chemistry/rheology evaluation. This work determines the effects of changes in feed composition and treatment on the physical, chemical, and rheological properties of the feed, using nonradioactive chemical simulants. These chemical simulants are developed based on the processes used to generate the waste originally and on the currently planned waste pretreatment processes (e.g., water wash, caustic leach, filtration, etc.). These process-based simulants contain the bulk of the chemical species expected to be present in the actual waste with the exception of trace-quantity species and radioactive components.

The process-based simulants used by the Feed Chemistry/Rheology Evaluation task are subjected to laboratory-scale tests designed to simulate various operating scenarios in the HLW Vitrification Plant feed treatment process. Variations in chemical additions, temperatures, and other operating parameters are applied to the simulants to determine the resulting off-gas generation, condensate composition, slurry chemistry, slurry physical properties, cold cap reactions, glass redox,

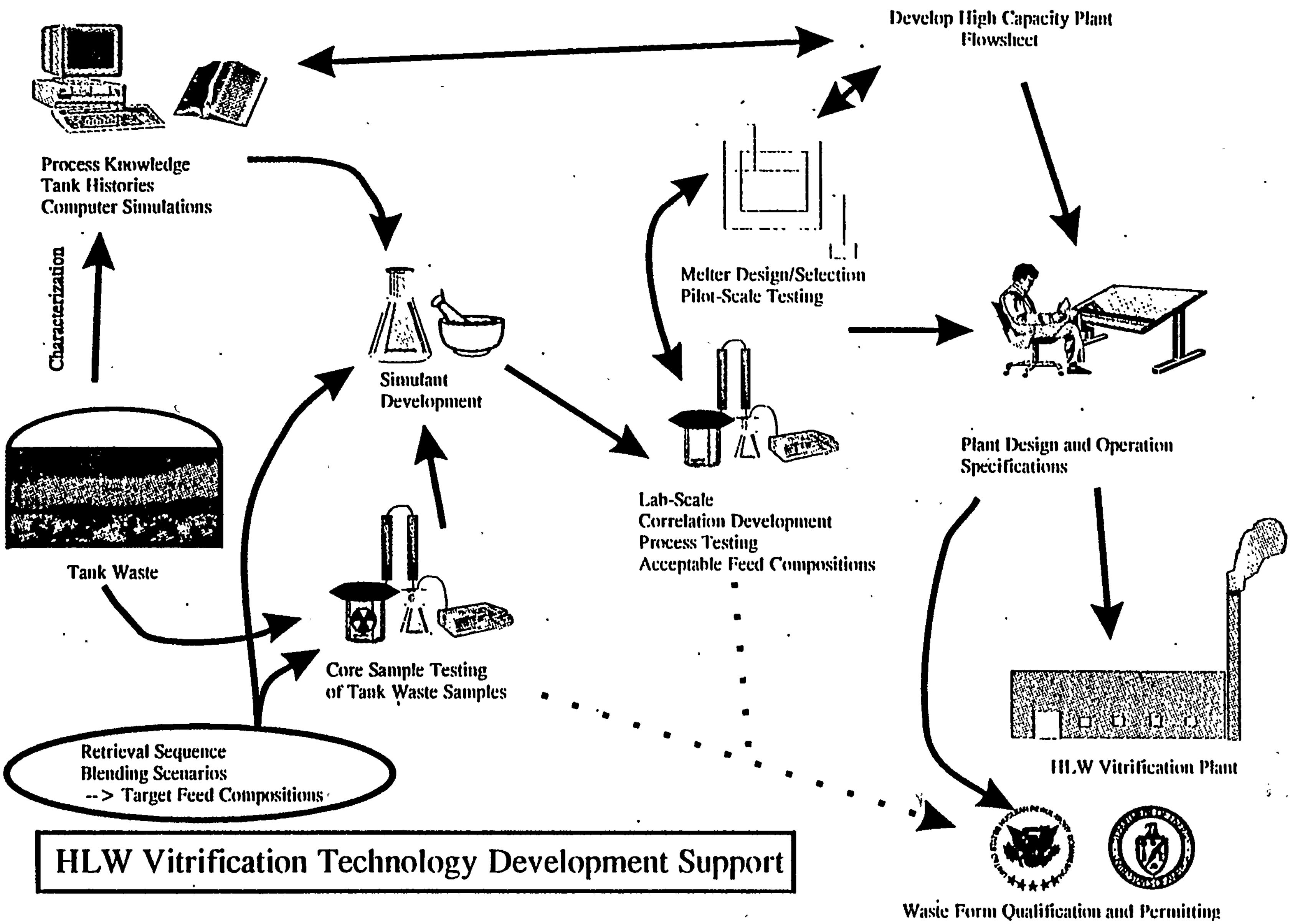


Figure 1.1. HLW Vitrification Technology Development Support

and potentially uncontrollable exothermic reactions. These data are coupled with data from larger-scale tests to support the HLW Vitrification Plant design efforts. For example, the off-gas generation data are used to size off-gas components and determine what gas treatment methods will be needed to maintain plant gas effluent within regulatory limits. The rheological characteristics of the slurry are used to assess agitation and transfer requirements and to determine if there is the potential for gel formation, which could severely impact plant operation.

The second laboratory-scale task that uses simulants is glass formulation development. This task determines the effect of glass composition on the physical properties of the molten glass and the properties that measure the suitability of the glass product for storage in a repository. Glass melt properties like viscosity, electrical conductivity, and component solubilities all have a direct bearing on the design and operation of the HLW Vitrification Plant melter. Cooled glass product properties like durability and crystallinity directly impact the acceptability of the glass for disposal in a repository.

The U.S. Department of Energy Waste Acceptance Preliminary Specifications (WAPS) governs the characterization, control, and documentation of high-level nuclear waste glass produced by a vitrification plant. These specifications are generally accepted as representative of the requirements that will be established formally for all waste glass producers, including the Hanford HLW Vitrification Plant. WAPS Specification 1.3 requires that glass be at least as durable as the Defense Waste Processing Facility (DWPF) Environmental Assessment (EA) glass as determined by the 7-Day Product Consistency Test (PCT). WAPS Specification 1.3 allows that the test result may be predicted rather than measured from actual production glass if an acceptable means for this is provided and defended. The WAPS specification is subject to change.

As part of glass formulation development, an empirical relationship is being determined between the composition and durability of simulated high-level nuclear waste glasses. The relationship has been determined as part of the "Composition Variability Study" (CVS) (Hrma 1994). Glasses of various compositions have been and are being prepared for this statistically designed study, and their properties measured. The CVS study includes two static leach tests for each of the glasses in the test matrix: the Materials Characterization Center (MCC-1) 28-day test and the 7-Day PCT. The leach test results were used to fit empirical models.

The empirical models will be validated as part of the CVS, using results from simulated glasses. To validate the model for application to radioactive glass, radioactive waste data and comparisons are necessary. Although the radioactive durability data generated in this study may add to the understanding of radioactive glass reactions in aqueous solutions and eventual performance in a repository, the key objective is to validate glass property models used to ensure conformance to repository waste acceptance criteria. Results from the short-term, static leach test defining waste acceptability (i.e., PCT) cannot currently be related to long-term performance in the repository.

Unlike the feed chemistry/rheology evaluation simulants, the simulants used by the Glass Formulation Development task are not process-based. Instead, the postulated key chemical components are mixed together in the required quantities, usually as dried powders. It is implicitly assumed that the glass properties are relatively insensitive to the initial simulant composition and particle morphology, provided that the simulant contains the proper elemental composition with the elements in their expected valence states. In other words, regardless of the chemical species fed to the melter, the elements will be converted quickly to their respective oxides by the high melt temperature. Process-based simulants are not used for this task because of the large number of different simulants required and the fact that the current, less-expensive approach is thought to be acceptable.

The laboratory-scale testing of radioactive samples supports both the glass formulation development and the feed chemistry/rheology evaluation. The radioactive testing provides a means for checking the results of simulant tests against the behavior of actual waste. Without this verification, there is no assurance that the process and product, using actual waste, will behave the same. With this verification, the simulant-based approach allows for the most cost-effective plant design to be developed with a relatively low risk of failure. It is important to realize that for each estimated feed composition, two types of simulants need to be verified.. Both the process-based simulants and the dry powder simulants must be compared to actual waste.

1.3 Report Contents

This report includes process/product testing results from the first and second radioactive core samples from Tank 241-AZ-101 and the first radioactive core sample from Tank 241-AZ-102. Data are provided for all steps of the process starting with washed solids characterization through characterization of the resulting glass.

Off-gas data were collected during formating, digestion, and recycle addition of the first core sample from Tank 214-AZ-102 and simulants. This data is not contained in this report but is fully documented.[a]

(a) Langowski, M.H., E.V. Morrey, J.M. Tingey, and M.R. Beckette. 1993. *Offgas Characterization from the Radioactive NCAW Core Sample (102-AZ-C1) and Simulant During HWVP Feed Preparation Testing*. Letter Report for U.S. Department of Energy. Pacific Northwest Laboratory. Richland, Washington.

2.0 Conclusions and Recommendations

2.1 Conclusions

2.1.1 Process-Related

Analysis of the pretreated waste showed that chemical and radiochemical compositions are well within the design range established for the previously planned HWVP. A comparison of chemical composition of simulant 102-AZ Core 1 and the corresponding core sample indicates that accurate chemical simulants can be prepared.

Comparison of the concentration of Na in the washed solids, the sludge prior to washing, and the reference nominal value for the previously planned HWVP indicates that acceptable washing efficiencies are being achieved on the laboratory-scale processes. During the formating process CO_3^{2-}, NO_3^-, and NO_2^- react to produce gas, and the concentrations of these anions in the sample decrease. Slurry chemistry and offgas generation reactions are similar between the core sample and simulants. Observed offgas differences between simulant and core sample with the exception of H_2 could be explained by differences in testing conditions and slurry chemical composition. Peak and total H_2 generation in 102-AZ Core 1 was approximately one-third that generated by its simulant.

Specific activities indicate that the processed solids will be transuranic, and all of the liquid streams will be non-transuranic. The liquid streams will contain significant quantities of ^{137}Cs unless advanced processing of these liquid waste streams is incorporated. An offgas system for the formating process will be needed to trap the ^{129}I which may be lost during the formating process. Offgas systems will also be needed during the melter process to trap volatilized ^{99}Tc and ^{137}Cs. These radionuclides exhibited volatility in the laboratory-scale tests, but tests at this scale are not expected to be representative of full-scale systems.

The physical properties of the washed-solids slurries vary significantly, but these differences are based upon variances in the solids concentrations. With few exceptions, when the slurry concentrations fell within the previous HWVP design range, the other physical and rheological properties were also within that same design range. A correlation between the density of the formated slurries and the solids concentration exists, and simulants are representative of actual waste with respect to this correlation. The core samples settled much faster and achieved a significantly higher settled solids packing than was observed in the simulants. Decreased solids packing in the simulants compared to the core samples is also observed in the centrifuged solids data.

Rheologic properties of the actual and simulant formated slurries were well below the design limit for the prior-planned HWVP plant, and were measureably different from each other. After accounting for differences in concentration, the simulant formated waste exhibited yield stresses and apparent viscosities roughly two times greater than those for actual formated waste. Actual formated waste samples exhibited greater initial settling rates, greater degrees of settling, and denser centrifuged solids than simulant samples, indicating a difference in microstructure. A comparison with rheological data from full-scale simulant tests showed essentially identical results with laboratory-scale simulant data from this study. A comparison with historical NCAW formated simulant data dating back to 1985 showed actual formated waste results to be equal to or lower than the weakest (i.e., lowest yield stresses and apparent viscosities) simulants reported.

Rheologic properties of the actual and simulant melter feed were below the design limit for the prior planned HWVP plant, but were measureably different from each other. After accounting for differences in concentration, the simulated waste melter feed exhibited yield stresses and apparent viscosities roughly 1.5 to 2 times greater than those for actual waste. Actual melter feed exhibited greater initial settling rates, equal or greater degrees of settling, and denser centrifuged solids than simulant melter feed, indicating a difference in microstructure. A comparison with historical melter feed simulant data showed the actual melter feed rheology to be be within the range measured for simulant melter feed.

Glass redox measurements (Fe^{+2}/Fe^{+3}) on the actual waste glasses were within the design limit for the prior planned HWVP plant and compared well with historical simulant data.

2.1.2 Product-Related

Three NCAW core samples were characterized, prepared as melter feed, and vitrified into a glass product with acceptable properties relative to waste disposal. Durabilities of actual waste glasses as measured by PCT were between 20 and 100 times greater than the durability of environmental assessment (EA) glass. Crystallinity in each of the three actual waste glasses were determined to be less than 1%.

Actual waste loadings in the radioactive glass were between 2% and 5% greater than targeted values because of inaccuracies in total oxide measurements. Actual waste loadings were estimated to be between 27% and 30%.

Radionuclide releases from the actual waste glasses were generally consistent with results from other actual waste glass studies. As with prior studies, Am-normalized releases were significantly lower than B (0.1% to 6% of B); Tc, U, Np, and Cs were generally more soluble than Am (≥ 10% of B normalized release). Not consistent with prior studies, Pu exhibited normalized releases near B.

The durability of actual glass as measured by 7-day PCT and 28-day MCC-1 was found to be slightly greater than simulant glasses of the same composition and model predictions. The magnitude of the increase from radioactive to simulant PCT boron releases ranged from 28 to 67% of the lesser value and was statistically significant. Actual and simulant waste glass releases were less than model predictions, and fell within the 95% prediction interval for the model 56% and 89% of the time, respectively.

Biases in MCC-1 leach tests in the hot cell were attributed to temperature variations, temperature inaccuracies, and differences in leach containers. Small temperature variations in the MCC-1 test appear to affect dissolution rate of the glass significantly. A comparison between fused-silica and Teflon™ containers in a MCC-1 28-day test showed significant differences. The amount of silica released from the fused-silica liner is significant when compared to Si released from the glass. Comparisons of results between PCT 7-day leach tests conducted in fused-silica, Teflon, and stainless-steel containers showed little or no differences between containers. The amount of silica released from the fused-silica liner during a PCT test was insignificant when compared to Si released from the glass.

2.2 Recommendations

- Additional testing of actual waste representing different waste types should be performed to expand the envelope of validated simulant compositions and to broaden the properties being validated. Additional measurements should be made on the glass product to validate simulant behavior with respect to critical process properties (i.e., off-gas speciation during melting, glass liquidus temperature, glass viscosity, radioisotope volatility, and fissile material distribution) and product properties (i.e., enhanced crystallinity measurement with scanning electron microscope [SEM], gel layer and secondary phase characterization of radioactive and simulant leached glasses by transmission electron microscope [TEM] to verify similarity of reactions).
- Measure particle morphology in future slurry samples by TEM to increase understanding of physical and rheological differences between actual and simulated waste.
- Perform simple irradiation tests on NCAW simulant to determine radiation effects on rheology.
- Perform irradiated leach tests on archived simulant glasses from this study to determine if radiation effects are the cause of the increased durability of radioactive glasses.
- Develop subsampling systems to obtain representative samples from the melter feeds.
- Perform detailed study of the radionuclide versus surrogate volatility during formating and melting processes. The radionuclides of greatest interest are ^{129}I, ^{137}Cs, and ^{99}Tc.

3.0 Experimental Method

3.1 Test Objectives

The overall objective of the tests was to confirm that nonradioactive feed simulants and resulting glasses are adequate representations of actual radioactive feeds and glasses to support feed processibility assessments and glass property model development. Specific objectives of the tests were as follows:

- Prepare and process test nonradioactive simulant slurries for comparison of chemical, rheological, physical, and off-gas behavior. Simulant slurries were also used for development and verification of test procedures.
- Perform feed concentration, formic acid addition, and recycle addition on the radioactive core samples and simulants and complete characterization of the formated slurries. Compare properties of the radioactive and simulant samples to each other and to results from other studies.
- Formulate, prepare, and add frit to the radioactive core samples and simulants and complete melter feed characterization.(a) Compare properties of the radioactive and simulant melter feed samples to each other and to results from other studies.
- Perform vitrification of the radioactive and simulant melter feeds and complete characterization of the glass. Glass composition data is needed for preliminary supporting information for the Waste Form Qualification Report (WQR), possible input to the Feed Processability Assessment, and CVS models and normalized release calculations.
- Perform MCC-1 and PCT leach tests on radioactive and simulant glass for validation of the CVS models.
- Theorize observed differences between the simulants, models, and radioactive data.
- Provide a qualitative assessment of the adequacy of analytical techniques for process and glass characterization.

(a) This laboratory-scale test uses crucibles instead of a melter system for vitrification. The formated slurry/frit mixture is referred to as "melter feed" throughout this report to be consistent with larger-scale terminology.

3.2 Method/Approach

Test plans were developed that detail the processing of the small-volume NCAW samples through retrieval, pretreatment, and vitrification process steps.(a),(b) Physical, rheological, chemical, and radiochemical properties were measured throughout the process steps. Appropriate pretreatment and vitrification process steps were developed based on a wide range of nonradioactive simulant tests. The processing and characterization of NCAW simulants and actual tank samples are used to evaluate the operation of these processes. Figure 3.1 shows the flowsheet for the sampling, processing, and testing performed on the 101-AZ Core 1 NCAW sample. Characterization flowsheets for the subsequent two core samples are essentially the same, with minor modifications. The HLW vitrification flowsheet has not been finalized and has changed since these tests were performed. Current flowsheet plans include options for additional pretreatment steps, alternate reductant acid, and increased waste loading.

3.2.1 Simulant Preparation

Process-based waste simulants were used in the laboratory-scale radioactive testing to develop and test hot cell procedures and to provide a direct comparison with the core sample feed chemistry and rheology. The slurry simulants were prepared using PNL procedure WTC-006-36, "Procedure for Preparation of Simulated HWVP Feed for Laboratory-Scale Redox/Rheology and Radioactive Process/Product Tests," which simulates the waste-processing history. Major components, including Al, Fe, Mn, Ni, and Zr, were precipitated with NaOH from nitrate solutions and washed to remove the sodium and nitrate. The insoluble minor components (Ag, Cd, Ce, Cr, La, Li, Mg, Nd, Pb, Pd, Rh, Ru, Si, Te, Ti, and Zn) were co-precipitated, washed, and blended with the major components. Soluble and slightly soluble minor components (B, Ba, Ca, Cs, Cu, Na, and Sr) were added directly as oxides, hydroxides, fluorides or sulfates. Sodium was added in multiple forms to match the carbonate and anion (F, Cl, NO_2, NO_3, PO_4, OH and SO_4) concentrations. The simulants were tested on the same apparatus and in the same manner as were the radioactive samples.

Glass simulants were prepared using PNL procedure PSL-417-GBM, "Procedure for Glass Batching and Melting, Rev. 0," to provide a direct comparison with the radioactive glass product quality and characteristics. Simulant glasses were prepared to match the chemical composition of each core sample glass by batching and melting appropriate amounts of dry chemicals (e.g., Fe_2O_3, Na_2CO_3, ZrO_2, Al_2O_3, SiO_2, $CaCO_3$, MgO, H_3BO_3, Li_2CO_3, KCl). Each of the glasses were melted at 1150°C for 1 hour in a platinum crucible, cooled, crushed in a tungsten carbide disc mill, remelted for two hours at 1150°C for better homogeneity, poured into bar forms, and annealed for 2 hours at 500°C.

(a) Larson, D.E., May 1989. Laboratory Vitrification of Radioactive Pretreated Neutralized Current Acid Waste Test Plan, Report No. HWVP-89-1VJ0030402B, Rev. 0.

(b) Test Plan Laboratory-Scale Testing of the First Core Sample from Tank 102-AZ, Report No. PHTD-C92-05.05A, March 1992.

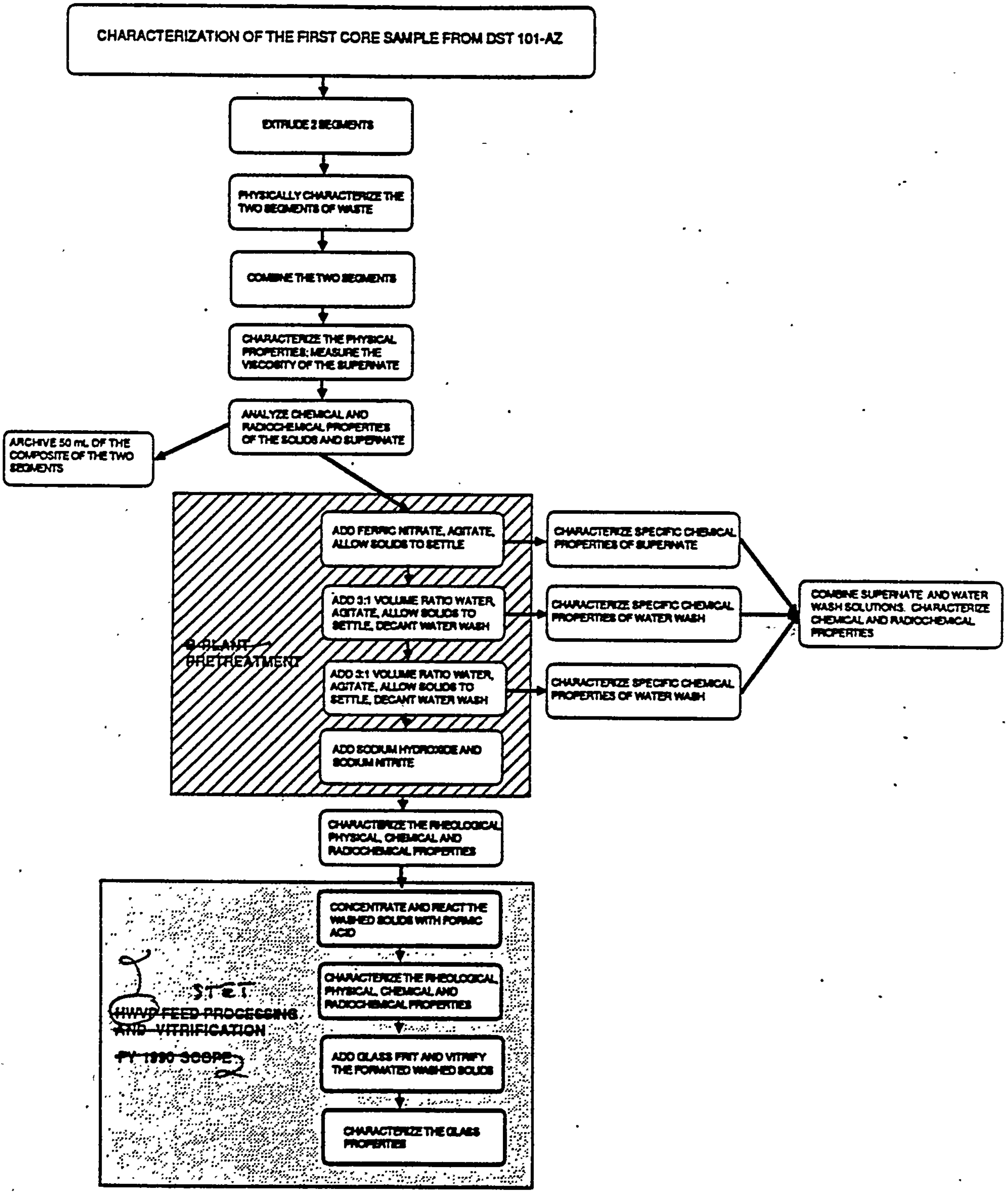


Figure 3.1. Characterization Flowsheet for 101-AZ Core 1

3.2.2 Sampling and Pretreatment

NCAW samples were retrieved from the double-shell tanks 241-AZ-101 (two core samples) and 241-AZ-102 (one core sample), pretreated, and processed according to the flowsheet shown in Figure 3.1. A specially designed and equipped core-sampling truck was used to retrieve samples of NCAW from the Hanford underground storage tanks. Cylindrical segments of the waste, 1 in. in diameter and 19 in. in length, were collected in stainless steel core samplers. The samplers were designed to maintain the waste stratification present in the tanks.

Sludge depth measurements were taken before the tanks were sampled; the average sludge depth for tanks 101-AZ and 102-AZ before sampling were 17 and 33 in., respectively. The total waste depth in these tanks were 357 and 347 in. for tanks 101-AZ and 102-AZ, respectively; therefore, approximately 26 ft. of supernatant lies above the samples taken from these tanks. In the sampling process, waste from 38 in. to 19 in. above the tank bottom (segment 1) and 0 in. to 19 in. above the tank bottom (segment 2) were expected to be obtained; thus, both solids and supernatant would be obtained from these samples. It was expected that sufficient supernatant would be obtained from these samples to characterize the entire supernatant layer in the tanks and perform the process testing needed for the vitrification processes; therefore, the entire depth of waste in the tanks was not sampled.

Each of the three core samples consisted of two 19-in.-long segments (245 ml) of waste. The samplers were transferred into the shielded facility (hot cell) where the samples were removed from the core samplers. Visual observations of the samples were recorded, and the samples were subsampled for chemical, radiochemical, and physical characterization. Core segments were homogenized and then composited to form a representative sample for use in process testing. The results of the characterization of these core samples and the detailed flow process of these samples are reported in the tank waste characterization reports for these cores (Peterson 1989).[a],[b]

The NCAW pretreatment process consisted of adding ferric nitrate to the sample (1.0 M ferric nitrate was added to achieve 0.025 moles of ferric nitrate per liter of sample), stirring the sample for 30 minutes, and allowing the sample to settle for 62 hours. Based on the prior reference flowsheet, the waste entering the pretreatment facility was to contain 20 vol% settled solids, which formed the basis for establishing the laboratory pretreatment processing steps. The base pretreatment process included decanting the supernatant to achieve a target composition of 50 vol% settled solids. Only a limited amount of supernatant was obtained from the three NCAW core samples; therefore, only enough supernatant necessary to characterize the sample was decanted from the settled sample. The remaining slurry was then washed with 3 volumes of deionized water per

---

(a) Gray, W.J., M.E. Peterson, R.D. Scheele, J.M. Tingey. 1991. "Characterization of the Second Core Sample of Neutralized Current Acid Waste from Double-Shell Tank 101-AZ." Letter report for U.S. Department of Energy. Pacific Northwest Laboratory. Richland, Washington.

(b) Gray, W.J., M.E. Peterson, R.D. Scheele, and J.M. Tingey. 1990. "Characterization of the First Core Sample of Neutralized Acid Waste from Double-Shell Tank 102-AZ." Letter report for U.S. Department of Energy. Pacific Northwest Laboratory. Richland, Washington.

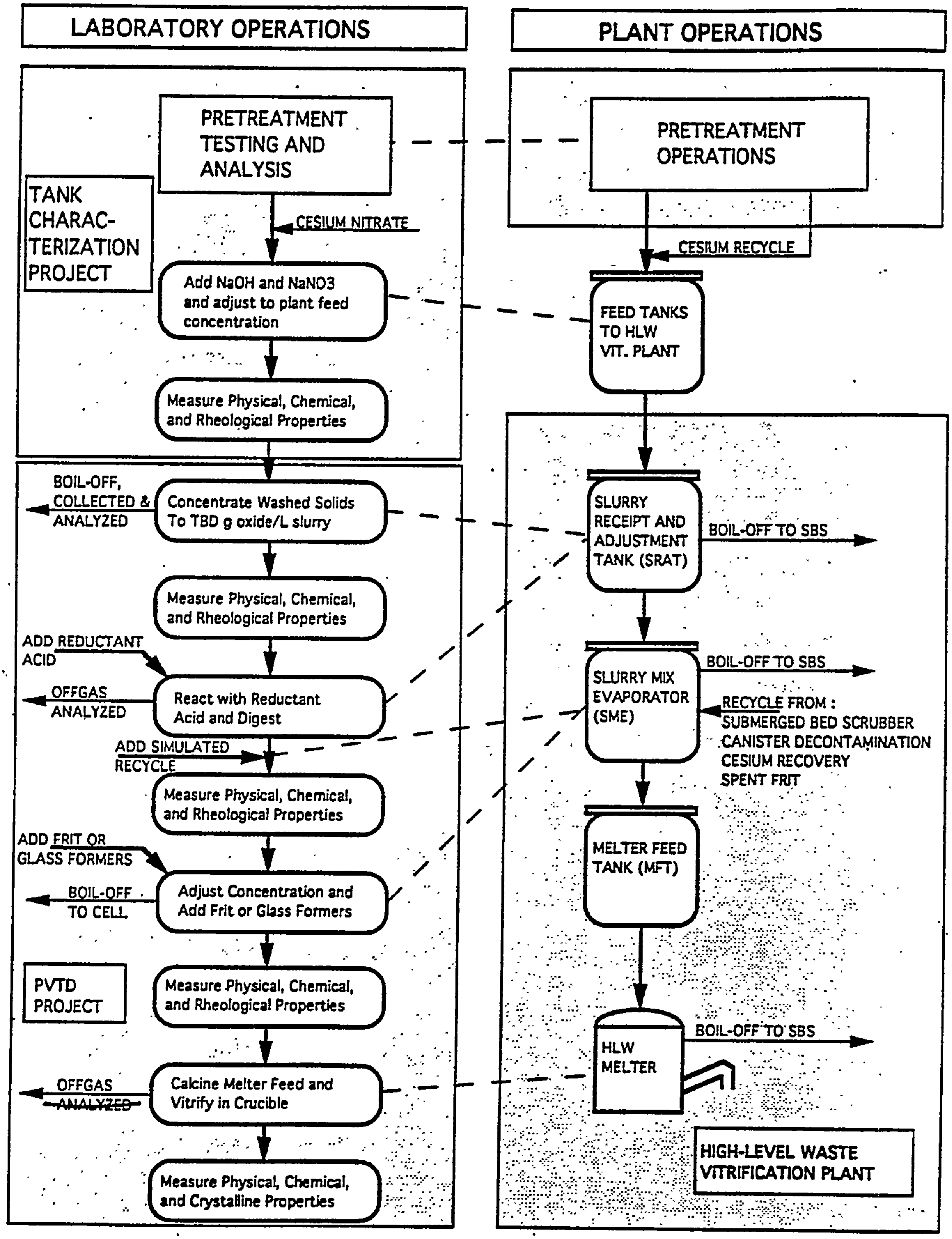


Figure 3.2. HLW Vitrification Process Steps for the NCAW Core Samples

Table 3.1. Differences in Process Steps and Parameters

Process Step/Parameter	101-AZ-C1 (1st Core) 101-AZ-C2 (2nd Core) NCAW Simulant	102-AZ-C1 (3rd Core) 102-AZ-C1 (Simulant)
Cesium Recycle	Not Added	Added
Washed Solids @ 80°C Concentration condensate to cell	@ 101°C,(1) condensate collected and analyzed	
Sample sizes @ 125g TO/L	0.164 L (101-AZ-C1) 0.304 L (101-AZ-C2)	0.115 L (102-AZ-C1) in 0.058 L batches
Formic Acid (90 wt%) Addition Rate	19.5 to 36.1 gal/min (plant scale equivalent(2))	2.0 gal/min (plant scale equivalent(2))
Formic Acid Addition 3 * (moles (moles)	5.7 * moles NO_3(3) (core) NO_3 + 2 * (moles NO_2)	7.7 * moles NO_3(3) (sim)
Formating Temp.	95°C ± 3°C	95° ± 1°C
Digestion Temp.	95°C ± 3°C	101°C ± 1°C
Digestion Period	2 hours	4 hours
Recycle Digestion	0 hours, not added	2 hours
Off-Gas Analysis	None	Formating, digestion, recycle addition
Formated Slurry Concentration	125 g total oxide/L (125 g waste oxide/L)	157 g total oxide/L (140 g waste oxide/L)
Target Waste Loading	25%	28%

---

(1) Core sample was inadvertently dried during concentration.
(2) Plant scale equivalent on volume basis.
(3) Includes nitrate in washed solids and recycle.

volume of slurry. The diluted slurry was agitated for 1 hour and then allowed to settle for 6.5 hours. The wash solution (supernatant following the washing process) was decanted, and the washing and decanting process was repeated one additional time.

The washed solids (solids remaining after the second wash step) from the pretreatment of the NCAW sample became the feed slurry for the HLW vitrification process. The HLW vitrification process steps were concentration, formic acid addition, and frit addition. Due to the evolution of the testing, the first two core samples were processed under slightly different conditions than the third core sample. Differences between the process steps and parameters of these core samples and related simulants are shown in Table 3.1. Chemical additions of $NaNO_3$ and $NaNO_2$ were not required for the two core samples from 101-AZ, but were required for 102-AZ Core 1. Chemical additions of 1.711 g $NaNO_2$, 0.966 g $NaNO_3$, and 0.1105 g $CsNO_3$ were made to the as-received 1O2-AZ Core 1 washed solids sample containing 13.3 g of equivalent waste non-volatile oxides.

3.2.3 Formic Acid Addition

Formic acid was added to the samples to adjust the feed rheology and to reduce the redox-sensitive species in the melter feed. These species must be sufficiently reduced to avoid foaming in the melting process, but must not be reduced to the extent that the metal oxides in the waste precipitate and form an electrically conductive sludge, which could lead to premature failure of the ceramic melter. The amount of formic acid added to the washed solids to achieve the appropriate properties was determined empirically.. This empirical relationship was based upon the redox state of the glass as measured by the ferrous-to-ferric ratio in the vitrified product.

For the two core samples from Tank 101-AZ and the simulant for these core samples, 3 moles of formic acid per mole of nitrate plus 2 moles of formic acid per mole of nitrite in the washed solids were added to the waste to achieve an acceptable redox state in the glass. For the core sample from Tank 102-AZ and its simulant, excess formic acid was added to the sample (moles formic = 5.5 x moles NO_3^-) to ensure hydrogen generation. The sample was heated to 95°C ±3°C and maintained at this temperature while the formic acid (90 wt% solution) was added at a rate of 1 mL/min for the 101-AZ cores and 0.019 ml/min for the 102-AZ core. The equivalent formic acid addition rates on the plant scale are listed in Table 3.1. A range of addition rates is given for the 101-AZ sample because the addition rate (1 mL/min) was held constant, but the sample size was varied. Following the addition of formic acid, the formated sample was digested at 95° or 101°C for a specified period of time (2 to 4 hours). A simulated recycle stream was added to the 102-AZ core after the initial digestion period, and digestion was continued for another 2 hours. The composition of the simulated recycle stream is given in Appendix A, Table A.24. Offgases generated during the formic acid addition, digestion, and recycle addition were collected and analyzed from the Tank 102-AZ sample. The results of this analysis were reported previously.[a]

3.2.4 Frit Addition

Following digestion, the samples were adjusted to concentrations of 125 to 157 g total non-volatile oxides/L, characterized, and combined with glass frit (glass formers) to achieve a waste oxide loading of 25 to 28 percent. Readily available baseline frits (HW39-4 and FY91 New Frit) and one specially designed frit were used to yield glasses with acceptable processing and durability properties. The specific frit added to each core sample was as follows: HW39-4 frit to 101-AZ Core 1, specially designed frit to 101-AZ Core 2, and FY91 New Frit to 102-AZ Core 1. Compositions of the three frits are included in Table 4.7. Before melting each core sample, CVS model predictions of viscosity, electrical conductivity, MCC-1 and PCT durability, and liquidus temperature were evaluated for acceptability. A glass simulant of 101-AZ Core 1 (based on washed solids composition, targeted waste loading, and frit composition) was prepared and analyzed for crystallinity before melting the radioactive glass. To validate glass property models better, different frits were used with each core sample to produce glasses of significantly different compositions. Water was removed by concentration from this frit/slurry mixture to achieve a total non-volatile oxide content of 500 grams per liter.

(a) Langowski, M.H., E.V. Morrey, J.M. Tingey, and M.R. Beckette. 1993. *Offgas Characterization from the Radioactive NCAW Core Sample (102-AZ-C1) and Simulant During HWVP Feed Preparation Testing*. Letter Report for U.S. Department of Energy. Pacific Northwest Laboratory. Richland, Washington.

3.2.5 Calcining and Vitrification

Calcining and vitrification were performed to transform the melter feed into a homogeneous glass under conditions similar to those in a ceramic melter. The melter feed was calcined to drive off volatiles such as carbon dioxide, nitrogen oxides, and water while converting elements into their oxide form, similar to the chemical decompositions that occur in the ceramic melter cold cap. The calcine was then vitrified to fuse the frit and waste into a homogeneous glass.

Dried melter feed was transferred to a Denver Fire Clay (DFC) crucible and calcined for two hours at 600°C in a Lindberg muffle furnace. A low heating rate was used for calcining to avoid solids loss from sudden volatilization. The solids were placed in a cold furnace, and the temperature of the furnace was increased to 200°C over a 20-minute period. The solids were maintained at this temperature for 1 hour and then increased to 400°C over a 15-minute period, held for 1 hour, and then increased to 600°C over a 20-minute period. The solids were then held for 2 hours at 600°C, after which the furnace was turned off and allowed to cool with the door open. After cooling, the calcined feed was weighed and weight loss was calculated. Weight loss ranged from 7.6 to 23 percent. The calcine was ground to -40 mesh for improved homogeneity and was split into thirds for melting.

The feed was melted in a DFC crucible at 1150°C for two hours in a Lindberg muffle furnace. The first third of calcine was transferred to a DFC crucible and placed in the furnace at 1150°C for 30 minutes. The crucible was removed from the furnace and the second third of the calcine was added. This sequence was repeated until all of the calcine was melted. After the last calcine addition the glass was held for 2 hours at 1150°C. Based on past experience with crucible melts, a two-hour soak was deemed sufficient to achieve glass homogeneity.

The molten glass was removed from the furnace and poured into a stainless steel bar mold that had been preheated to 300°C on a hot plate (see Figure 3.3). The bar mold was preheated to avoid shocking the glass and causing it to fracture. When the glass had solidified, the sides of the bar mold were removed (see Figure 3.4) and the glass bar was placed in an annealing furnace at 500°C. The bar was annealed for 2 hours, after which the power to the furnace was shut off and the glass was allowed to cool slowly overnight. Approximately 35% glass weight loss was incurred because the glass stuck to the sides of the crucible. This weight loss is a disadvantage of making small quantities of glass in a radiochemical hot cell. The glass-pouring efficiency is low because of the time required to remove a sample from the furnace and pour the glass using a 10-foot hot cell manipulator. A photograph of glass from 101-AZ Core 1 is shown in Figure 3.5.

3.2.6 Glass Sample Preparation

In preparation for PCT leach and glass analytical testing, a section of each glass bar was crushed to ≤100 mesh. The crushed glass particles were sieved to obtain samples of -100/+200 mesh for use in the PCT test. Portions of the -200 mesh glass were submitted for chemical and radiochemical analysis. In preparation for MCC-1-type leach testing, glass was cut into cubes using a low speed wafering saw with a diamond blade. Cubes were cut with surface areas between 220 and

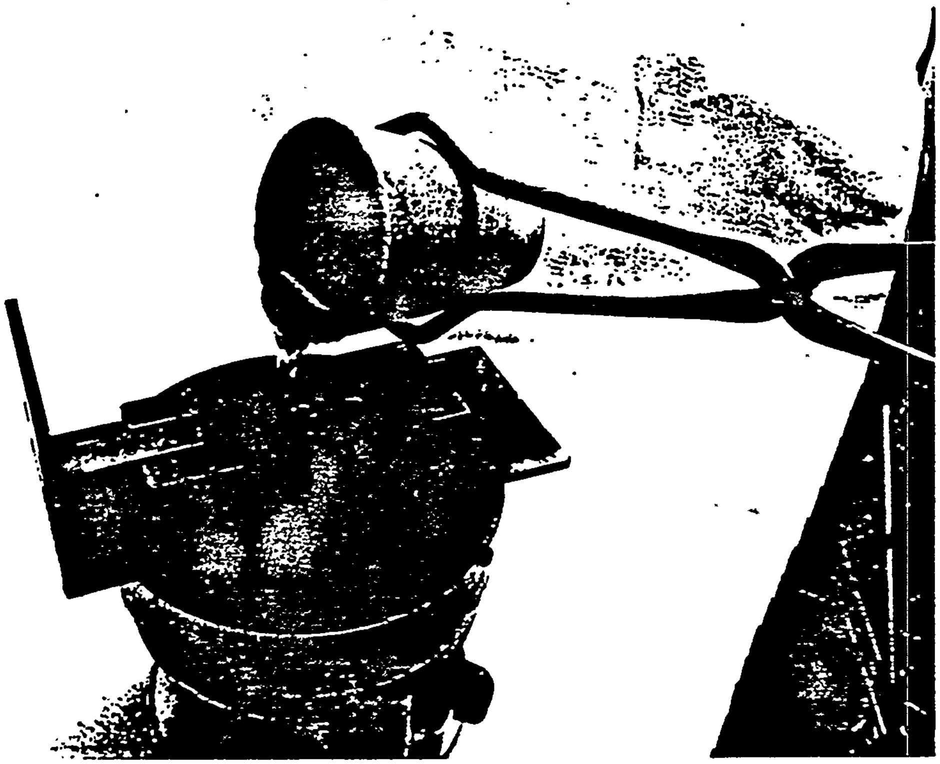


Figure 3.3. Hot Cell Method of Pouring and Quenching Molten Glass from Crucible

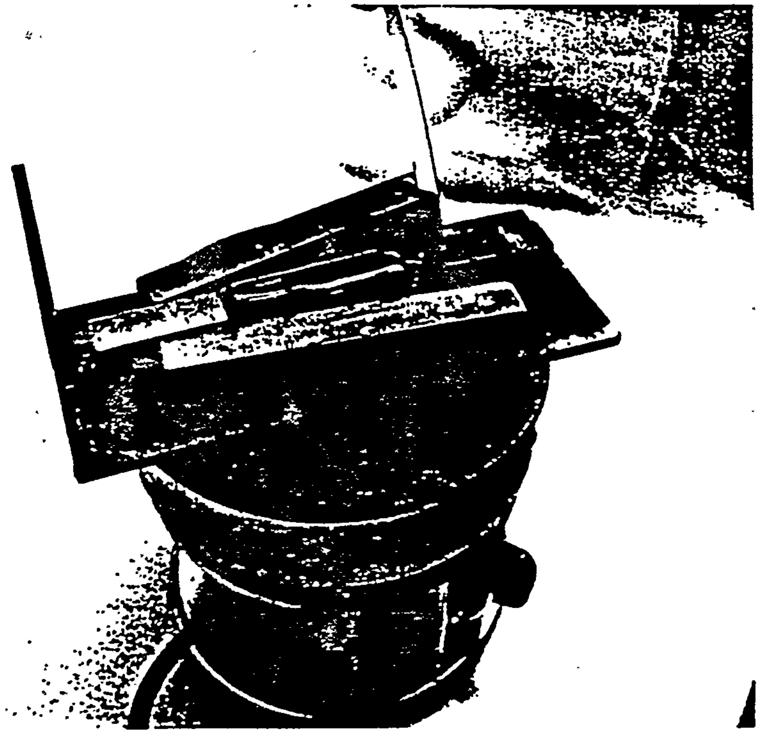


Figure 3.4. Separation of Glass Bar from Mold Before Annealing

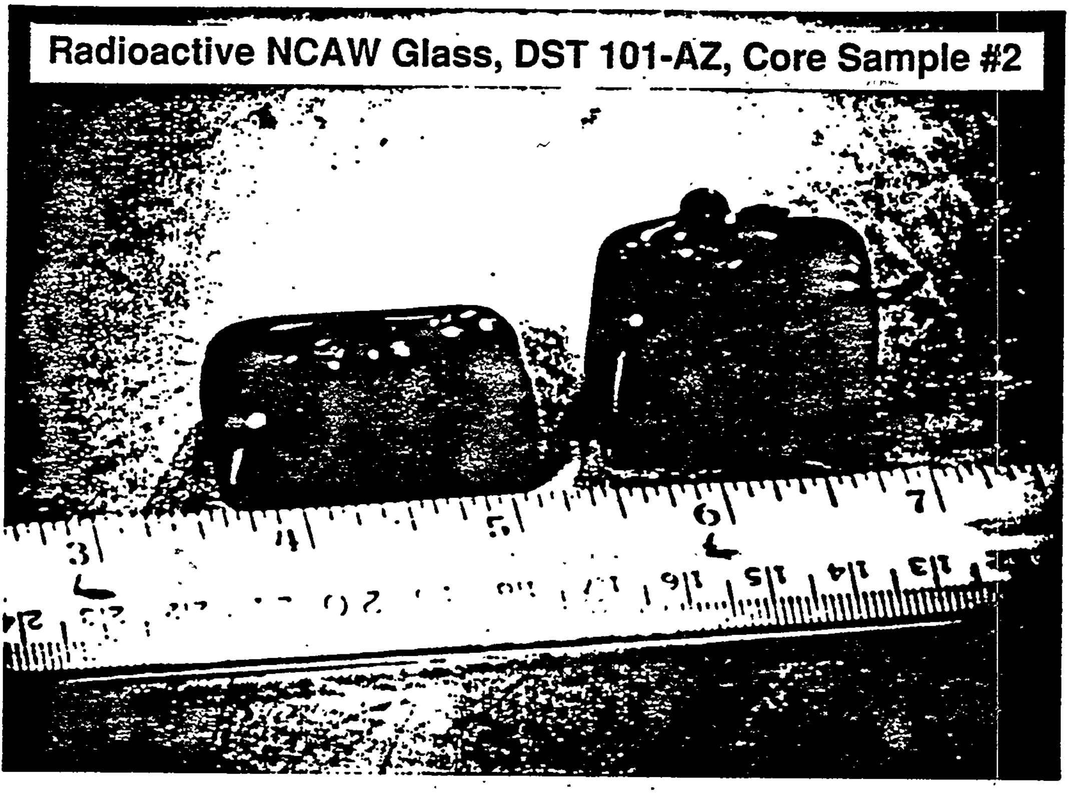


Figure 3.5. Actual Waste Glass from 101-AZ Core 2

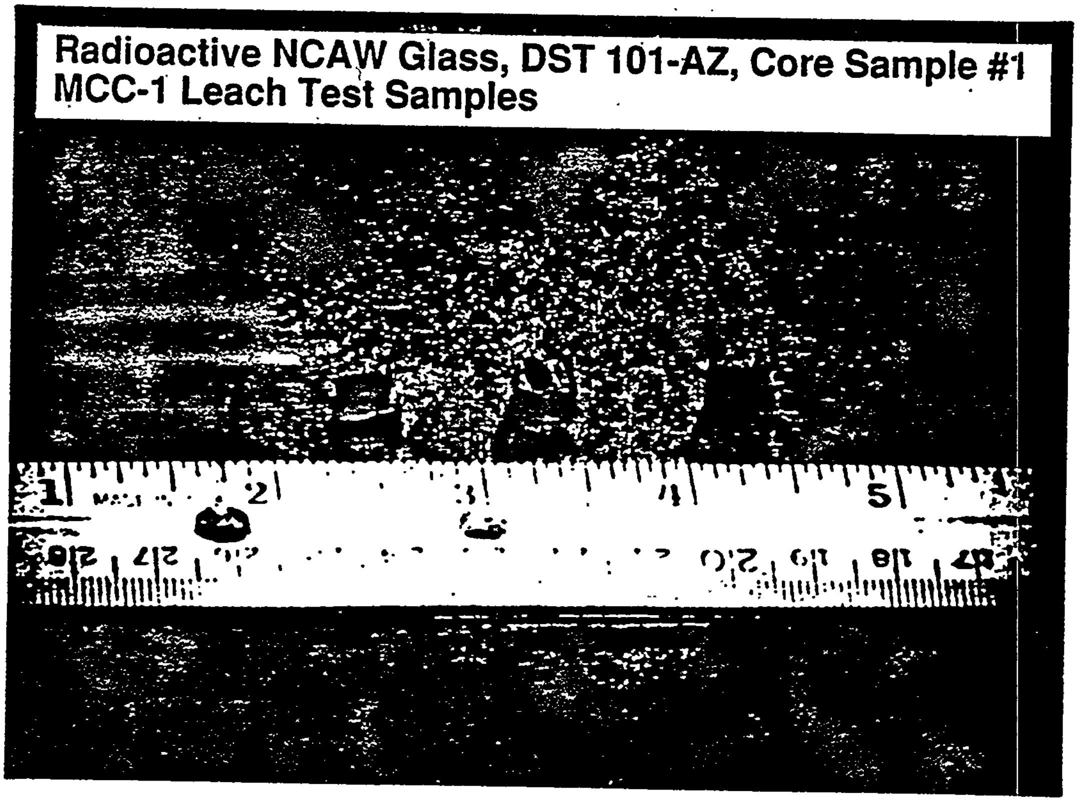


Figure 3.6. MCC-1 Monolith Samples from 101-AZ Core 1

240 mm^2. The final cube dimensions were measured by micrometer and were between 5 and 7 mm on each side (see Figure 3.6). Edges of the glass bars were trimmed so that none of the sides of the cubes originated from the glass-to-metal (i.e., bar mold) interface.

3.2.7 Durability Testing

Five hot cell and 12 laboratory durability tests involving 10 different glasses were conducted to compare simulant and predicted results to those of radioactive glasses. Three of the glasses were fully radioactive glasses prepared from the three core samples, three were direct simulants of the radioactive glasses and the remaining four were reference glasses. A summary of the tests performed and the purpose of each sample is given in Table 3.2.

In hot-cell test #1, the radioactive glasses from the first two core samples were leach-tested using a durability test procedure similar to MCC-1 (Materials Characterization Center, 1984). The procedure was altered to allow leaching in a radioactive environment. Because of the highly radioactive environment, fused-silica-lined stainless-steel containers were used instead of Teflon[•] containers. The fused-silica-lined containers were used to prevent radionuclide plate-out on the stainless steel. The plenum space in the containers was backfilled with argon to prevent uranium complexation with CO_2 and nitric acid generation from the radiolytic production of NO_x. The tests were conducted for 28 days at 90°C using de-aerated, deionized water as a leachant. The glass surface area-to-leachant volume ratio was 0.01 mm^{-1} (220 mm^2 glass surface area in 22 mL of water). The blocks of glass were suspended from the bottom of the leach container using a fused silica pedestal. The leach test was performed using three samples of each of the radioactive glasses, two samples of ATM-10 glass as a reference, two samples of NCAW simulant, and two blanks (i.e., containers of leachant without glass samples). The NCAW simulant glass was leached in the radioactive environment to permit a comparison between leaching this glass both in and out of a radiochemical hot cell.

Results from hot cell test #1 indicated an apparent bias and excessive intra-sample variability that was later attributed to differences in fused-silica lined and Teflon[•] containers and to slightly reduced temperature control in the leach oven. A discussion of the efforts to resolve these issues is contained in Section 3.4 of this report. Laboratory tests #1 and #3 were conducted as part of the investigation to determine the cause of the discrepancies. Laboratory tests #2, #4, #5, and #10 and hot-cell test #2 were performed to select an appropriate container type for continued hot cell PCT durability tests and to establish a basis for continuance of hot-cell testing. Because of the unavoidable, potential differences between tests conducted in the hot cell and the laboratory (e.g., container type) and to minimize the long-term variability associated with the simulant to actual waste glass comparison, simulants of the core samples were tested in the laboratory and in the hot cell adjacent to radioactive samples. This allows a direct comparison between the simulant and the actual waste glass and a direct comparison between the laboratory-tested simulant and the model predictions. Provided hot cell and laboratory durability tests are comparable, a direct comparison of actual waste glass to model

Table 3.2. Summary of Durability Testing

Test Number, Type and Container	Glass Type	Samples	Purpose
Hot Cell Test #1 (MCC-1 in fused-silica)	101-AZ Core 1 101-AZ Core 2 NCAW Simulant ATM-10 Blanks	3 3 2 2 2	Measure MCC-1 durability of 101-AZ Core 1, Core 2 and hot cell processed simulant
Hot Cell Test #2 (PCT in stainless steel)	NCAW Simulant	3	Demonstrate hot cell PCT in stainless steel
Hot Cell Test #3 (PCT in stainless steel)	101-AZ Core 2 101-AZ-C2 Simulant CVS-IS-HW39-4 Blanks	3 3 3 2	Obtain direct PCT comparison between 101-AZ Core 2 and simulant
Hot Cell Test #4 (PCT in stainless steel)	101-AZ Core 1 101-AZ-C1 Simulant CVS-IS-HW39-4 Blanks	3 4 3 2	Obtain direct PCT comparison between 101-AZ Core 1 and simulant
Hot Cell Test #5 (PCT in stainless steel)	102-AZ Core 1 102-AZ-C1 Simulant CVS-IS-HW39-4 Blanks	3 4 3 2	Obtain direct PCT comparison between 102-AZ Core 1 and simulant
Laboratory Test #1 (MCC-1 in Teflon)	NCAW Simulant	2	Obtain comparable laboratory MCC-1 results
Laboratory Test #2 (PCT in Teflon)	NCAW Simulant	2	Obtain comparable laboratory PCT results
Laboratory Test #3 (MCC-1 in fused-silica)	NCAW Simulant Blank	2 1	Investigate MCC-1 biases from fused-silica containers
Laboratory Test #4 (PCT in fused-silica)	NCAW Simulant Blank	2 1	Comparison of candidate PCT leach container
Laboratory Test #5 (PCT in stainless steel)	NCAW Simulant Blanks	3 2	Comparison of candidate PCT leach container
Laboratory Test #6 (PCT in Teflon)	101-AZ-C1 Simulant	2	Obtain laboratory PCT results for direct model comparison
Laboratory Test #7 (PCT in Teflon)	101-AZ-C1 Simulant	2	Obtain laboratory MCC-1 results for direct model comparison
Laboratory Test #8 (PCT in Teflon)	101-AZ-C2 Simulant	2	Obtain laboratory PCT results for direct model comparison
Laboratory Test #9 (MCC-1 in Teflon)	101-AZ-C2 Simulant	2	Obtain laboratory MCC-1 results for direct model comparison
Laboratory Test #10 (PCT in stainless steel)	101-AZ-C2 Simulant CVS-IS-HW39-4 EA Glass Blank	2 2 1 1	Comparison of laboratory PCT in stainless steel to hot cell PCT in stainless and laboratory PCT in teflon
Laboratory Test #11 (PCT in Teflon)	102-AZ-C1 Simulant	2	Obtain laboratory PCT results for direct model comparison
Laboratory Test #12 (MCC-1 in Teflon)	102-AZ-C1 Simulant	2	Obtain laboratory MCC-1 results for direct model comparison

predictions is also appropriate. Hot cell tests #3, #4, and #5 and laboratory tests #6, #7, #8, #9, #11, and #12 were conducted to make these comparison for each of the three core samples.

3.2.8 Description of Analytical Methods

Extensive physical, rheological, chemical, and radiochemical characterizations were performed. Physical characterizations included:

- density
- settling rate
- vol% settled solids
- vol% and wt% centrifuged solids
- wt% total solids
- wt% dissolved solids
- wt% total oxides
- particle size
- shear stress versus shear rate (apparent viscosity).

Chemical analyses included:

- pH
- elemental analyses by inductively coupled argon plasma atomic emission spectroscopy (ICP/AES)
- anions by ion chromatography (IC)
- total carbon (TC), total organic carbon (TOC), total inorganic carbon (TIC)
- total uranium by fluorescence
- X-ray diffraction.

Radiochemical analyses included:

- gamma energy analysis (GEA)
- Cm-242, 243 & 244
- Am-241
- Np-237
- I-129
- Tc-99
- Sr-90
- Se-79
- C-14
- H-3
- Pu-238, 239 & 240
- Total α
- Total ß.

The methods used for these characterizations and analyses are described in Appendix G.

3.2.9 Analyses Performed

Analyses completed on the core samples and simulants are summarized in Table 3.3. Chemical, radiochemical, physical, and rheological analyses were generally performed on each of the washed solids, formated slurry, and melter feed for each core sample. Similar analyses minus the radiochemical were performed on the simulant slurries. Radioactive glasses were characterized with respect to density, chemical, and radiochemical composition, redox (Fe^{+2}/total Fe), crystallinity by X-ray diffraction, and durability. Nonradioactive glasses were characterized with respect to redox (Fe^{+2}/total Fe [NCAW simulant only]), viscosity, electrical conductivity, and durability. Glass durabilities were measured using PCT and MCC-1-type leach tests.

Not shown in Table 3.3 are the number of replicate analyses. In most cases, physical and rheological analyses were performed in duplicate and chemical and radiochemical analyses were performed in single. A notable exception is with ICP analysis, which requires two preparations for each solids sample, and leachate analyses, which were performed in duplicate or triplicate. In the process of adjusting the washed solids, formated slurries, and melter feed samples to the proper concentration, additional sets of density, wt% solids, and wt% oxides data were obtained. Additional analyses related specifically to the offgas generation study (e.g., NH_4, acid/base neutralization capacity) were performed on 102-AZ core 1 and Simulant but are not included in this report (see footnote on page 3.8).

Table 3.3. Summary of Analyses Performed

	101-AZ Core 1									101-AZ Core 2								
	Actual Waste						Glass Simulant			Actual Waste						Glass Simulant		
Analyses	WS	FS	MF	G	MCC-1	PCT	G	MCC-1	PCT	WS	FS	MF	G	MCC-1	PCT	G	MCC-1	PCT
Anions by IC	X	X	X		X	X			X	X	X	X		X	X			X
Elemental by ICP	X		B	X	X	X		X	X	X		B	X	X	X		X	X
Carbon Analysis TC/TIC/TOC	X	X	X								X	X						
Fe^{+2}/Fe Total				X									X					
pH		X	X		X	X		X	X	X	X	X		X	X		X	X
Formic Acid pH Profile	X																	
Crystallinity by XRD				X									X					
Electrical Conductivity							X									X		
Gamma Energy Analysis	X		B	X	X					X		B	X	X				
Total Alpha					X					X				X				
Total Beta			X	X	X					X		X	X	X				
H-3	X		X							X		X						
C-14	X		X							X		X						
Se-79	X		X									X						
Sr-90	X		X	X	X					X		X	X	X				
Tc-99	X		X	X	X					X		X	X	X				
I-129	X		X	X						X		X	X					
Np-237	X			X	X					X			X	X				
Am-241	X			X	X					X			X	X				
Cm-242, 243 + 244	X			X	X					X			X	X				
Pu-238, 239 + 240	X			X	X					X			X	X				
Total U	X			X	X					X			X	X				
Density	X	X	X	X						X	X	X	X					
Settling Rate	X	X	X								X	X						
Vol% Settled Solids	X	X	X							X	X	X						
Density Cent. Solids & Sup.	X	X	X							X	X	X						
Vol% & Wt% Cent. Solids	X	X	X							X	X	X						
Wt% Dissolved & Total Solids	X	X	X							X	X	X						
Wt% Total Oxides	X	X	X							X	X	X						
Particle size distribution		X	X							X	X	X						
Shear Stress vs Shear Rate	X	X	X								X	X						
Viscosity							X									X		

WS = washed solids; FS = formated slurry; MF = melter feed; G = glass; MCC-1 = leachates from MCC-1; PCT = leachates from PCT; B = both slurry and supernatant; Cent. = centrifuged

Table 3.3. (Continued) Summary of Analyses Performed

	102-AZ Core 1												NCAW Simulant						
	Actual Waste						Glass Simulant			Slurry Simulant				Run 1		Run 2		Run 3	
Analyses	WS	WSC	FS	MF	G	PCT	G	MCC-1	PCT	WS	FS	MF	WS	FS	MF	FS	MF	FS	MF
Anions by IC	X	X	X	X		X			X		X	X		X			X	X	X
Elemental by ICP	X	X	B	B	X	X	X	X	X		B	B					X		B
Carbon Analysis TC/TIC/TOC	X		X	X							X	X		X			X	X	X
Fe^{+2}/Fe Total					X														X
pH	X		X	X		X		X	X		X	X						X	X
Formic Acid pH Profile	X																		
Crystallinity by XRD					X		X												
Electrical Conductivity							X												
Gamma Energy Analysis	X				X	X													
Total Alpha					X	X													
Total Beta					X	X													
H-3	X																		
C-14	X		X																
Se-79	X																		
Sr-90	X				X	X													
Tc-99	X				X	X													
I-129	X		X		X														
Np-237	X																		
Am-241	X				X	X													
Cm-242, 243 + 244	X				X	X													
Pu-238, 239 + 240	X		B		X	X													
Total U	X				X	X													
Density	X		X	X	X						X	X		X	X	X	X	X	X
Settling Rate			X	X							X	X						X	X
Vol% Settled Solids	X		X	X							X	X						X	X
Density Cent. Solids & Sup.	X		X	X							X	X						X	X
Vol % & Wt % Cent. Solids	X		X	X							X	X						X	X
Wt% Dissolved & Total Solids	X		X	X							X	X		X	X	X	X	X	X
Wt% Total Oxides	X		X	X							X	X		X	X	X	X	X	X
Particle Size Distribution	X		X	X							X	X						X	X
Shear Stress vs Shear Rate	X		X	X							X	X						X	X
Viscosity							X												

WS = washed solids; FS = formated slurry; MF = melter feed; G = glass; MCC-1 = leachates from MCC-1; PCT = leachates from PCT; B = both slurry and supernatant ; Cent. = centrifuged

3.3 Test Equipment

The equipment used for formic acid addition of the samples from cores 1 and 2 from Tank 101-AZ is shown in Figure 3.7. The sample is placed in the 500-ml reaction vessel; the temperature and pH of the sample are monitored with a Type K thermocouple connected to a digital thermometer and a combination pH electrode connected to a pH meter. The sample is stirred with a mechanical stirrer. A heating mantle surrounding the reaction vessel obtains the temperatures required for the digestion and formic acid addition steps. The temperature of the heating mantle is controlled by a temperature controller in line with a variac. The heating mantle temperature is monitored and controlled by a Type K thermocouple placed between the heating mantle and the reaction vessel. Formic acid is pumped from the reservoir to 1 in. below the surface of the sample with a peristaltic pump. The flow rate of the pump was calibrated before the addition of formic acid. The vapor and offgases produced passes through a water-jacketed double condenser and the condensate runs back into the reaction vessel. The temperature of the cooling water was ≈25°C.

A schematic of the equipment used for formic acid addition of the sample from Tank 102-AZ and related simulants is given in Figure 3.8. This system is significantly different than the previous system because off-gas measurement capabilities were added to the system. The volume of the reaction vessel is ≈120 mL. The temperature and pH of the sample in the reaction vessel is monitored with a thermistor and combination electrode connected to a pH meter, which records both temperature and pH. The pH is corrected for temperature effects. The formic acid is added through a line below the surface of the sample. The formic acid is pumped from a reservoir to the reaction vessel with a peristaltic pump. All gases produced during the digestion, formic acid addition, and recycle addition pass through a double condenser system where the condensate is trapped after each condenser. The temperature of the sample is maintained with a heat tape wrapped around the reaction vessel. The temperature of the sample is controlled by a temperature controller and Type K thermocouple placed between the heat tape and the reaction vessel. This system is shown in Figure 3.9.

Radioactive calcining and vitrification were performed in a 1200°C capacity Lindberg muffle furnace with Type S thermocouple temperature measurement. The DFC crucibles measured about 2.5 in. in diameter. Crucibles were removed from the furnace with tongs and glass was poured onto a preheated stainless steel bar mold (see Figures 3.10 and 3.11). The glass bars were annealed in a 1200°C capacity Thermolyne muffle furnace with Type K thermocouple temperature measurement.

Glass sample crushing and sieving was performed with a Plattner style, stainless-steel mortar and pestle and 3-in.-diameter stainless-steel sieves. Glass was cut with a Struers Miniton cutoff machine with diamond blades. The saw was equipped with a micrometer on the chuck to allow cuts to be made at precise locations.

Durability testing was performed in a Blue M forced-air convection oven with a customized rack for holding up to 12 leach vessels. Leach vessels used were Parr Instrument Co. 45 ml screw cap bombs fabricated from 304L stainless steel and Teflon gasket. Fused-silica liners and pedestals were used in some hot cell and laboratory leach tests.

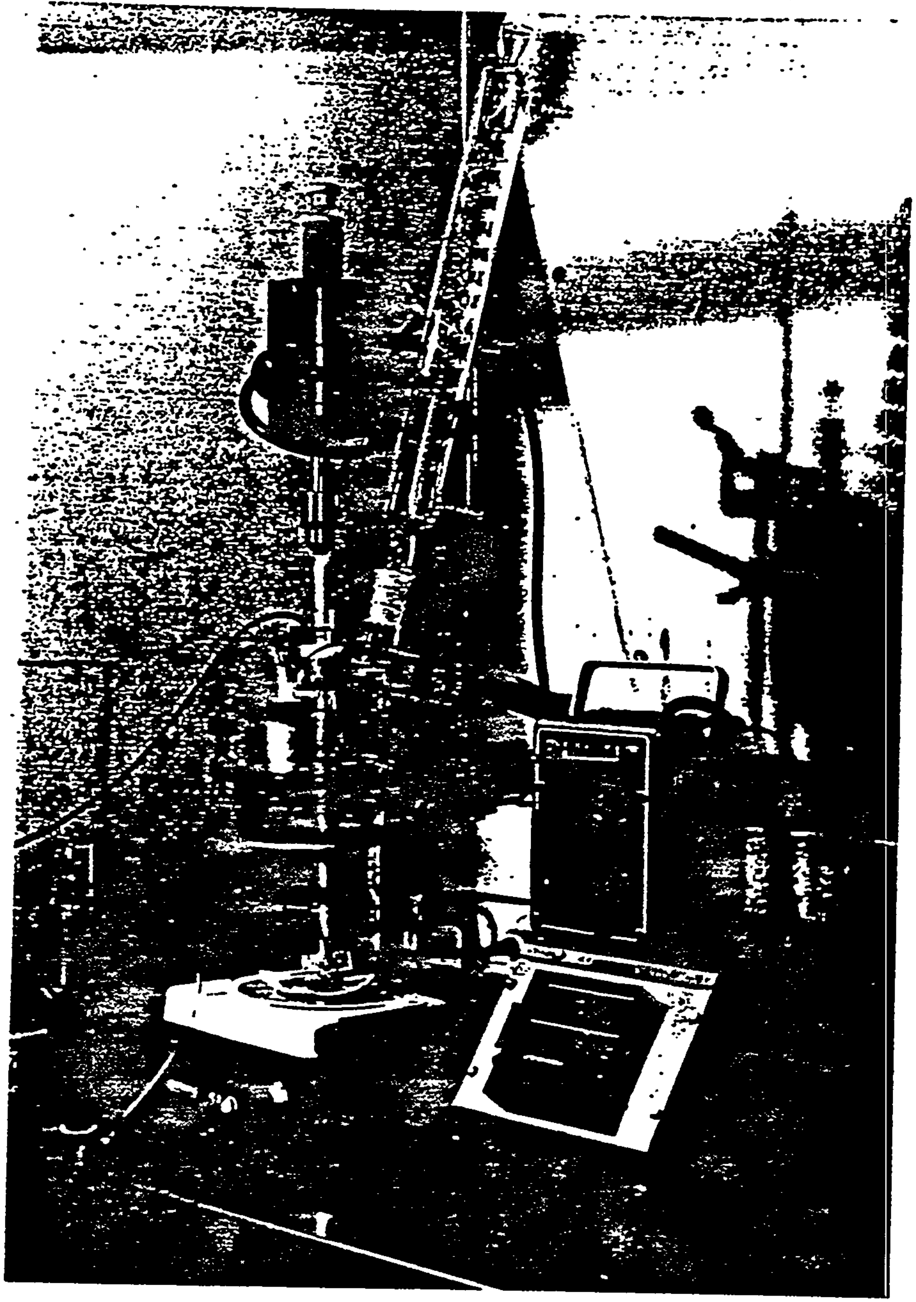


Figure 3.7. Hot Cell Equipment Setup for Formic Acid Addition on 101-AZ Core Samples

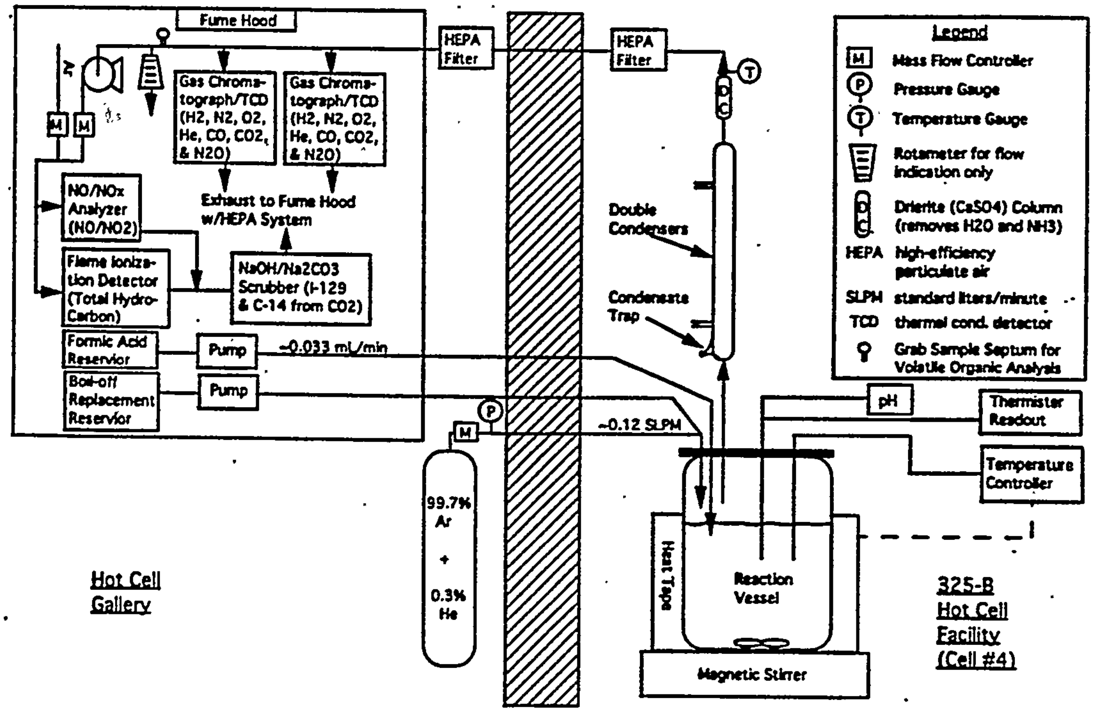


Figure 3.8. Hot Cell Equipment Setup for Formic Acid Addition and Offgas Analysis on 102-AZ Core 1

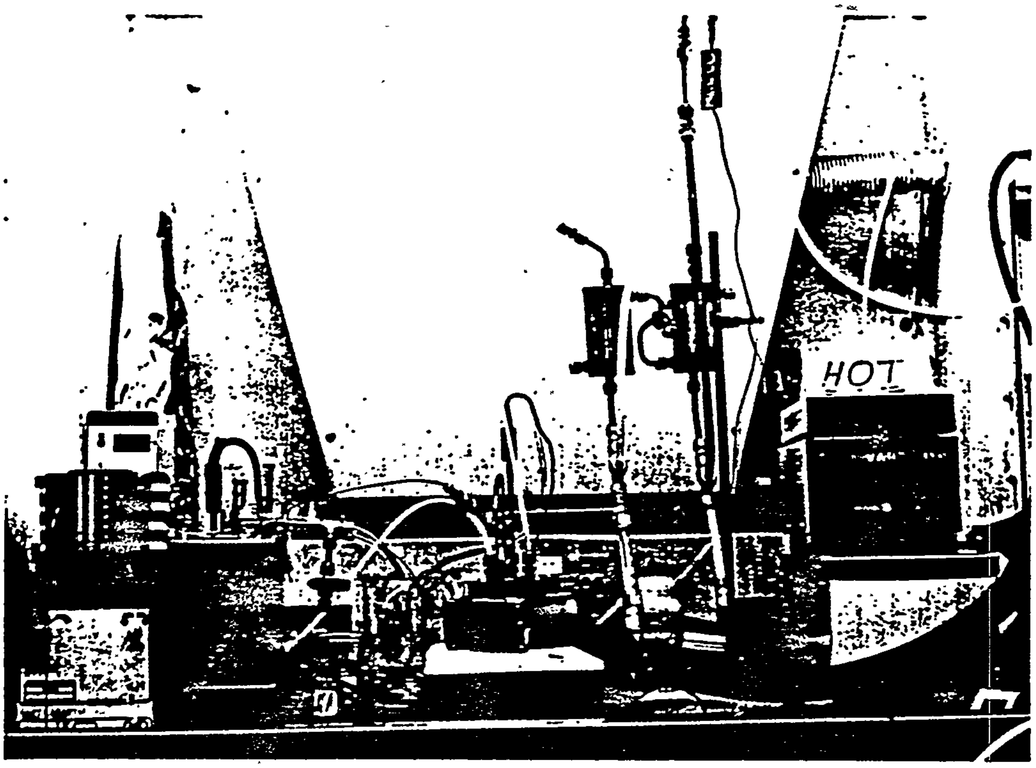


Figure 3.9. Hot Cell Formic Acid Addition Equipment for 102-AZ Core 1

3.4 Test Anomalies

Three anomalies in the processing and characterization of the core samples should be considered when interpreting selected data. A pH probe broke into 101-AZ Core 2 formated slurry and resulted in additions of K and Cl to the waste, but should not affect results or conclusions from that core sample. Inadvertent drying of the washed solids from 102-AZ Core 1 probably resulted in altered physical and rheological properties, including higher than normal settling rates and lower than normal yield stress and apparent viscosity. The use of fused-silica-lined leach containers and less than optimal temperature control in the MCC-1 testing of 101-AZ Core 1 and 2 probably resulted in decreased releases for all samples present in Hot Cell Test #1.

3.4.1 pH Probe Broken into 101-AZ Core 2 Formated Slurry

During formating of the 101-AZ Core 2 washed solids, a pH electrode was broken in the reaction vessel, resulting in a 10 ± 1 ml addition of 3.0M KCl to the waste slurry (309.3 ml of washed solids at 123 g total oxide (TO)/L). Impacts of the KCl addition on processability, final glass composition, and glass acceptability were assessed and judged to be acceptable before proceeding. The frit composition designed for this core sample was adjusted downward in Na to account for the addition of K. Sodium and K are both considered as Na for the purpose of CVS modelling. Estimated impacts to washed solids composition and projected glass composition resulting from the KCl addition are as follows:

Composition	Before KCl Addition	After KCl Addition
K in washed solids	<DL	3450 mg/kg ws
Cl in washed solids	40 mg/kg ws	3170 mg/kg ws
K_2O in glass at 30% waste loading	0.0 wt%	1.08 wt%
Cl in glass at 30% waste loading	0.01 wt%	0.82 wt%

Notes: kg ws = kilograms of washed solids at 0.112 g waste oxide/g slurry
<DL = less than detection limit

3.4.2 Drying of 102-AZ Core 1 Washed Solids

The 102-AZ Core 1 slurry was inadvertently allowed to dry out twice while in the washed solids state. Radiolytic degradation of the storage container lid allowed the slurry to dry at room temperature over a period of months. The problem was discovered when the washed solids were retrieved for processing. Deionized water was added to the dried solids to bring the slurry to 31g TO/L before a boil-off and condensate-collection step. The sample was concentrated at boiling (approximately 101°C) to achieve a target concentration of 125g TO/L. Because of evaporative losses in the system and the inability to observe the slurry level in the reaction vessel visually, the sample was allowed to dry out again. Deionized water was added to the dried solids to achieve the desired concentration and the sample was processed as planned.

Experience in the hot cell with several tank-waste samples that have been dried and rewetted showed alteration to the physical and rheological properties but no change to the chemical properties. For those samples that have been dried and rewetted, settling rates increased substantially and rheological strengths decreased significantly. These observations can also be seen with the 102-AZ Core 1 when compared to the other two core samples in this report.

One plausible explanation is that the small colloidal sized particles become close enough to other colloidal or larger sized particles to bond together. The ratio of bond strength to particle mass involved with these small particles is high enough to prevent breakage when diluting or mixing. Irreversible agglomeration of these small particles would tend to increase the settling rate and decrease the yield stress and apparent viscosity.

3.4.3 In-Cell MCC-1 Testing Discrepancies

An apparent bias and excessive replicate sample variability were experienced for all glasses in the MCC-1 test completed in the hot cell. Comparative in-cell and out-of-cell data on non-radioactive glasses included in the test are provided in Figures 3.10 and 3.11. The in-cell releases for NCAW simulant were an average of 43% lower than those for the out-of-cell test. The differences for the ATM-10 glass were not as significant. The boron, lithium, and sodium releases were about 13% lower for the in-cell tests, while the silicon release was 24% higher. Variation between replicate samples of all glasses tested is illustrated in Figures 3.12 through 3.15. The 101-AZ Core 1 glass exhibited the greatest replicate sample variability with sample #3 being an average of 43% lower than sample #1.

A number of hypotheses were developed to explain the discrepancies between in-cell and out-of-cell results and replicate samples. Possible contributions to one or both of the discrepancies include difference in container type, inaccurate temperature measurement, temperature gradients within the oven, inconsistency in leachability of silica liners, poor sample preparation, radiolysis of air within the leach container plenum, and analytical errors. Analytical errors were ruled out by examining standards run before and after test samples. Poor sample preparation was ruled out because the releases were low, not high. A poor washing would cause fines to be placed in the leachant, which would increase releases because of increased surface areas. Radiolysis from background sources is a possible contribution, but was not specifically investigated. The questions of container type, temperature variation within the oven, and inaccurate temperature measurement are discussed below.

3.4.3.1 Investigation of Type of Container

The most likely explanation for the difference between in-cell and out-of-cell results is the fact that two sets of tests were conducted in different leach containers. The in-cell tests were conducted in fused-silica-lined stainless-steel containers that were backfilled with argon; the out-of-cell tests were conducted in Teflon* containers with no backfilling. The blanks from the in-cell test had a significant background of Si (4.2 to 6.5μg/ml), between 16% and 42% of the total concentration of Si found in the radioactive samples. Dissolved silica is well known to decrease the rate of dissolution of nuclear waste glasses. The Si source in the blanks is the fused-silica liners and pedestals. The leachants were checked for Si contamination before the test and were found to be clean. To investigate the effect of the liners, the out-of-cell test was repeated using fused-silica-lined stainless-steel containers with argon backfilling to simulate the in-cell test. Results from this and prior MCC-1 tests on NCAW glass are shown in Table 3.4 and Figure 3.16. The laboratory test in fused-silica produced B, Li, and Na releases 20% lower than laboratory tests in Teflon. This bias is not of the same magnitude as the

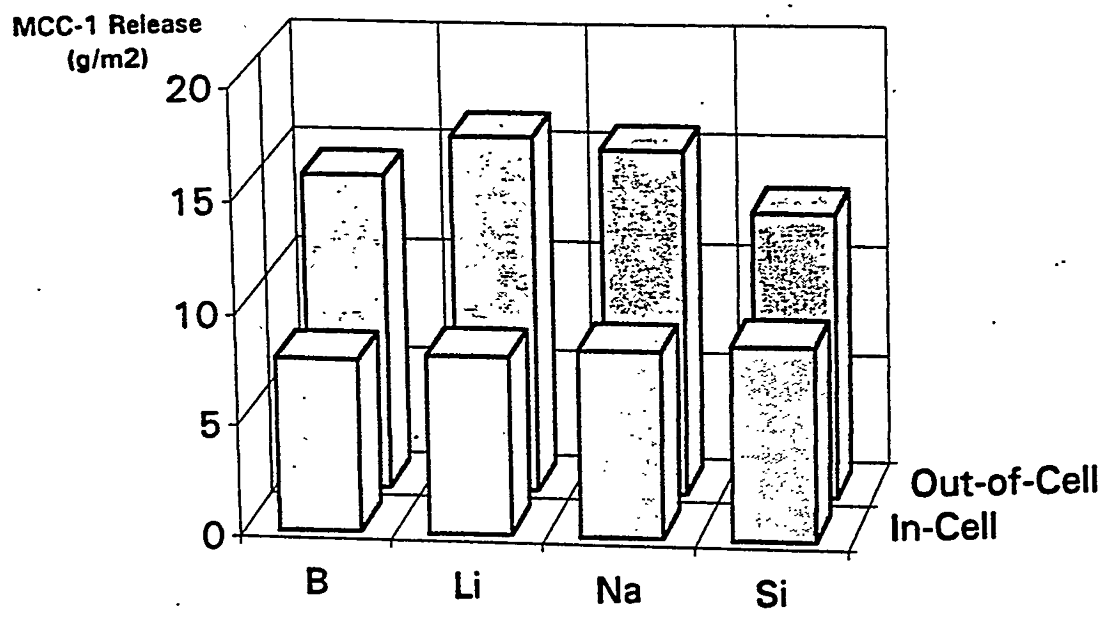


Figure 3.10. In-Cell versus Out-of-Cell MCC-1 Discrepancy for NCAW Glass

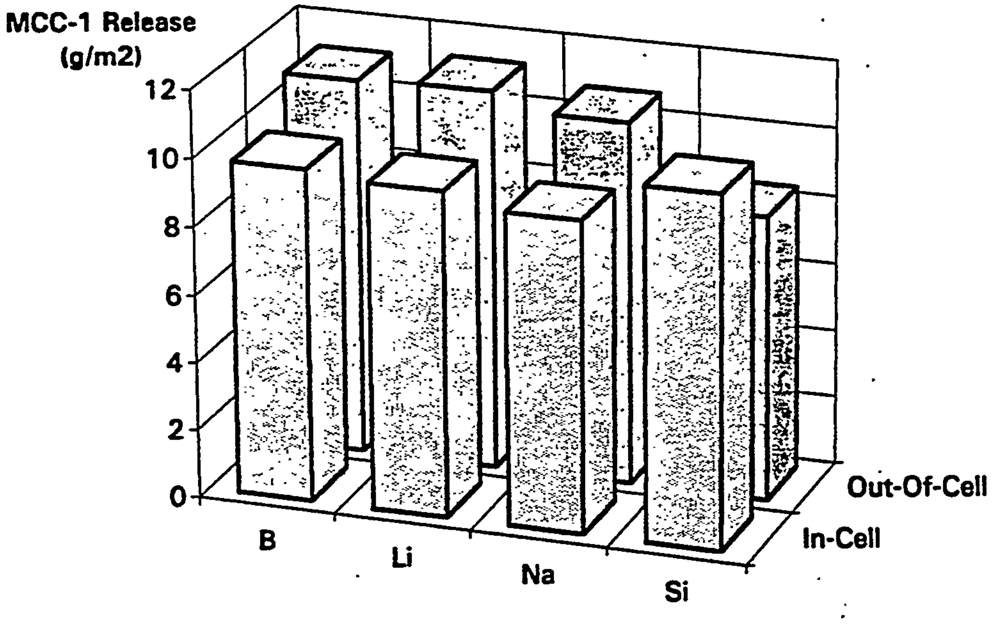


Figure 3.11. In-Cell versus Out-of-Cell MCC-1 Discrepancy for ATM-10

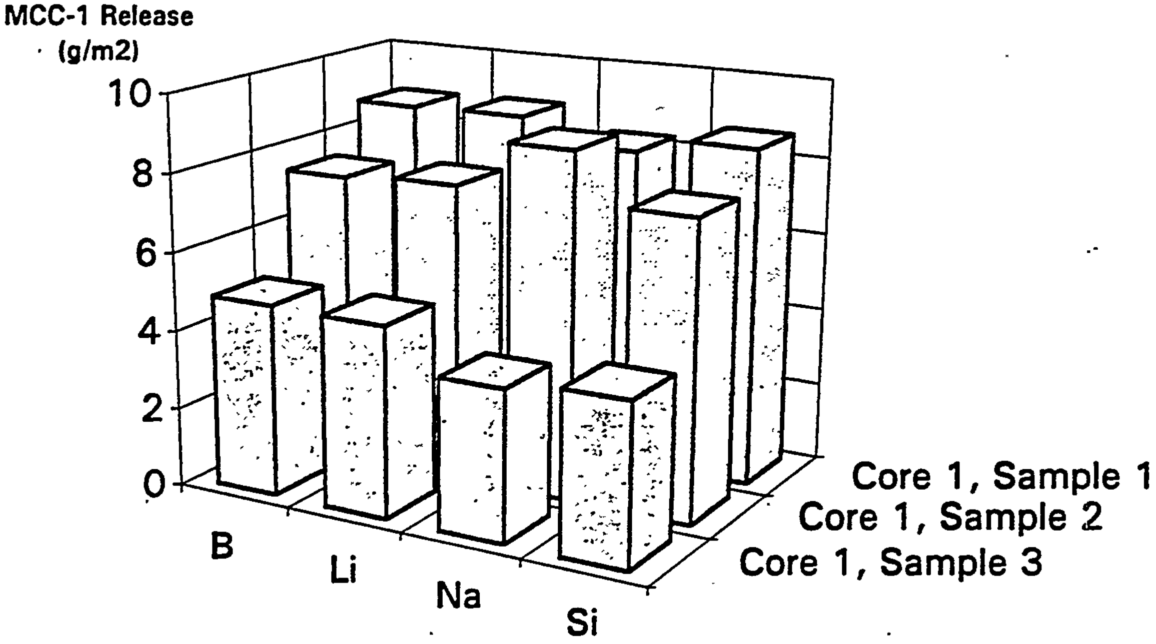


Figure 3.12. Replicate Sample MCC-1 Variability for 101-AZ Core 1

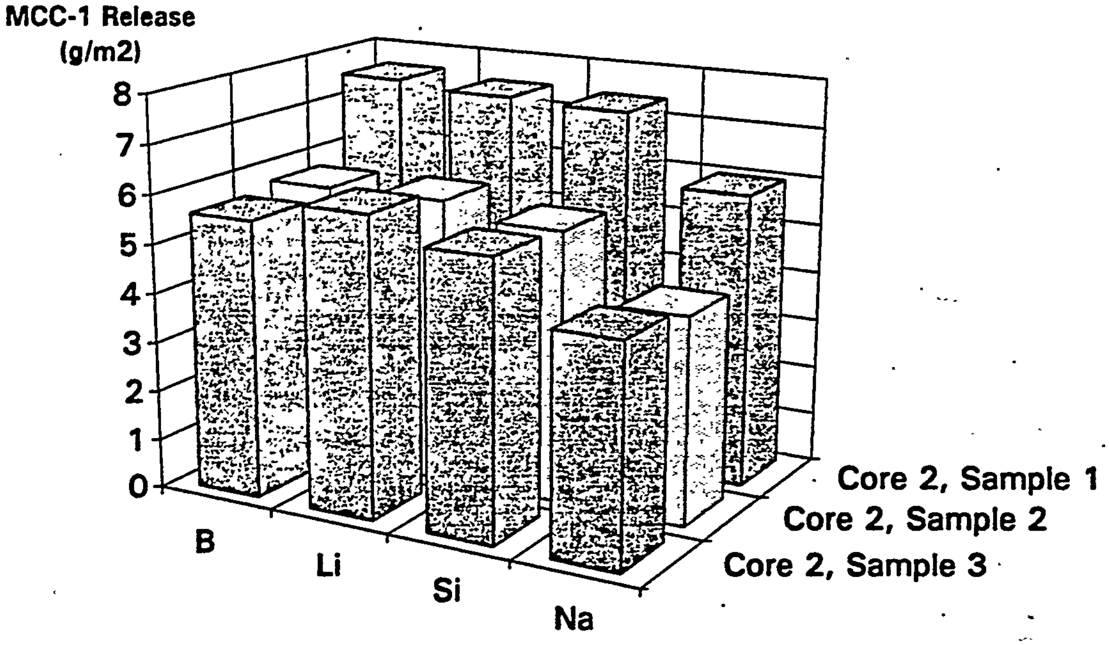


Figure 3.13. Replicate Sample MCC-1 Variability for 101-AZ Core 2

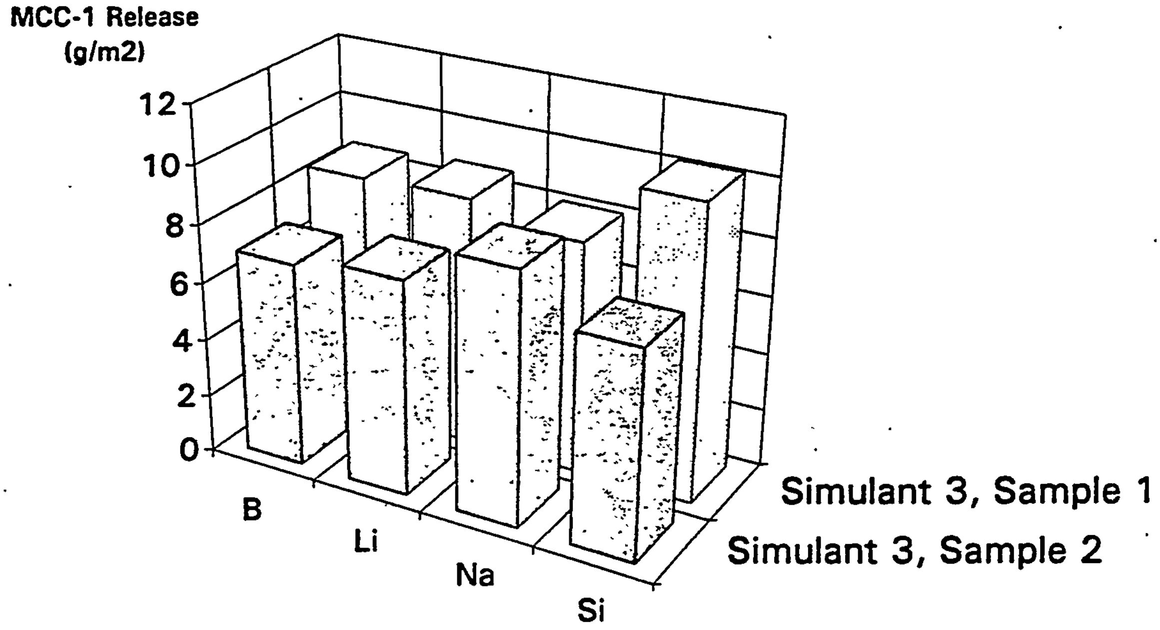


Figure 3.14. Replicate Sample MCC-1 Variability for NCAW Simulant

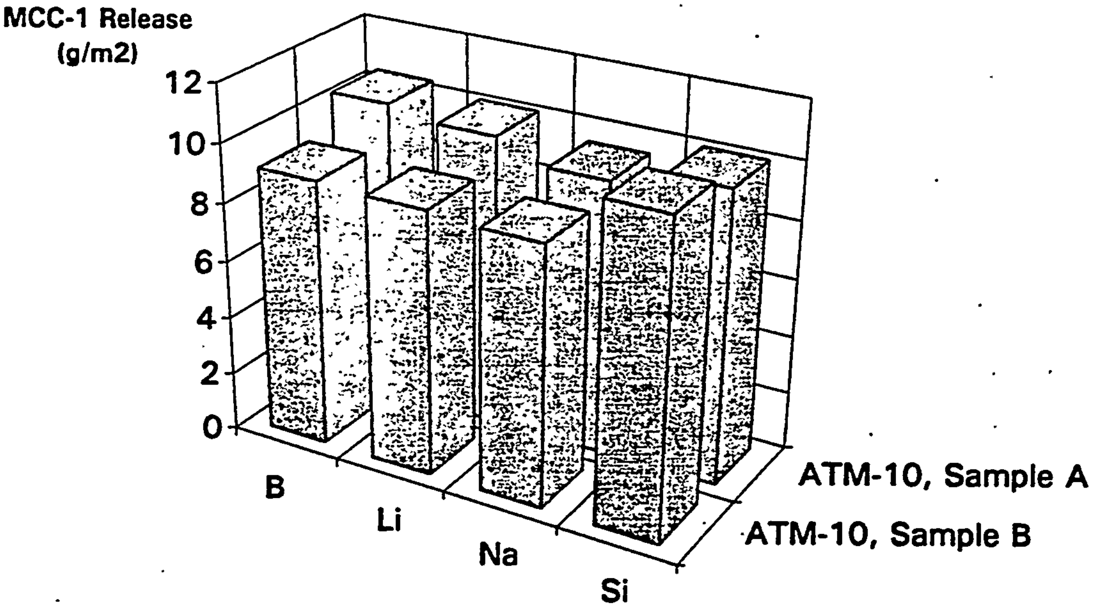


Figure 3.15. Replicate Sample MCC-1 Variability for ATM-10

Table 3.4. MCC-1 Leach Test Results for NCAW Simulant Glass

			——Ln Transformed——			——Exp Transformed——		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
(Lab Test #1 in Teflon - based on sample size of 2)								
B	14.327	2.04	2.662	2.845	2.479	14.326	17.207	11.928
Li	16.149	0.00	2.782	2.782	2.782	16.149	16.149	16.149
Na	15.624	1.39	2.749	2.873	2.624	15.624	17.696	13.794
Si	12.949	7.90	2.561	3.271	1.851	12.948	26.326	6.368
(Hot Cell Test #1 in fused-silica - based on sample size of 2)								
B	7.533	13.39	2.015	3.218	0.812	7.499	24.966	2.253
Li	8.403	10.57	2.126	3.075	1.176	8.380	21.659	3.242
Na	8.463	16.57	2.135	3.623	0.646	8.456	37.463	1.909
Si	8.799	26.82	2.159	4.569	-0.250	8.665	96.415	0.779
(Lab Test #3 in fused-silica - based on sample size of 2)								
B	11.816	10.70	2.467	3.428	1.505	11.783	30.820	4.505
Li	12.261	10.35	2.504	3.433	1.574	12.228	30.984	4.826
Na	12.867	10.10	2.552	3.460	1.645	12.834	31.807	5.179
Si	17.292	10.19	2.849	3.765	1.933	17.273	43.163	6.912

CI = Confidence Interval
MCC-1 = Materials Characterization Center (MCC-1)
Exp Transformed = retransformed data from ln form to original form

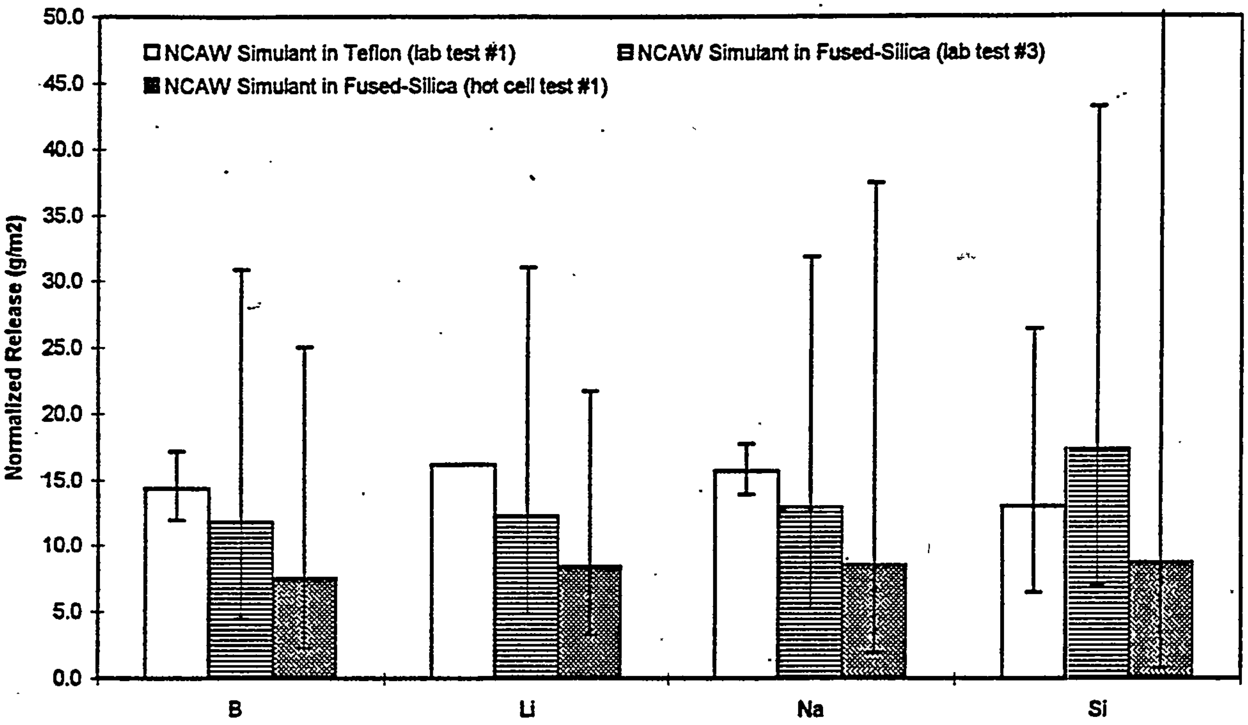


Figure 3.16. Comparison of Alternate Leach Vessels considered for MCC-1 (Error Bars = Estimated 95% Confidence Interval)

43% bias experienced in the hot cell, but does indicate a probable contributing cause. Note also the rather large percent RSDs and confidence intervals for tests in fused-silica liners. A possible cause of this is variability in the release of Si from the liners. The liners used in testing were fabricated in an onsite glass shop and certainly experienced non-uniform temperature histories.

3.4.3.2 Investigation of Temperature Effects

A complete temperature map of the leach oven in the hot cell was performed after the fact to correlate the variation in oven temperature with the differences in releases from replicate samples. A temperature gradient of 2.6°C was measured from highest to lowest temperature sample location. These temperature measurements were done without vessels present, however. Subsequent testing in the hot cell indicated that the temperature gradient increased by approximately 1°C when the oven was fully loaded with 12 vessels, which suggests the 2.6°C temperature gradient is understated by 1°C. This additional gradient is due to the sample tray design, which inhibits convection from top to bottom when all sample locations are filled. Boron releases for replicate samples were plotted as a function of relative temperature to show any apparent variation due to temperature gradient (see Figure 3.17). The data indicate a possible variation in replicate samples due to oven temperature gradients.

Temperature dependence of glass corrosion reaction rates are typically expressed through the Arrhenius equation:

$$k = A \exp\left[\frac{-E_{act}}{RT}\right] \tag{3.1}$$

where k is the reaction rate constant, A is a constant, E_{act} is the activation energy, T is absolute temperature, and R is the gas constant. Several parameters, including glass composition, solution composition, and controlling reaction mechanisms, will impact the activation energy. Activation energies have been reported for nuclear waste glass from 22 to 150 kJ/mol (Cunnane 1994).

Model simulations were performed on the radioactive glasses to estimate effects of temperature change on MCC-1 results. The concentration of Si in solution was so low that it had little effect on dissolution rate. The dissolution rate is essentially the forward rate and is nearly constant throughout the 28-day test. Temperature effects were close to that defined by the Arrhenius Law. Based on this and an assumed activation energy of 80 kJ/mole, the estimated temperature dependencies are as follows:

90°C - 89°C = 7% reduction in reaction rate
90°C - 88°C = 14% reduction in reaction rate
90°C - 87°C = 20% reduction in reaction rate
90°C - 86°C = 26% reduction in reaction rate
90°C - 85°C = 31% reduction in reaction rate

Due to the temperature gradient in the oven, thermocouple placement, and minor thermocouple inaccuracy, actual sample temperatures for the in-cell MCC-1 test were estimated to range between 86.5°C and 89.1°C. This is an average of 87.8°C or 2.2°C below the set point for the test. This easily could have resulted in an average of 15% reduction in reaction rate.

Based on the above investigations, the discrepancies observed in the MCC-1 hot cell test were attributed to container type, temperature gradients within the oven, and minor temperature measurement inaccuracies. To alleviate the discrepancies in future tests, stainless steel vessels were used instead of fused-silica, thermistors were used for temperature measurement instead of thermocouples to increase temperature accuracy, and a new hot cell oven was procured to minimize thermal gradients. Subsequent PCT tests were conducted in the hot cell with good agreement with out-of-cell results (see Section 4.5.1.5). No further MCC-1 hot cell tests were conducted.

Table 4.1. Chemical Composition of Washed Solids Slurries

Non-Volatile Components (wt% oxide basis)	101-AZ-C1	101-AZ-C2	102-AZ-C1	Simulant #3	Simulant(c) (102-AZ-C1)	HWVP Reference[a]		
						Nominal	Minimum	Maximum
Al_2O_3	17.0	9.0	21.5	10.0	18.7	9	2	26
BaO	0.2	0.3	0.1	0.4	0.1	0.4	0	20
CaO	1.0	1.5	1.4	0.6	0.8	0.3	0	20
CdO	1.3	3.3	3.7	2.6	3.2	3	0	10
Cr_2O_3	0.8	0.2	0.5	0.7	0.4	0.5	0	2
Fe_2O_3	38.0	46.0	40.0	34.0	35.0	28	8	60
La_2O_3	0.6	1.8	1.1	0.9	1.0	2.9	0	8
Nd_2O_3	0.6	1.1	0.9	3.4	2.4	1.6	0	8
MnO_2	1.9	0.8	1.0	1.2	0.5	0.6	0	20
MoO_3	0.03	< 0.017	< 0.038	1.4		1.2	0	8
Na_2O	15.0	11.0	12.0	16.0	25.8	18	4.5	22
NiO	1.2	2.9	2.4	2.8	2.0	2.3	0	8
P_2O_5	2.7	< 2.4	< 2.09	< DL	1.5	0.88	0	4
PdO	NM	NM.	NM		0.13	0.2	0	1
Rh_2O_3	< 0.08	< 0.19	< 0.12	< DL	0.11	0.2	0	1
Ru_2O_3	< 0.08	0.34	< 0.13	0.82	0.40	0.6	0	1
F^-	0.20	0.16	0.07			1.2	0	6.9
SO_3					0.5	0.65	0	2
Volatile Components (g/100g oxides)								
Cl^-	0.05	0.02	0.02		0.05	0.3	0	0.3
CO_3^{2-}	(b)	(b)	(b)		(b)	17	2.4	30
NO_2^-	6.09	3.68	1.89		8.76	2.4	0	36
NO_3^-	6.24	1.87	0.69			19	0	36
TOC	9.70	0.25	0.98			2	0	11

(a) HWVP reference composition from WHC-SD-HWV-DP-001, HWVP Technical Data Package, Section 13, Rev. 6.
(b) Carbonate analysis methods have been shown to be inaccurate for the waste matrix.
(c) Except when indicated, concentrations are measured values. Oxide basis ignores excess Na in total oxides.
(d) Excess sodium was added as Na_2CO_3 to match CO_3^{2-} value.
(e) In absence of measured concentrations or reliable measured values, theoretical values/added amounts are reported.

was removed in the washing process. Comparison of the concentration of sodium in the washed solids, the sludge before washing, and the reference nominal value for the previously planned HWVP indicates that acceptable washing efficiencies are being achieved on the laboratory-scale processes. The concentrations of the major components and those components of special interest for all three washed-solids slurries are within the limits of the previously planned HWVP reference.

In Figure 4.1, the concentrations of the analyzed elements for each of the washed solids slurries are compared. A comparison of chemical composition of simulant 102-AZ Core 1 and the corresponding core sample indicates that accurate chemical simulants can be prepared using the method described in Section 3.2.1. Significant differences in the concentration of the major analytes were observed between the three core samples and the NCAW simulant. It should be noted that the weight fraction of solids for each of the samples varied significantly. If the data are presented based on dried or calcined weight, the concentrations of the major analytes in the three actual waste samples correspond more closely than are observed on a wet-weight basis. The NCAW simulant does not match any of the actual waste samples because it was prepared to the previously planned HWVP reference nominal values, with the exception of the nitrate and nitrite composition. The nitrate and nitrite concentrations were based on the concentrations of these analytes in the actual waste sample from 101-AZ Core 1.

4.1.2 Radiochemical Characterization

The radionuclide compositions of the washed-solids slurries from the three core samples are reported in Appendix B, Tables B.1 through B.3. The major radionuclides present in these samples are ^{137}Cs, ^{90}Sr, ^{144}Ce, and ^{106}Ru. All three samples are transuranic (they contain > 100 nCi/g transuranic isotopes). The majority of the transuranic activity is due to americium and plutonium. A comparison of the specific activity of the measured radionuclides in these three samples is given in Figure 4.2. The only radionuclides affected by the washing steps were ^{137}Cs and ^{125}Sb. The activity of the wash solutions is due primarily to ^{137}Cs. None of the supernatants or wash solutions were transuranic. Measured radionuclide activities were within the previous HWVP specifications, with the exception of ^{129}I, ^{90}Sr, and ^{60}Co.

The simulants were prepared as chemical simulants only and did not contain any radionuclides. The absence of radionuclides in the simulants eliminated many of the regulations and shielding necessary to handle radioactive samples, and allowed the simulant studies to be performed in a more timely and cost-efficient manner.

4.1.3 Physical Characterization

Physical properties for the three actual waste samples and the two simulants are reported in Appendix C. Tables C.1 through C.5 present a summary of the physical properties measured in these samples. The particle-size distribution for the washed solids and the settling behavior were reported previously in the tank characterization reports (Peterson et al. 1989; see footnotes a and b on page 4.1). Particle-size distribution of the simulant-washed solids slurries was not measured. A comparison of the physical properties of the core sample and simulant washed-solids slurries is given in Table 4.2.

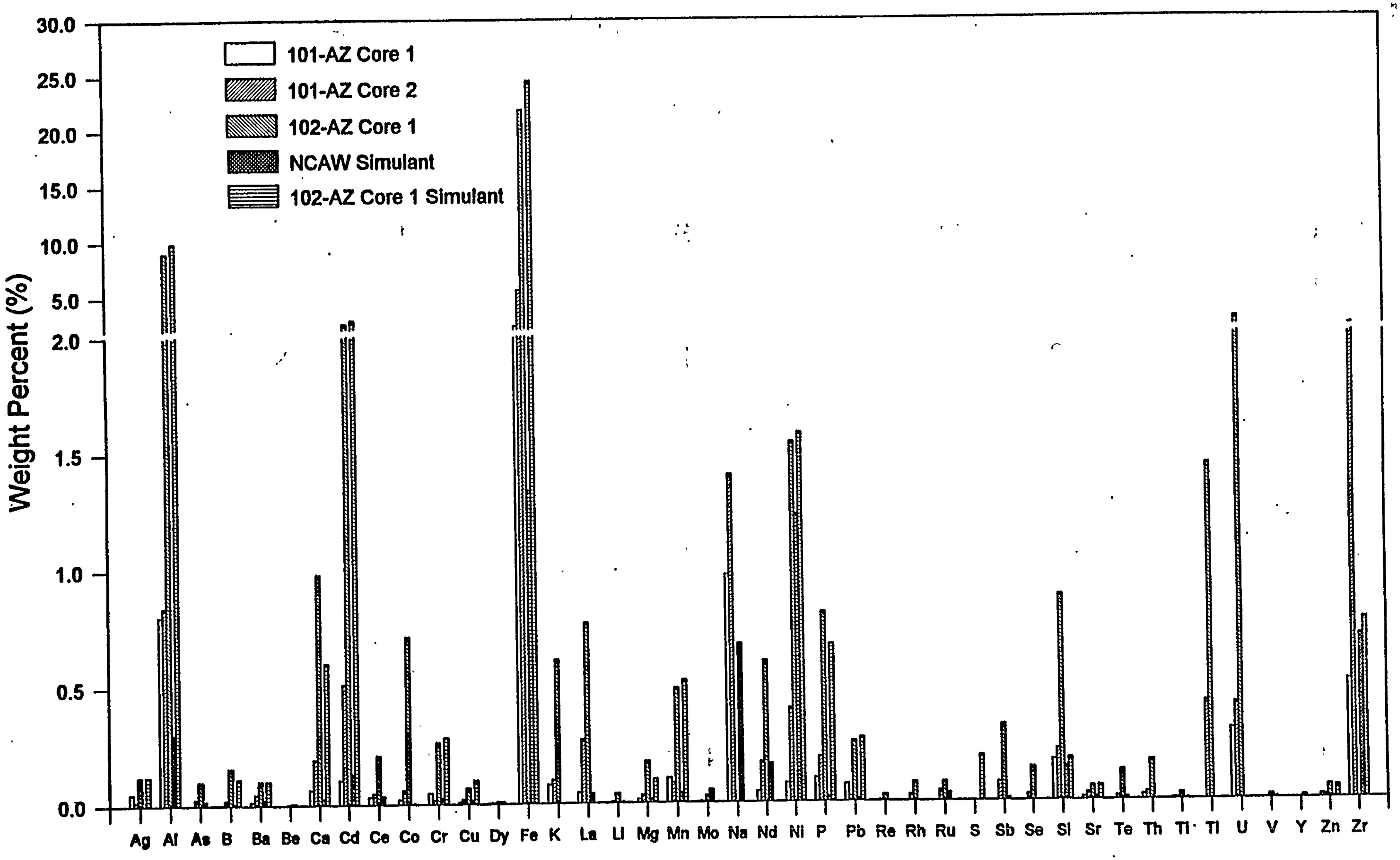


Figure 4.1. Chemical Composition of Washed Solids

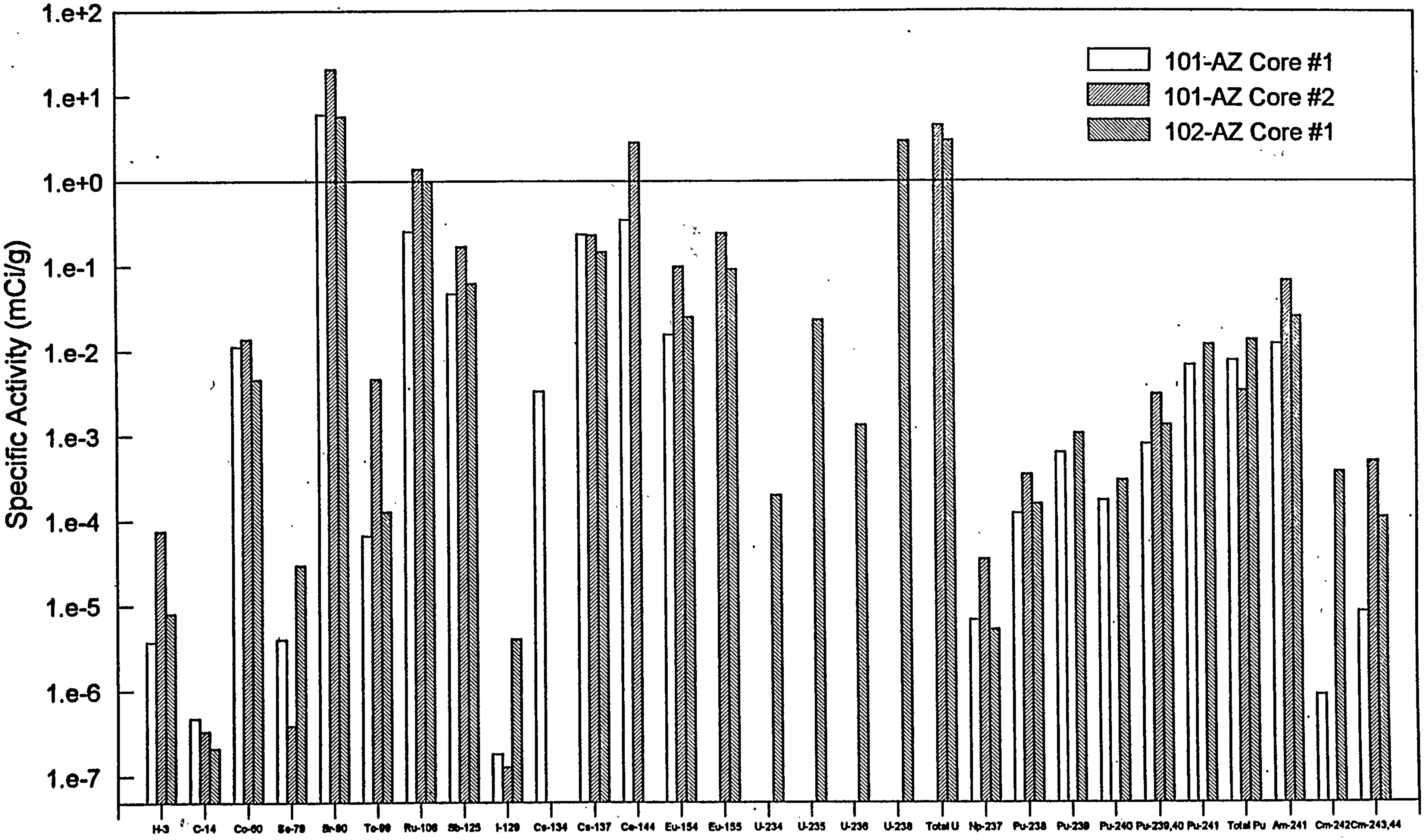


Figure 4.2. Specific Activity of Radionuclides for Washed Solids

Table 4.2. Washed-Solids Slurries Physical Properties

	101-AZ Core 1	101-AZ Core 2	102-AZ Core 1	NCAW Simulant	102-AZ Core 1 Simulant
Density (g/ml):					
Slurry	1.04	1.14	1.11	1.11	1.14
Centrifuged Solids	1.40	1.53	1.52		
Centrifuged	0.98	0.98	0.99		

converted to other concentration units. The composition based on dry weight can be calculated from the weight-fraction solids reported in Appendix C, Tables C.1 through C.5.

During the formating process, carbonate, nitrate, and nitrite may react to produce gas and the concentrations of these anions in the sample will decrease. The data from these samples indicate that reductions in the carbonate, nitrate, and nitrite concentrations are observed. In Table 4.3, the concentrations of the anions in the washed-solids slurries, melter feed, and formated slurries of the three NCAW core samples and the NCAW simulant are compared. Note that elevated values of Cl^-, and F^- were measured in the formated slurry and melter feed of 101-AZ Cores 1 and 2. Interferences, possibly from formic acid, were reported by the analyst.

During the formating of the washed solids from 102-AZ Core 1, the off-gases generated during this process were monitored and compared with similar studies using simulants. These results were reported previously (Langowski et al. 1993). A few simplified reaction mechanisms to account for a majority of the measured off-gases have been hypothesized(a). These hypothesized reactions are as follows:

$CO_3^{2-} + 2H^+ \text{-->} CO_2 + H_2O$(4.1)
$2NaNO_2 + 4HCOOH \text{-->} N_2O + 2CO_2 + 2NaCOOH + 3H_2O$(4.2)
$3HNO_2 \text{-->} H^+ + NO_3^- + 2NO + H_2O$(4.3)
$HCOOH \text{-->} H_2 + CO_2$ (Rh catalyzed)(4.4)
$NaNO_3 + 5HCOOH \text{-->} NH_3 + 4CO_2 + NaCOOH + 3H_2O$(4.5)
$2NaNO_3 + 5HCOOH \text{-->} 2NO + 3CO_2 + 2NaCOOH + 4H_2O$(4.6)

The carbonate-destruction reaction (equation 4.1) is the first phase of the reactions, and is represented by the first large CO_2 peak observed at the beginning of the formating procedure. The generation of CO_2 is followed by the production of N_2O and NO_x, as was postulated in equations 4.2 and 4.3. Hydrogen generation was observed during the digestion and recycle steps after the N_2O and NO_x concentrations had declined significantly. The ratio of N_2O/NO_x and the limited amount of H_2 observed for this sample is indicative of a feed with limited amounts of noble metals, or reduced effectiveness of the noble metals as catalysts for these reactions in this feed (Langowski, 1993).

The results indicate that slurry chemistry and off-gas generation reactions are similar between the core sample and simulants.

Table 4.3 Anion Concentrations of the Washed-Solids Slurries and Formated Slurries

	Washed Solids					Formated Slurry				Melter Feed	
	101-AZ Core 1 (g/100g Oxide)	101-AZ Core 2 (g/100g Oxide)	102-AZ Core 1 (g/100g Oxide)	NCAW Simulant (g/100g Oxide)	102-AZ Core 1 Simulant (g/100g Oxide)	101-AZ Core 1 (g/100g Oxide)	101-AZ Core 2 (g/100g Oxide)	102-AZ Core 1 (g/100g Oxide)	NCAW Simulant (g/100g Oxide)	101-AZ Core 1 (g/100g Oxide)	NCAW Simulant (g/100g Oxide)
NO_2^-	6.09	3.68	1.89	0.39	8.75	1.45	2.4	0.65	<0.31	0.62	<0.11
NO_3^-	6.24	1.87	0.69	0.4	NM	5.90	0.77	1.28	3.68	1.46	1.08
F^-	0.20	0.16	0.07	0.009	NM	6.90	1.42	<0.9	0.19	2.29	0.04
Cl^-	0.05	0.02	0.02	<0.019	0.046	7.27	5.88	<0.2	<0.12	2.29	<0.04
SO_4^{-2}	1.61	0.9	0.64		0.57	1.27	1.001	0.10		0.47	
PO_4^{-3}	0.24	0.19	0.19		2.05	<.36	<0.01	<.04		<0.01	
TIC	1.97	0.46	2.78	0.138	NM	<0.0008	0.85	0.05	4.26	0.09	0.86
TOC	9.70	0.25	0.98	0.011	0.86	0.06	3.38	8.46	1.12	2.06	0.31
TC	11.67	0.72	2.78	0.149	NM	0.06	4.25	8.53	5.38	2.13	1.17

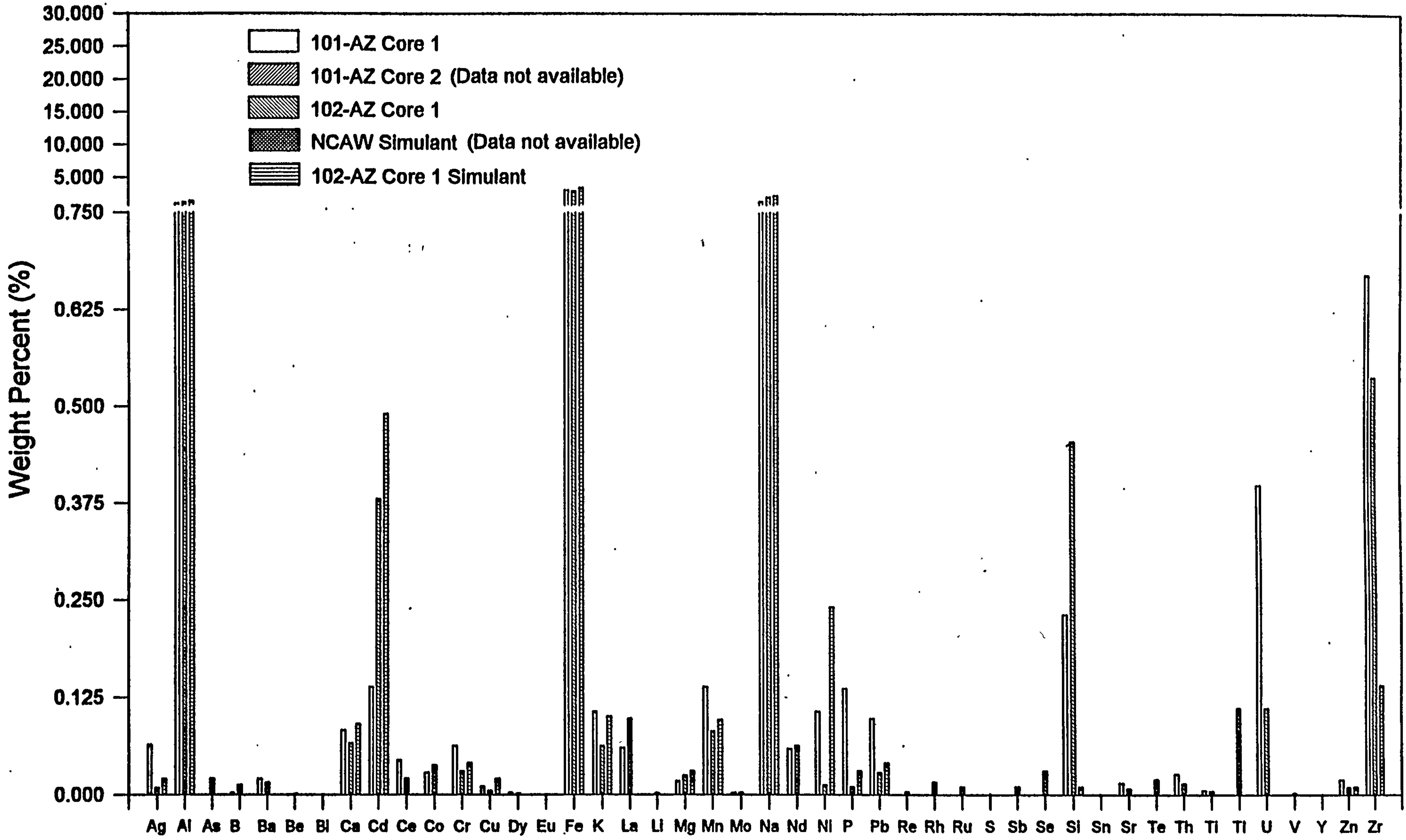


Figure 4.3. Chemical Composition of Formated Slurry

Table 4.4. Formated Slurries Physical Properties

	101-AZ Core 1	101-AZ Core 2	102-AZ Core 1	NCAW Simulant	102-AZ Core 1 Simulant
Density (g/ml):					
Slurry	1.10	1.19	1.12	1.11	1.14
Centrifuged Solids	1.44	1.50	1.65	1.27	1.34
Centrifuged Liquid	1.04	1.01	1.04	1.01	1.04
Weight Fraction:					
Solids	0.159	0.122	0.186	0.192	0.222
Oxides	0.108	0.092	0.131	0.145	0.129
g Total Oxide/L	118	101	147	161	147
Settled Solids (vol%)	57	37	38	87	84
Centrifuged Solids:					
(vol%)	21	19	18	40	42
(wt%)	28	25	26	46	48

4.2.2 Physical Characterization

Physical properties for the three actual waste samples and the two simulants are reported in Appendix C. Tables C.1 through C.5 present a summary of the physical properties measured in these samples. A comparison of the physical properties of the core sample and simulant washed-solids slurries is given in Table 4.4.

The density of the formated slurries from the core samples and simulants ranged from 1.10 to 1.19 g/ml. In Figure 4.4, the density of the NCAW simulant and actual waste-formated slurries are plotted as a function of solids concentration. The curve fit on this data indicates that there is a correlation between the density of the formated slurries and solids concentration, and that simulants are representative of actual waste with respect to this correlation. The weight-fraction total solids of the formated slurries reported in Table 4.4 varied from 0.122 to 0.222. The density of the centrifuged liquid (1.03 ± 0.02) was similar for all of the formated slurries and was comparable to the density of water, but the centrifuged solids density was significantly lower in the simulants than in the core samples. The average centrifuge solids density for the three core samples was 1.53 ± 0.11, and the simulants had an average centrifuged solids density of 1.31 with a reproducibility of 5%.

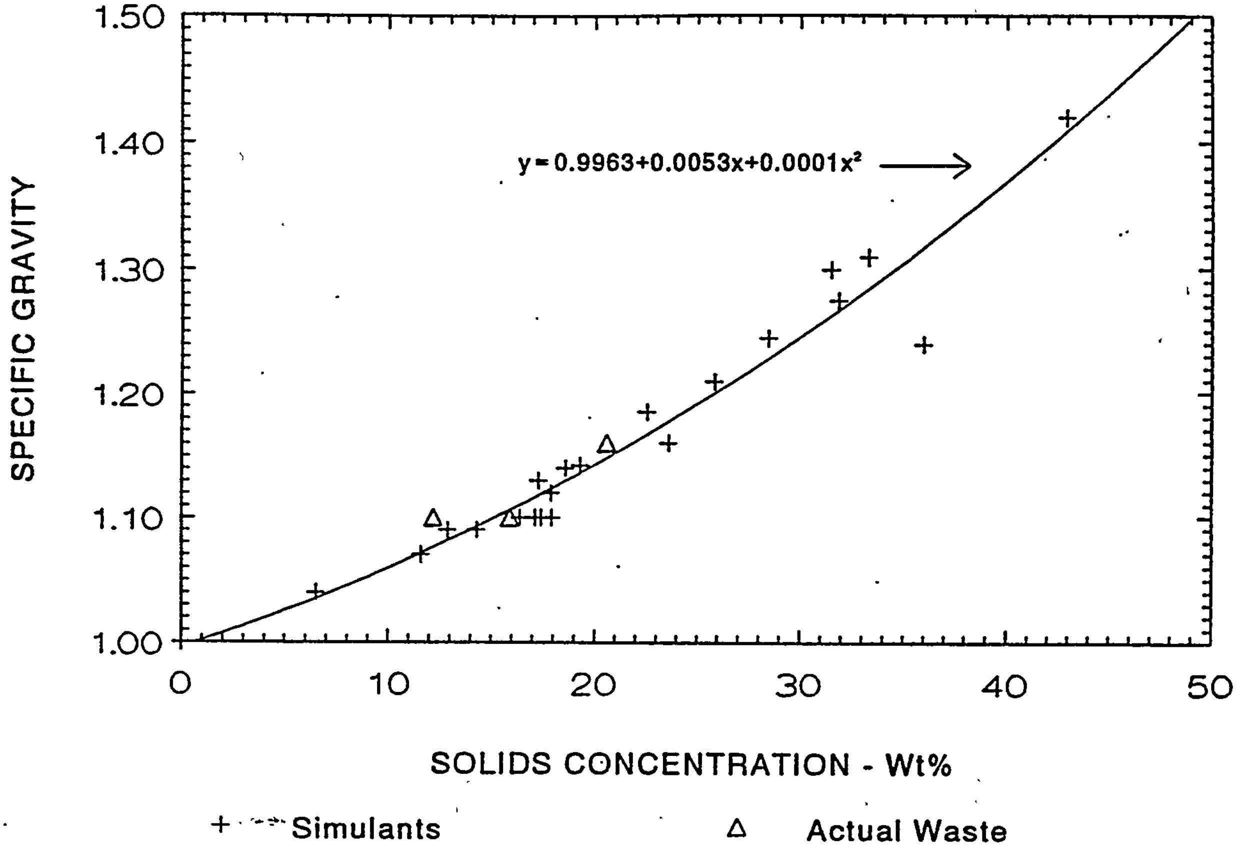


Figure 4.4. Density of NCAW Simulants and Core Samples as a Function of wt% Solids

The oxide concentration in the formated slurries of the three core samples ranged from 101 to 147 grams of total oxide/L. The oxide concentration of the NCAW simulant (161 grams of total oxide/L) was significantly higher than the core samples, but the oxide concentration of the 102-AZ Core 1 simulant was less than 2% lower than the 102-AZ Core 1 sample. The density, weight-fraction solids, and oxide concentration of all of the samples were within the range for the previously planned HWVP, except for the oxide concentration of the NCAW simulant and the density of the 101-AZ Core 2 sample.

The settling behavior of the formated slurries is shown in Figure 4.5. The settling behavior of the simulants did not match the behavior of the core samples. This is also observed in the vol% settled solids reported in Table 4.4. The core samples settled much faster and achieved a significantly higher settled-solids packing than was observed in the simulants. The majority of the settling of the core samples occurs in the initial 10 hours of settling. The settling behavior of 101-AZ Core 1 was slower than was observed in 101-AZ Core 2 and 102-AZ Core 1; the final vol% settled solids for 101-AZ Core 1 was higher than was observed for the other two core samples. The initial settling rates (average settling rate from 0 to 1 h) and the curve fits for the settling rate curves are shown in Table 4.5.

Figure 4.5. Formated Slurries Settling Rate Behavior

	Amount Settled (Curve Fit)	Average Settling Rate from T=0 to T=1 (cm/h)
101-AZ Core 1	$y = 3.07\ e^{-0.25t} + 3.89 - 2.54t\ e^{-1.8t}$	1.1
101-AZ Core 2	$y = 4.4\ e^{-0.9t} + 2.63 + 0.35t\ e^{-0.5t}$	2.4
102-AZ Core 1	$y = 4.37\ e^{0.4t} + 2.66$	1.4
NCAW Simulant	$y = 0.89\ e^{-0.06t} + 6.05$	0.05
102-AZ Core 1 Simulant	$y = 1.18\ e^{-0.05t} + 5.9$	0.06

The particle-size distribution of the formated slurries, based on volume distribution and population distribution, are reported in Appendix C. The majority of the particles were less than 5 μm in diameter; a significant number of the particles were less than 2 μm in diameter. There were a significant number of particles larger than 5 μm, which tends to skew the volume distribution toward higher particle diameters. This was most apparent in 102-AZ Core 1 and the NCAW simulant. The largest particle diameter observed in the core samples was between 100 and 110 μm.

With few exceptions, when the slurry concentrations fell within the previously planned HWVP design range, the other physical and rheological properties were also within that same design range. The properties of the actual waste samples are compared to those of simulants and simulant correlations to confirm the validity of using simulants to support design.

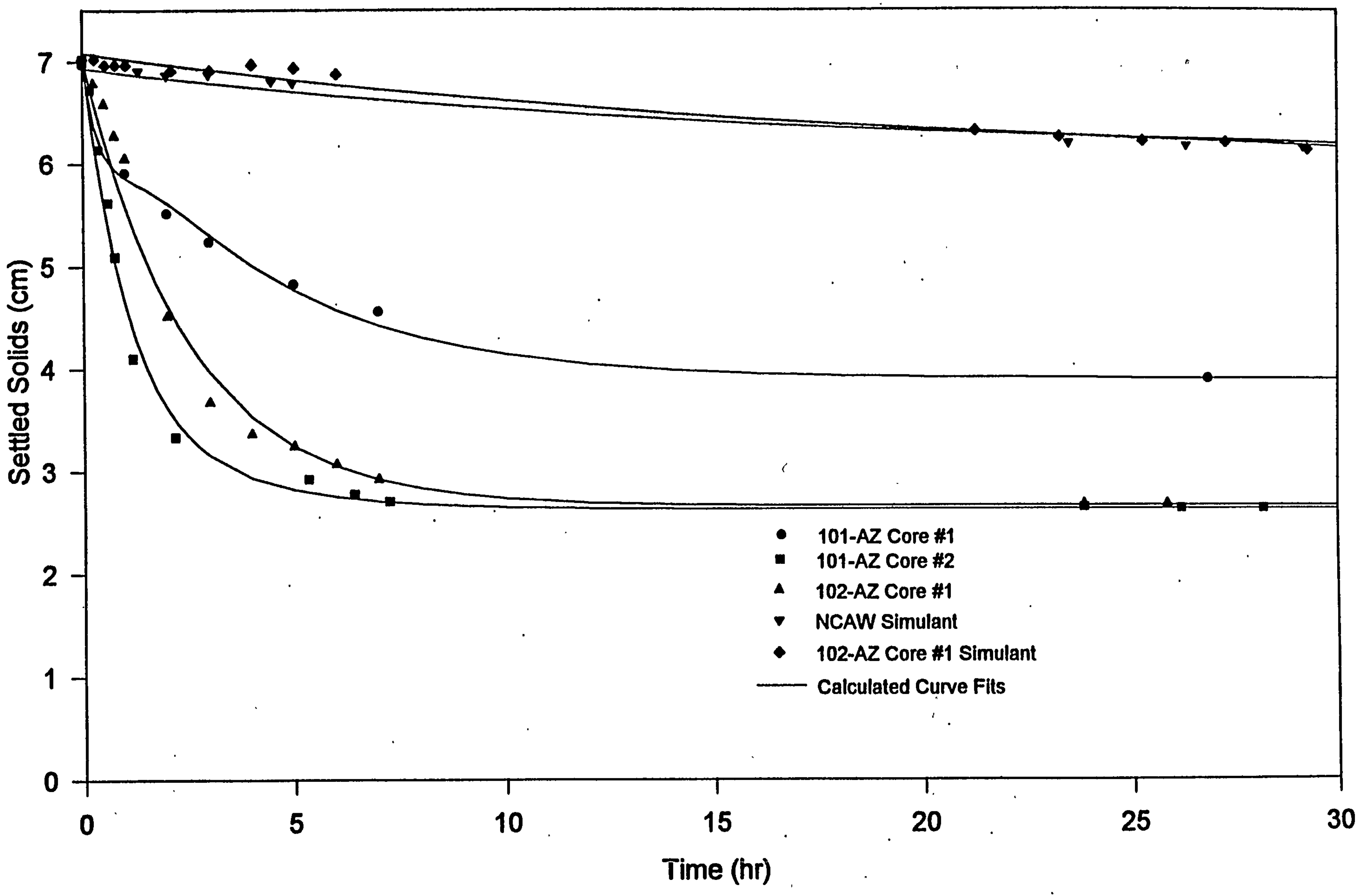


Figure 4.5. Settling Behavior of Formated Slurries

4.2.3 Rheological Characterization

Rheological properties for the three actual waste samples and the two simulants are reported in Appendix D, Tables D.1 through D.5 present a summary of the rheological properties measured in these samples. All of the samples exhibited yield pseudoplastic behavior.

These results and comparable simulant results from full-scale testing are summarized in Table 4.6 and Figure 4.7 below. Unless otherwise noted, references to simulants in the following discussion refer to laboratory-scale simulants tested in this study and not the full-scale simulants reported in Table 4.6. The flow behavior of the radioactive and simulant samples was best represented as yield pseudoplastic, as evidenced by the R^2 values. Both radioactive and simulant samples were slightly shear-thinning (i.e., viscosities decreased with increasing shear rate). A small degree of hysteresis occurred in each of the three core samples and the NCAW simulant (see Appendix D, Figures D.1, D.11, D.21, D.31, and D.41), indicating some degree of agglomeration. Yield stresses for the radioactive samples ranged from 0.085 Pa to 0.23 Pa compared to simulant yield stresses of 1.2 Pa. Yield stresses of the radioactive samples are low enough that they could be an artifact of the equipment; that is, the shear stresses at the low shear rates were at or below detection for the sensors. Apparent viscosities of the core samples at $50s^{-1}$ ranged from 3.8 cP to 7.2 cP compared to the simulant viscosities of 32 cP. The rheology of both the processed waste samples and the simulants were well below the design limits for the prior-planned HWVP plant (see Figure 4.6). Note that rheological data from 102-AZ Core 1 is suspect, because the washed solids from this core samples were inadvertently dried (see Section 3.4.2 for details).

Table 4.6. Rheological and Physical Properties of Formated Slurry

Property	101-AZ Core 1	101-AZ Core 2	102-AZ Core 1	NCAW Simulant	102-AZ Core 1 Simulant	Full-Scale NCAW Simulants[a]	
						w/o Recycle	w/Recycle
Equation (Yield Pseudoplastic)[b]	$\tau = .0558 + 9.25E^{-3}\gamma^{.829}$	$\tau = .036 + .043\gamma^{.518}$	$\tau = .0651 + 3.63E^{-3}\gamma^{.905}$	$\tau = .350 + .232\gamma^{.431}$	$\tau = .9128 + .0414\gamma^{.726}$	--	--
R^2	.8544	.9862	.9887	.9761	.9987	--	--
Yield Stress, Pa (Bingham Plastic)	.164	.232	.085	1.20	1.21	1.19	1.05
R^2	.8528	.9388	.9874	.9141	.9889	--	--
Apparent Viscosity cP							
50 s^{-1}	5.9	7.2	3.8	32.1	32.4	30.8	29.7
100 s^{-1}	4.8	5.0	3.0	20.4	20.8	18.5	16.9
250 s^{-1}	3.8	3.2	2.4	11.4	12.8	12.9	12.0
450 s^{-1}	3.4	2.3	2.2	8.0	9.8	12.1	11.5
Physical Properties							
Total Solids, wt%	15.9	12.2	18.6	19.2	22.2	19.6	20.5
Total Oxides, wt%	10.8	9.2	13.1	14.5	12.9	13.5	13.9
Total Oxides, gWO/L	118	101	147	161	147	154	158
Slurry Density, g/ml	1.10	1.10	1.12	1.11	1.14	1.14	1.14
pH	5.3	--	5.0	6.9	5.3	6.0	6.1
Mean Particle Size μm (volume dens.)	9.0	5.4	47.4	25.6	--	--	--
Mean Particle Size μm (number dens.)	1.27	1.42	1.14	1.30	--	--	--
Vol% Settled Solids	56.8	37.4	38.0	86.8	84.4	--	--
Centrifuged Solids Density g/mL	1.44	1:50	1.65	1.27	1.34	--	--
Initial Settling Rate, cm/h[c]	1.1	2.4	1.4	0.05	0.06	0.29	0.29

(a) Beckette, M. R., L. K. Jagoda. 1994. *Detailed Design Data Package NCAW Waste Simulant Properties (SIPT 2/LFCM 8 Campaign).* PHTD-K1017, Rev.0. Pacific Northwest Laboratories, Richland, Washington.

(b) See Appendix F.1 for definitions.

(c) Initial settling rates are average settling rates from time 0 to 1 h.

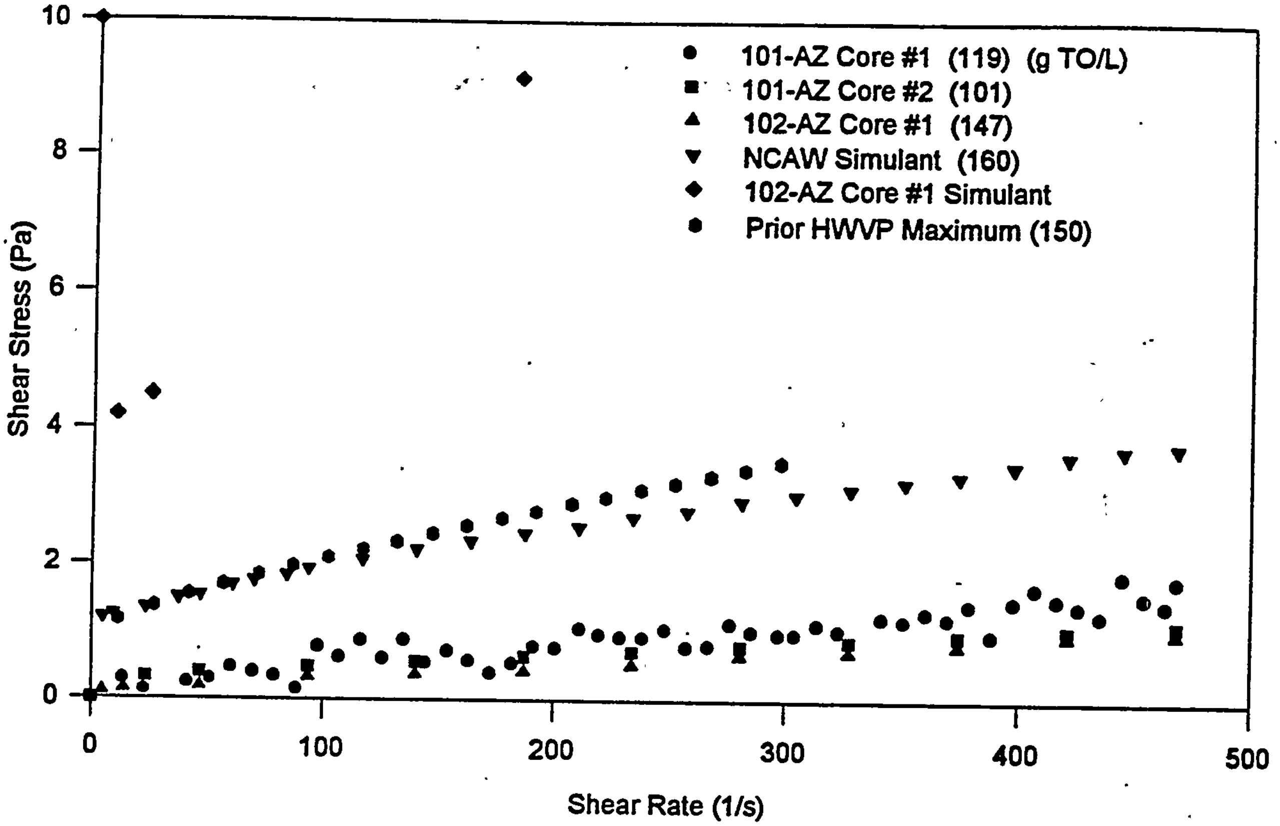


Figure 4.6. Rheology of Actual and Simulated Formated Slurry[a]

A comparison of laboratory-scale to full-scale simulant results for formated slurry rheology yields essentially identical results (see Table 4.6). Rheology of the full-scale simulants was tested with a Haake MVII sensor that had a larger gap size than the Haake MVI sensor used for laboratory-scale simulants. The concentrations and pH of the full-scale simulants (154 and 158g WO/L; pH of 6.0 and 6.1) were bracketed by laboratory-scale simulant concentrations and pH (147 and 161g WO/L; 6.9 and 5.3), enabling a direct comparison of rheological results. Full-scale simulants produced yield stresses of 1.1 Pa and 1.2 Pa, compared to 1.2 Pa for the laboratory-scale simulants. Viscosities at $50s^{-1}$ for the full-scale simulants were between 30 cP and 31 cP, compared to laboratory-scale simulant viscosities of 32 cP.

Two minor differences were observed between the two sets of simulants, as follows: (1) an increased settling rate of 0.29 cm/hr was observed for full-scale simulants versus 0.05 to 0.06 cm/hr for laboratory-scale simulants; and (2) a Newtonian flow behavior for the full-scale simulants was observed in the shear-rate range > $190s^{-1}$ versus a yield-pseudoplastic flow behavior throughout the entire shear-rate range for the laboratory-scale simulants. Differences in the test methods between the two simulant sets may have contributed to these differences. The height of the settling container for

---

(a) Prior HWVP maximum values obtained from WHC-SD-HWV-DP-001, Hanford Waste Vitrification Plant Technical Data Package, Section 13, Rev. 6.

the full-scale simulant was 19 cm versus 12 cm for the laboratory-scale simulants (differences in settling tube diameters are unknown).

Differences in concentration made it difficult to compare the simulant and radioactive rheology data directly. Data from Thornton[a] shows yield stress and apparent viscosity of formated slurries to be exponetially correlated to concentration in g WO/L. Using this correlation, one would expect a 2.8-fold increase in yield stress with an increase in concentration from 101g WO/L to 160g WO/L. Similarly, one would expect a 2.3-fold increase in apparent viscosity at 183s^{-1} with a similar increase in concentration. Actual increases in yield stress and apparent viscosity between 101-AZ Core 2 at 101g WO/L and NCAW simulant at 161g WO/L were 5.2-fold and 3.8-fold, respectively. Using the Thornton correlation, the effect of concentration alone does not account for the differences in concentration observed between actual waste and simulants.

A comparison of yield stress and apparent viscosity data from the core samples and simulants to historical data is shown in Figures 4.7 and 4.8. Radioative and simulant data from this study are superimposed on figures reported by Lanning.[b] The NCAW simulant and 102-AZ Core 1 simulant fit well within the historical data and are located near the midpoint of the data range. Rheological strength of the radioactive samples was equal to or less than the entire database of simulants reported. Although the historical data may be generally compared to the radioactive samples, the best comparison was between the simulants tested in this study and the core samples. The NCAW simulant was formulated to have the same nitrite and nitrate composition as 101-AZ Core 1 and was processed in the same manner as the core sample (e.g., amount of formic acid added, digestion time). Similarly, 102-AZ Core 1 and its simulant had like compositions and were processed in the same manner.

Physical property data in Table 4.6 provides some insight into possible causes of the observed differences between radioactive and simulant rheology. Vol% settled solids and intial settling rates indicated differences in the microstructure of the radioactive and simulant samples. The radioactive samples settled to a greater extent and at a greater initial rate than did the simulant samples. The density of centrifuged solids was greater in the core samples, indicating potentially denser aggregates with less interstitial fluid.

The particle-size distribution data provided little insight into the differences between actual and simulated formated slurries. The number-density mean-particle size of the radioactive samples (1.14 μm to 1.42 μm) are similar to that of the NCAW simulant (1.30 μm). The volume-density mean-particle sizes indicated a broader distribution in the NCAW simulant and 102-AZ Core 1 than the cores from Tank 101-AZ. The large volume-density mean-particle size for 102-AZ Core 1 (47.4 μm) was consistent with anticipated agglomeration affects of drying out a slurry (see Section 3.4.2). The large volume-density mean-particle size for NCAW simulant (25.6 μm) would make

---

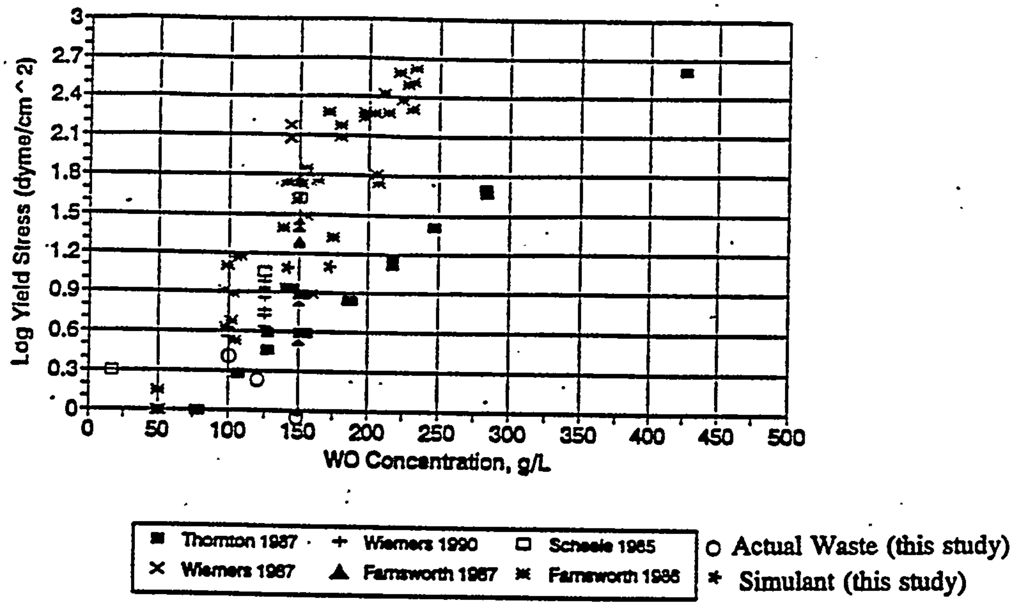
(a) Thornton, G. T., *Evaluation and Comparison of HWVP-Reference Feed Composition and Updated Neutralized Current Acid Waste Composition Simulants*. Letter Report HWVP-87-V110203C, Pacific Northwest Laboratory, 1987.

(b) Lanning, D.D., P.A. Smith, G. Terrones, D.E. Larson, *Summary of Rheological Studies Related to HWVP Slurries*, Letter Report PHTD-C93-03.02M, Pacific Northwest Laboratory, November 1993.

sense if the larger particles or agglomerates were less dense than the simulant particles. This information, as well as particle morphology, is not known and is needed to understand differences in behavior. Additionally, the method of particle-size analysis used, which included dilution and shearing of the sample, may have broken weaker agglomerates prior to measurement. The particle sizes being measured may have been agglomerates, primary particles or a combination of both not representative of the original samples. Transmission electron microscopy (TEM) is recommended for future studies to increase understanding in this area.

The effects of radiation dose on the NCAW simulant is also recommended to understand the observed rheological difference with actual waste. An analogous, although quite different, situation experienced at Savannah River Site is worth noting. The measured rheological properties of the actual potassium tetraphenylborate (KTPB) slurries yielded much lower values than those predicted by nonradioactive systems, prompting irradiation studies on the simulant.(a) After gamma irradiation of 5 x 10^7 rad, the yield stress went to essentially zero, and the consistency decreased by a factor of six or more. The irradiation also increased settling rates and final maximum wt% solids upon settling. Microscopic examination indicated that de-agglomeration of the sludge resulted from irradiation. As

Figure 4.7. Comparison of Formated Slurry Yield Stress with Historical Data(b)



(a) Ebra, M. A. 1985. *Technical Report: The Effects of Gamma Irradiation on the Rheology of KTPB Slurries*, DPST-85-926, Dupont Savannah River Laboratory.

(b) Lanning, D.D., P.A. Smith, G. Terrones, D.E. Larson, *Summary of Rheological Studies Related to HWVP Slurries*, Letter Report PHTD-C93-03.02M, Pacific Northwest Laboratory, November 1993.

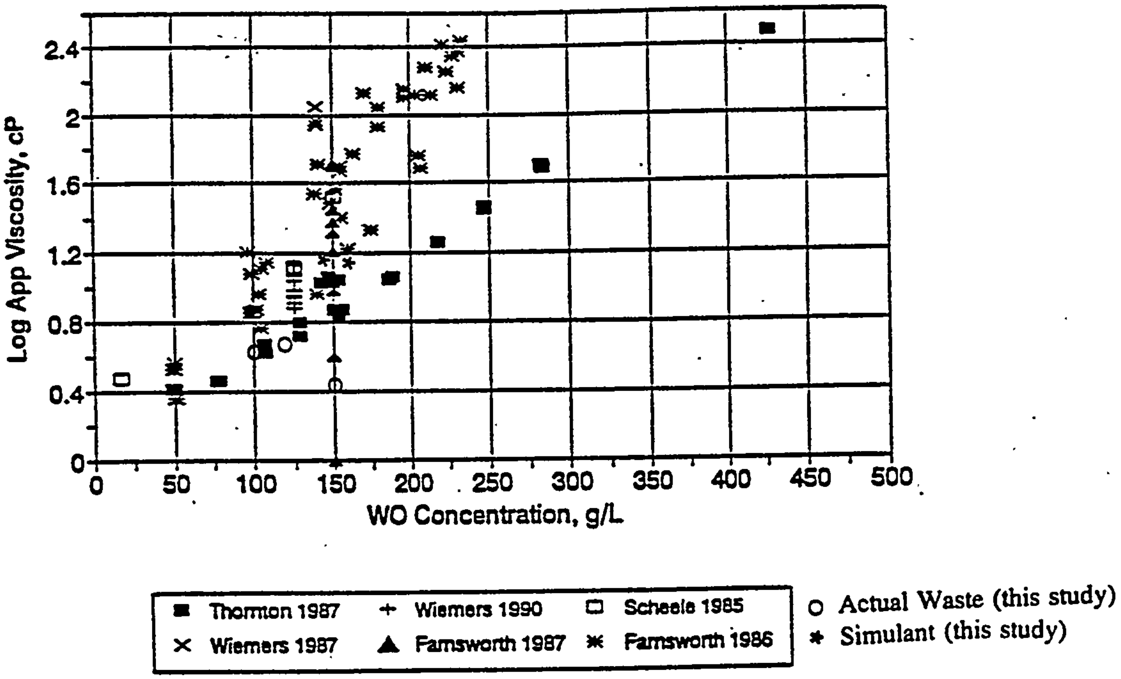


Figure 4.8. Comparison of Formated Slurry Viscosity at 183s^{-1} with Historical Data[8]

a result of the study, requirements for the slurry transfer pumps were reduced. Although the mechanism for de-agglomeration was not determined, it could very possibly be related to the organic nature of the precipitate, a condition not relevant to NCAW. However, transformations at the interfaces of NCAW particles caused by radiation dose could feasibly affect agglomeration. Simple irradiation tests on NCAW simulants may reveal an underlying cause of differences between actual and simulant waste rheology.

Another inherent difference in the actual and simulated waste is the duration of aging. The waste has been aged in a radioactive environment for many years. The NCAW simulant preparation has been designed to mimic the process by which the NCAW waste was generated but does not account for aging effects. A closer study of the particle morphology and chemical composition of actual waste particles using TEM should be pursued to identify differences resulting from aging or other historical differences.

4.3 Melter Feed

Glass frit was blended with the formated slurries to produce the melter feed. The glass frit was designed to yield a glass with acceptable processing and durability properties, as defined by linear correlations of properties with glass composition. These correlations, which are based on extensive vitrification testing of nonradioactive simulants, predict viscosity, electrical conductivity, and release rate of glasses as a function of composition (Hrma, 1994).

Three different frit compositions were used for these five samples. The same frit was used for both the NCAW simulant and 101-AZ Core 1. The frit blended with 101-AZ Core 2 was designed specifically for the core sample, including a modification to compensate for the potassium added to the sample when the pH probe broke during the addition of formic acid. The third frit was blended with both the core sample from tank 102-AZ and its corresponding simulant. The compositions of the glass frits blended with the three core samples and two simulants are reported in Table 4.7.

Table 4.7. Frit Compositions

	Frit Compositions (wt% oxides)		
	HW39-4 Frit 101-AZ Core 1 and NCAW Simulant	Custom Frit 101-AZ Core 2	FY91 New Frit 102-AZ Core 1 and 102-AZ Core 1 Simulant
B_2O_3	14.0	10.6	20.45
CaO	1.0	0.0	0.0
Li_2O	5.0	8.6	7.29
MgO	1.0	0.0	0.0
Na_2O	9.0	5.2	0.0
SiO_2	70.0	75.6	72.26

The chemical and radiochemical composition and physical and rheological properties of the melter feeds from the three NCAW core samples (101-AZ Core 1, 101-AZ Core 2, and 102-AZ Core 1) and two simulants (NCAW and 102-AZ Core 1 simulants) were determined and compared.

4.3.1 Chemical Composition

The chemical composition of the melter feeds from 101-AZ Core 1, 101-AZ Core 2, and the NCAW simulant is reported in Appendix A, Tables A.1, A.2, and A.4. The chemical composition of the supernatant from all three core samples and the NCAW simulant is also reported in Appendix A (Tables A.1 through A.4). The compositions of the melter feeds from 102-AZ Core 1 and 102-AZ Core 1 simulant were not measured because, based on prior samples, the solids settled so quickly that it was difficult to obtain representative subsamples. The composition of these two samples can be determined from the glass composition or the formated slurry/frit compositions; calculated values are reported in Section 4.6.

A comparison of the elemental composition of 101-AZ Core 1, 101-AZ Core 2, and the NCAW simulant is shown in Figure 4.9. Formated slurry was combined with frit to achieve melter feeds with targeted waste oxide-loadings of 25 to 28 percent; therefore, the major constituents in the melter feeds are the frit components. These major components include Si, Na, B, and Li. Other major constituents which came from the waste include Fe, K, Al, U, and Zr.

The frit components were added as the oxides; therefore, the majority of the elements in the melter feeds are as oxides. Other anions which are present in significant quantities are nitrate, nitrite, chloride, fluoride, and sulfate (see Table 4.3). The concentrations of carbonate (as measured by total inorganic carbon) and phosphate are significantly lower than the other measured anions. The majority of the carbon present in the samples is organic carbon, with the exception of the NCAW simulant. In this simulant, the concentration of carbonate is much higher than the carbonate observed in the core samples; therefore, the ratio of total organic carbon to inorganic carbon in this simulant does not represent that which is observed in the core samples.

The supernatant from the melter feeds contained only three cations in significant quantities (Na, K, and Li). The major anions associated with the supernatant are similiar to those observed in the slurries with the exception of the oxides, which are not soluble.

The data are reported based on wet weight (wt%) and on the oxide weight (grams/100 grams oxide). The density and oxide-loading are also reported, so that the analyte concentrations can be converted to other concentration units. The composition based on dry weight can be calculated from the weight fraction solids reported in Appendix C, Tables C.1 through C.5.

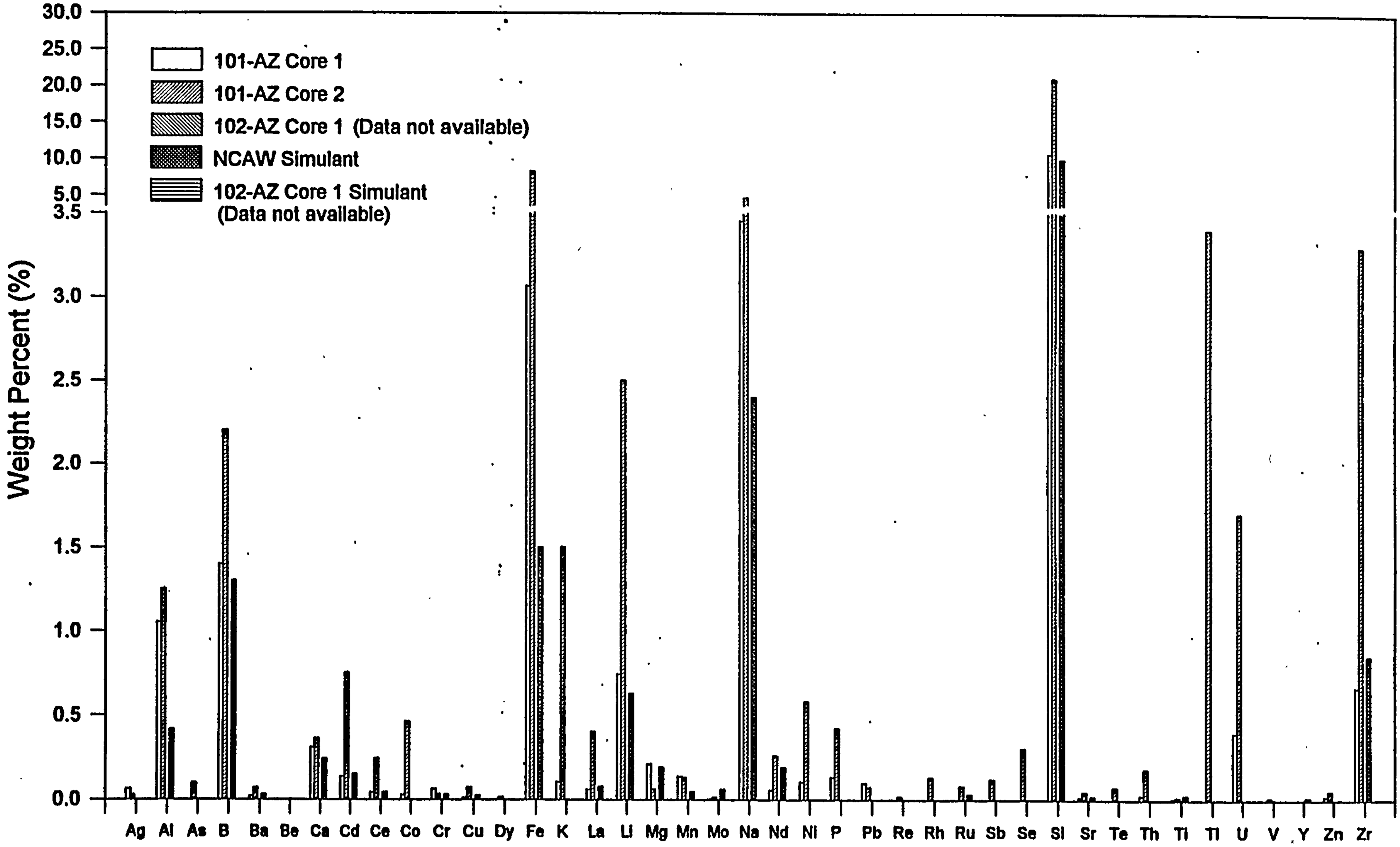


Figure 4.9. Chemical Composition of Melter Feed

4.3.2 Radiochemical Characterization

The radionuclide compositions of the melter feeds from the two core samples from tank 101-AZ are reported in Appendix B, Tables B.1 and B.2. The major radionuclides present in these samples are ^{137}Cs, ^{90}Sr, ^{144}Ce, ^{106}Ru, ^{154}Eu, ^{155}Eu, ^{125}Sb, and ^{241}Am. Both core samples are transuranic (they contain > 100 nCi/g transuranic isotopes) as is indicated by the activity of americium in the melter feeds. Only limited radiochemical analysis was performed on the melter feeds, and no data are available on the activity of Pu in these samples. A comparison of the specific activity of the measured radionuclides in these two samples is given in Figure 4.10.

The only radionuclide which may have been affected by the formating steps was ^{129}I. The data are not definitive, but it appears that some of the iodine may have been lost during the formating process.

The activity of the melter feed supernatant is due primarily to ^{137}Cs. None of the supernatants appear to be transuranic, since ^{241}Am was not observed in the gamma energy analysis (GEA).

The simulants were prepared as chemical simulants only and did not contain any radionuclides. The absence of radionuclides in the simulants eliminated many of the regulations and shielding that are necessary to handle radioactive samples, and allowed the simulant studies to be performed in a more timely and cost-efficient manner. The radionuclide activities in the melter feed from 102-AZ Core 1 were not analyzed because, based on prior samples, the solids settled so quickly that it was difficult to obtain representative subsamples.

4.3.3 Physical Characterization

Physical properties for the three actual waste samples and the two simulants are reported in Appendix C. Tables C.1 through C.5 present a summary of the physical properties measured in these samples. A comparison of the physical properties of the core sample and simulant washed-solids slurries is given in Table 4.8.

The density of the centrifuged liquid (1.04 ± 0.03) is similar for all of the melter feeds and formated slurries, and is comparable to the density of water. The centrifuged solids density in the melter feeds from the core samples are also similiar, but the density of the centrifuged solids and slurry from the NCAW simulant was significantly lower than measured in the core samples. The behavior of the centrifuged 102-AZ Core 1 simulant was not measured because of difficulties in obtaining a representative sample. The average centrifuge solids density for the three core samples is 1.69 ± 0.05 g/ml; the NCAW simulant has a centrifuged solids density of 1.50 g/ml.

The oxide concentration in the melter feeds of the three core samples ranged from 438 to 600 grams of total oxide/L. The weight fraction ratio of oxides to solids in the melter feeds is much higher than that which was observed in the previous samples, because of the introduction of the frit to the sample. The density, weight-fraction solids, and oxide concentration of all of the samples were within the range for the previously planned HWVP, except for the slurry density of the 101-AZ Core 1 sample.

Table 4.8. Melter Feed Physical Properties

	101-AZ Core 1	101-AZ Core 2	102-AZ Core 1	NCAW Simulant	102-AZ Core 1 Simulant
Density (g/ml)					
Slurry	1.47	1.36	1.34	1.28	1.42
Centrifuged Solids	1.74	1.64	1.69	1.50	Not Analyzed
Centrifuged Liquid	1.09	1.04	1.03	1.01	Not Analyzed
Weight Fraction					
Solids	0.47	0.43	0.44	0.37	0.48
Oxides	0.41	0.40	0.38	0.34	0.40
Dissolved Solids	0.068	0.020	Not Analyzed	0.014	Not Analyzed
g Total Oxide/L	600	479	515	438	573
Settled Solids (vol%)	84	87	64	87	100
Centrifuged Solids					
(vol%)	64	55	48	58	Not Analyzed
(wt%)	74	67	60	67	Not Analyzed

The density of the melter feeds from the core samples and simulants ranged from 1.28 to 1.47 g/ml. This is a significant increase from the densities observed in the formated slurries. This increase is due to increased solids-loading caused by the addition of the frit. The weight-fraction total solids of the melter feeds reported in Table 4.8 varied from 0.37 to 0.48. Only a small portion of the total solids are water soluble, as is observed in the low dissolved-solids fraction (< 15% [7%?] of the total solids). The density and oxide concentrations also increase with increased solids-loading.

The settling behavior of the melter feeds is shown in Figure 4.11. The settling behavior of the melter feed from NCAW simulant and the 101-AZ core sample is comparable, but the settling behavior of the 102-AZ Core 1 melter feed and the corresponding simulant did not match. The initial settling rates (average settling rate from time 0 to 1 h) and the curve fits for the settling rate curves are given in Table 4.9.

The 102-AZ Core 1 simulant did not settle, as is shown in Figure 4.11 and Table 4.8. The settling behavior for this sample was measured approximately three weeks after the frit was blended with the sample. Previous studies indicated that the frit used, when blended with this simulant, tends to gel after about 2 to 3 weeks time.(a) This is consistent with the behavior observed for this simulant.

(a) Beckette, M.R. and L.K. Jagoda *NCAW Waste Simulant Properties (SIPT 2/LFCM 8 Campaign* Letter Report PHTD-K1017 Rev. 0, Pacific Northwest Laboratories, May 1994.

The 102-AZ Core 1 melter feed settled much quicker than that which was observed in the other core samples and simulants. The washed solids from this core dried prior to processing the sample. Previous results indicate that when some tank-waste samples are allowed evaporate to dryness, the settling and rheological behavior of the sample are irreversibly altered. In most cases, when the sample is dried and water is added to the sample to obtain the original water content, the sample settles much more quickly, the volume percent of settled solids decreases, and the weight percent of dissolved solids decreases significantly when compared to the sample before drying. The same frit was used in this sample as was blended with the 102-AZ Core 1 simulant, but significant gelling of this sample was not observed. The settling behavior and rheological properties of this sample were measured after two weeks of blending the frit.

The particle-size distribution of the melter feeds, based on volume distribution and population distribution, are reported in Appendix C. Based on number density, the majority of the particles are less than 10 μm in diameter. There are a significant number of particles larger than 10 μm, which tends to skew the volume distribution toward higher particle diameters. This was most apparent in 101-AZ Core 2. The largest particle diameter observed in the melter feeds was between 60 and 70 μm.

Table 4.9. Melter Feed Settling Rate Behavior

	Amount Settled (Curve Fit)	Average Settling Rate from T=0 to T=1 (cm/h)
101-AZ Core 1	$y = 1.18\ e^{-0.2t} + 6.02$	0.1
101-AZ Core 2	$y = 0.89\ e^{-0.95t} + 6.11$	0.4
102-AZ Core 1	$y = 2.51\ e^{-0.45t} + 4.57$	0.9
NCAW Simulant	$y = 0.89\ e^{-0.1t} + 6.05$	0.06
102-AZ Core 1 Simulant	$y = 7.1$	0

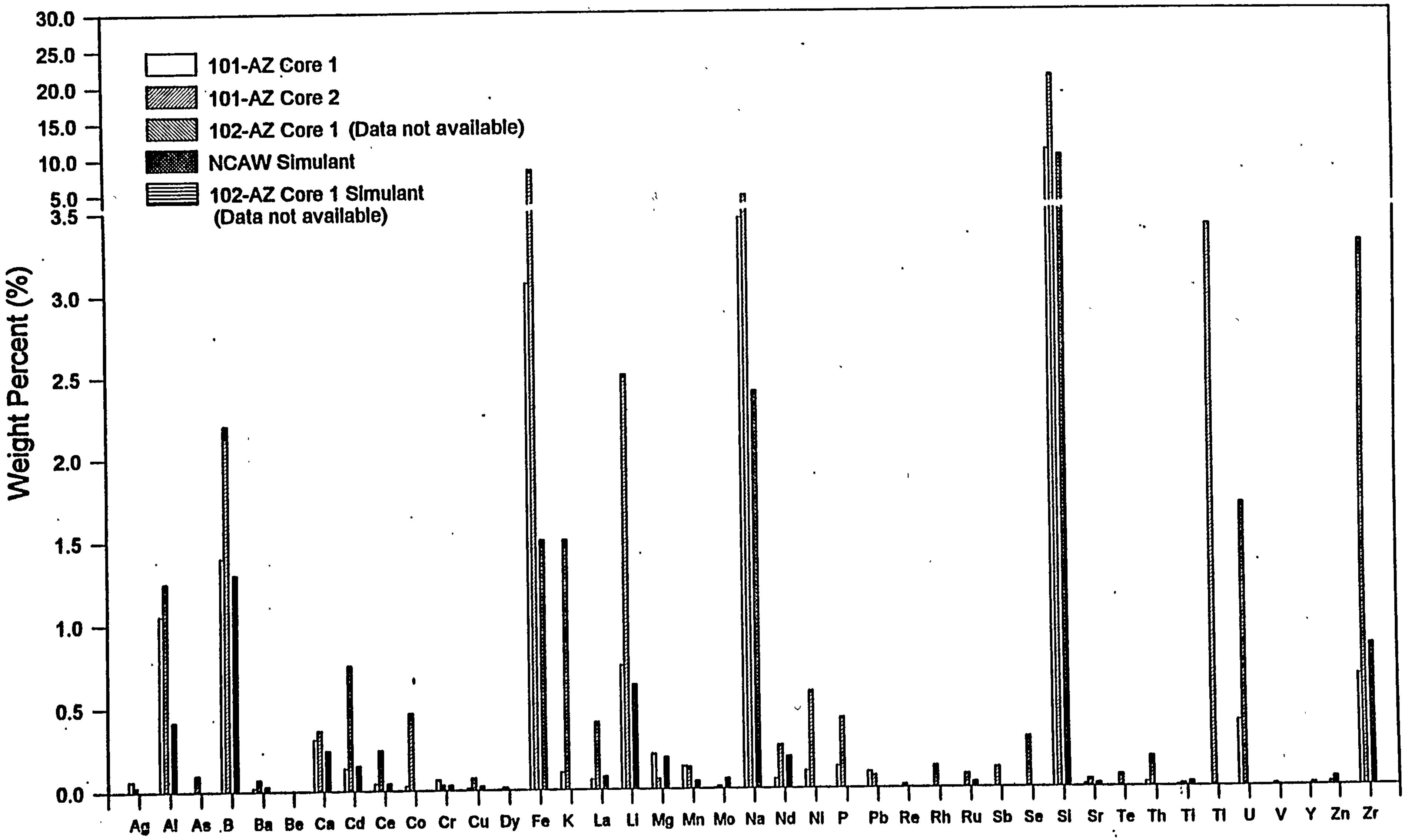


Figure 4.9. Chemical Composition of Melter Feed

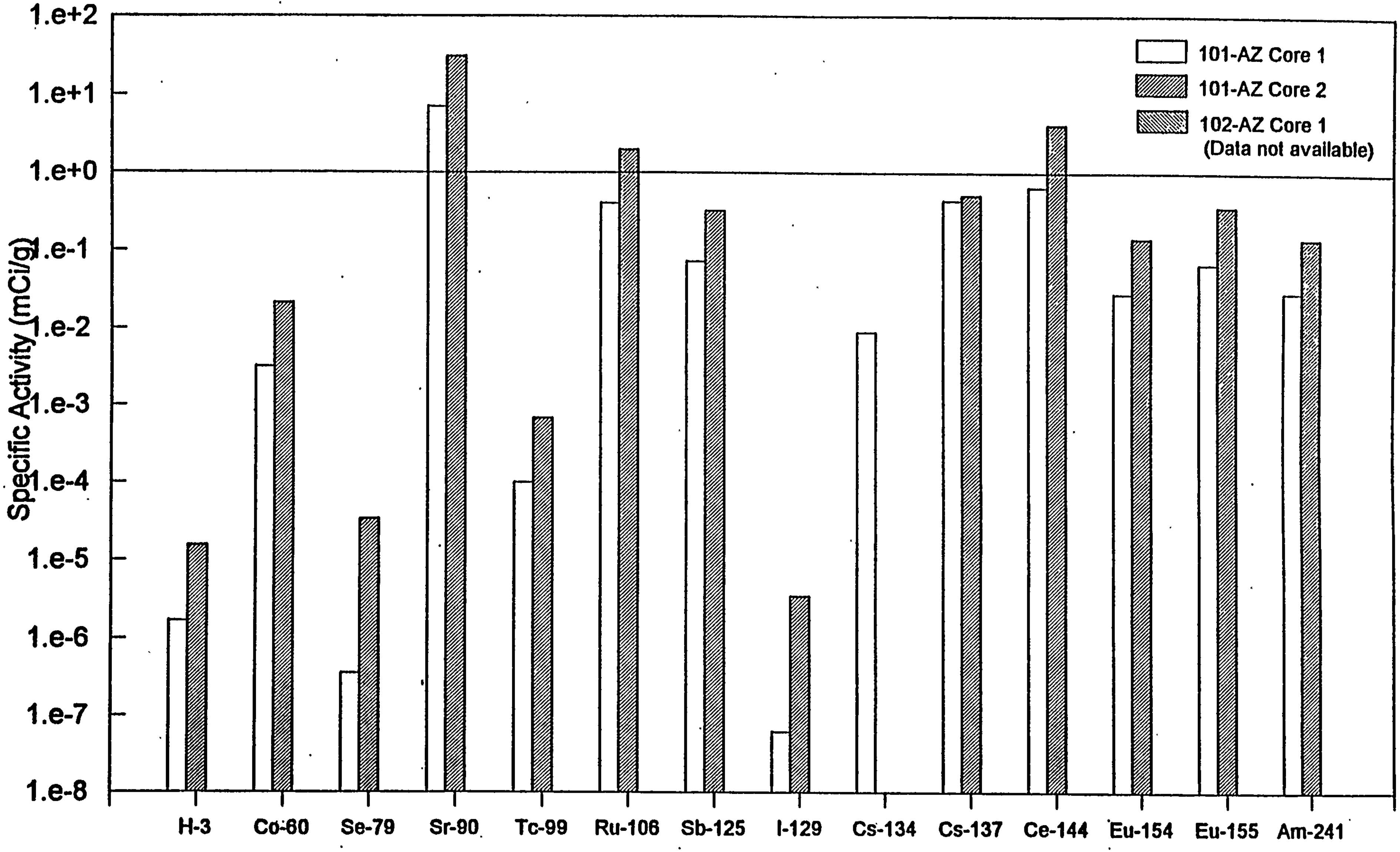


Figure 4.10. Specific Activity of Radionuclides for Melter Feed

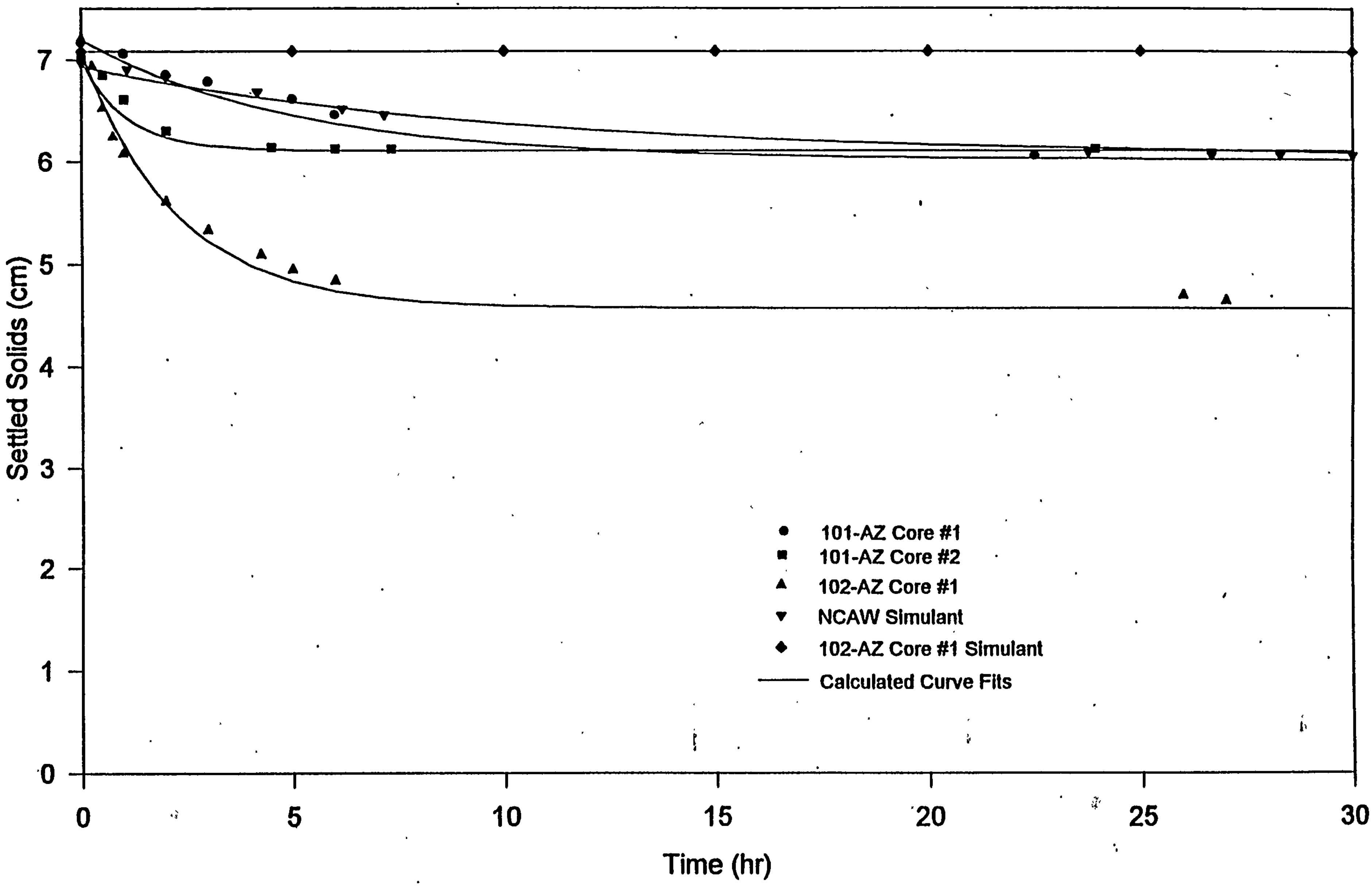


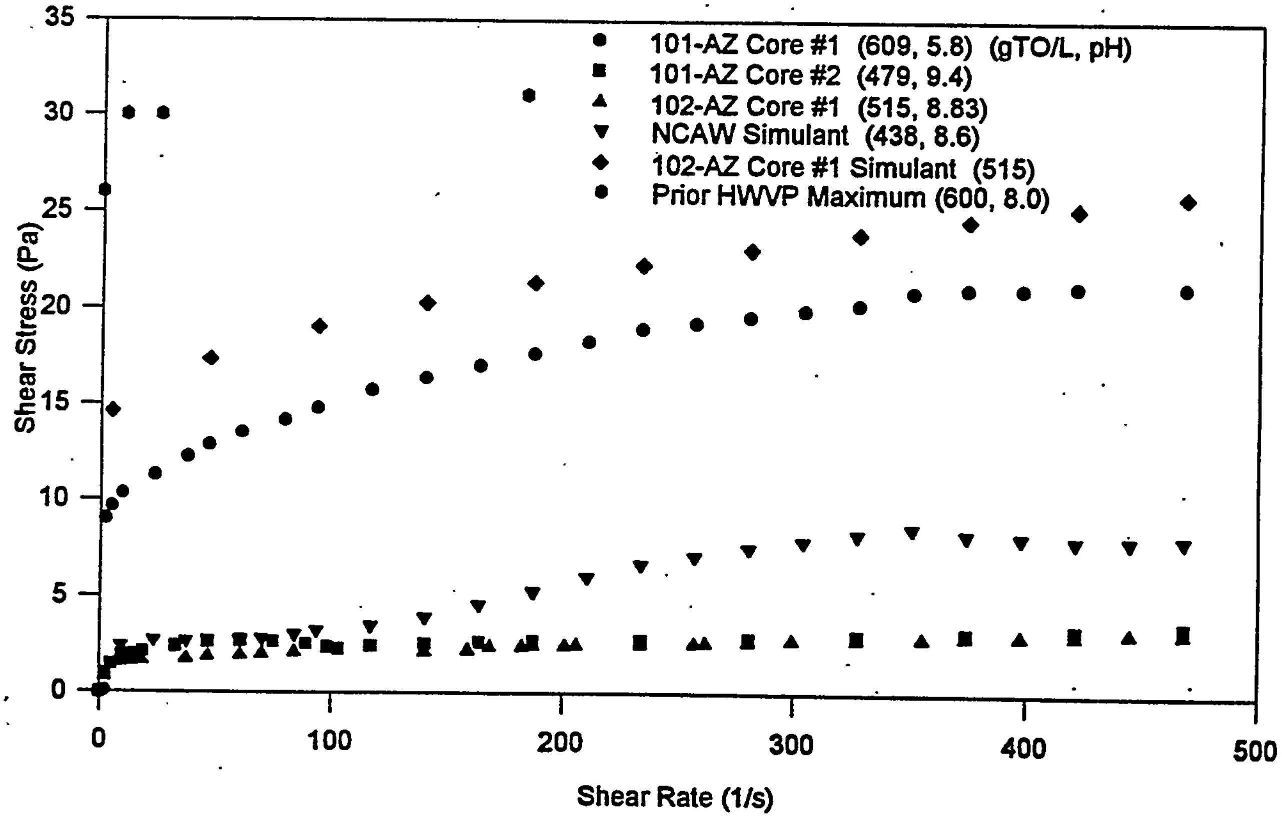
Figure 4.11. Settling Behavior of Melter Feeds

4.3.4 Rheological Characterization

Rheograms, curve fits, and corresponding physical data for the melter feeds of the three core samples and two simulants are provided in detail in Appendix D. The results are summarized in Table 4.10 and Figure 4.12 below. The radioactive and simulant samples exhibited thixotropic, yield pseudoplastic behavior. The up-curve rheograms fit well to the yield pseudoplastic model as evidenced by the R^2 values. Both radioactive and simulant samples were shear-thinning (i.e., viscosities decreased with increasing shear rate) and exhibited varying degrees of hysteresis (see Appendix D, Figures D.6, D.16, D.26, D.36, and D.46).

Yield stresses for the radioactive melter feeds ranged from 1.4 Pa to 10.3 Pa compared to simulant melter feed yield stresses of 2.2 Pa to 12.4. Viscosities of the radioactive melter feeds at $50s^{-1}$ ranged from 38 cP to 260 cP compared to the simulant viscosities of 58 cP and 365 cP. The rheology of both the radioactive and simulants melter feeds were well below the design limits for the prior-planned HWVP (see Figure 4.12). Note that rheological data from 102-AZ Core 1 was suspect, because the washed solids from this core samples were inadvertently dried (see Section 3.4.2 for details).

Figure 4.12. Rheology of Actual and Simulated Melter Feed[(a)]



(a) Prior HWVP maximum values obtained from WHC-SD-HWV-DP-001, Hanford Waste Vitrification Plant Technical Data Package, Section 13, Rev. 6.

Table 4.10. Rheological and Physical Properties of Melter Feed Slurry

Property	101-AZ Core 1	101-AZ Core 2	102-AZ Core 1	NCAW Simulant	102-AZ Core 1 Simulant
Equation (Yield Pseudoplastic)[a]	$\tau=2.86E^{-8}+5.23\gamma^{.234}$	$\tau=3.28E^{-10}+1.237\gamma^{.159}$	$\tau=.0635+.7955\gamma^{.2155}$	$\tau=1.208+.1089\gamma^{.6989}$	$\tau=.1469+10.39\gamma^{.1418}$
R^2	.9141	.9285	.9716	.9287	.9890
Apparent Yield Stress, Pa (Bingham Plastic)	10.3	1.8	1.4	2.2	12.4
R^2	.7589	.5564	.7996	.9144	.6170
Viscosity cP					
50 s^{-1}	261	46	38	58	365
100 s^{-1}	154	26	22	39	201
250 s^{-1}	76	12	11	25	92
450 s^{-1}	49	7	7	20	55
Physical Properties					
Total Solids, wt%	47.0	42.9	43.6	37.0	47.8
Total Oxides, wt%	41.0	39.9	38.4	34.1	40.4
Total Oxides, gWO/L	600	479	515	438	573
Slurry Density, g/ml	1.47	1.20	1.34	1.28	1.42
pH	5.8	9.4	8.8	8.6	--
Mean Particle Size μm (volume density)	4.3	15.3	--	--	--
Mean Particle Size μm (number density)	1.22	1.41	--	--	--
Vol% Settled Solids	84.0	87.0	62.5	87.0	100.0
Centrifuged Solids Density, g/mL	1.74	1.64	1.69	1.50	--
Initial Settling Rate, cm/h[b]	0.1	0.4	0.9	0.06	0.0

(a) See Appendix F.1 for definition.

(b) Initial settling rates are average settling rates from time 0 to 1 h.

As with the formated slurry samples, differences in concentration made it difficult to compare the simulant radioactive data directly. Comparing both the simulant and radioactive with historical simulant data provided an indication of the expected increase in rheological strength with increasing concentration, and a comparison with a wider database of melter-feed data. Yield stress and apparent viscosity data from the core samples, simulants, and historical data are provided in Figures 4.13 and 4.14. The simulant and radioactive data (with the exception of 102-AZ Core 1) fit within the rather broad spread of historical data. A comparison of the simulant and radioactive data from this study indicated that the simulants have greater yield stresses and apparent viscosities than the actual waste melter feeds.

Physical property data included in Table 4.10 provides some insight into possible causes of observed differences between radioactive and simulant rheology. Centrifuged solids density and initial settling-rate data indicate differences in the microstructure of the radioactive and simulant samples. The radioactive samples settled to an equal or greater extent and at a greater rate than did the simulant samples. The density of centrifuged solids was greater in the core samples, indicating potentially denser aggregates with less interstitial fluid.

Full-scale simulant rheological data are available but are not presented here due to differences in processing history that could be expected to make the rheological results quite different. Large-scale experience in melter feed testing has shown that frit type, temperature history, and aging can have a dramatic effect on rheology.[a] Because the full-scale melter feed was boiled following frit addition, whereas the laboratory-scale samples were maintained at room temperature, the data was not comparable.

(a) Beckette, M. R., L. K. Jagoda. 1994. *Detailed Design Data Package NCAW Waste Simulant Properties (SIPT 2/LFCM 8 Campaign).* PHTD-K1017, Rev. 0. Pacific Northwest Laboratory, Richland, Washington.

Figure 4.13. Comparison of Melter Feed Yield Stress with Historical Data(a)

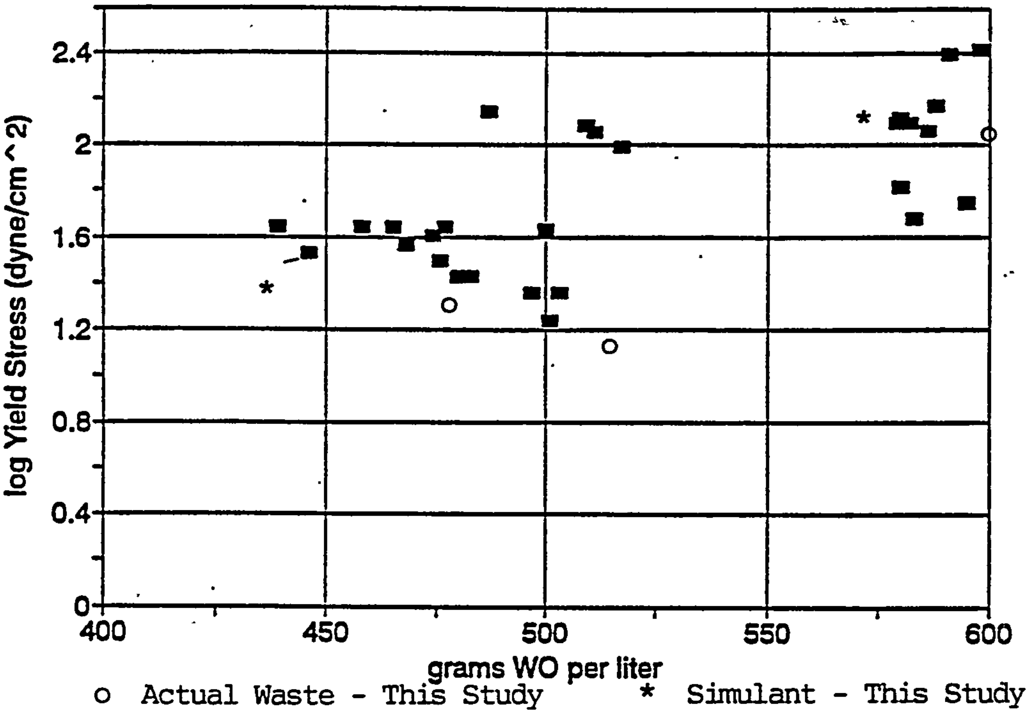
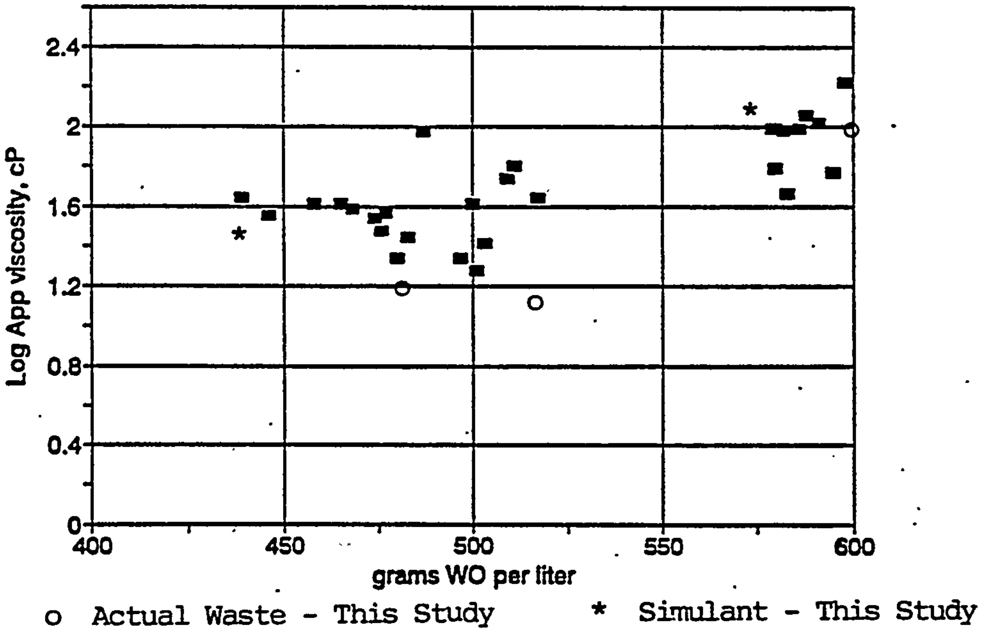


Figure 4.14. Comparison of Melter Feed Viscosity at $183 s^{-1}$ with Historical Data[10]



(a) Lanning, D.D., P.A. Smith, G. Terrones, D.E. Larson, *Summary of Rheological Studies Related to HWVP Slurries*, Letter Report PHTD-C93-03.02M, Pacific Northwest Laboratory, November 1993 (data at 4 and 6 pH from Farnsworth et al. 1986, Appendix B).

4.4 Glass Analysis

4.4.1 Chemical Characterization

Chemical compositions of the three radioactive glasses and the NCAW simulant are provided in Table 4.11. Initial analyses of 101-AZ Core 1 and 2 glasses were less than adequate, only accounting for 92% to 93% of glass (see Appendix A, Tables A.6 and A.7). Consequently, further analysis of the glasses was pursued including re-analysis using standard preparations (Na_2O_2 and KOH fusions), analysis using HF digestion preparation, and similar preparations/analyses on National Bureau of Standards (NBS) and internal standards (results are in Appendix A, Tables A.6 through A.9). Using this suite of data, more reasonable "adjusted" compositions were calculated for each glass to allow simulant glass preparation. As a result of the difficulties in these and other glass analyses, the sample preparation procedures were refined, resulting in a greatly improved analysis of 102-AZ Core 1 glass (accounted for 99.6% of the glass). The three core sample simulant glasses were batched to equal the composition of the respective core samples, but were not analyzed.

Table 4.12 compares the measured glass compositions to compositions calculated from washed solids or formated slurry measured compositions, frit compositions, and estimated waste oxide loadings in the glass. The estimated waste loadings were generated by minimizing differences between the measured and calculated compositions (major components only). After applying the estimated waste loadings, the measured and calculated compositions compared fairly well. In each case the estimated or calculated waste oxide loading was greater than targeted. The targeted waste loadings for 101-AZ Core s 1 and 2 were 25% and for 102-AZ Core 1 was 28%. The estimated loadings were 2%, 5%, and 2% greater than targeted values, respectively.

Table 4.11. Chemical Compostions of Actual and Simulant Glasses

	101-AZ Core #1 Adjusted (wt% oxide)	101-AZ Core #2 Adjusted (wt% oxide)	102-AZ Core #1 (wt% oxide)	NCAW Simulant (wt% oxide)
Ag_20	0.10^a	0.03^a	(0.016)	NA
Al_20_3	5.22^b	2.85^a	6.2	2.3
As_20_3			NA	NA
B_20_3	9.68^c	7.38^c	13	11
BaO	0.05^a	0.07^a	0.037	0.085
BeO	0.002^a	0.003^a	NA	NA
CaO	0.97^b	0.35^b	0.48	1.0
CdO	0.30^a	0.80^a	1	0.49
CeO_2	0.14^c		0.08	0.17
Co_20_3	0.10^c		NA	0.07
Cr_20_3	0.20^a	0.06^a	0.12	0.15
CuO	0.02^a	0.03^a	(0.019)	0.09
Dy_20_3	0.06^c		NA	NA
Fe_20_3	10.18^b	12.25^b	11	6.2
K_20	2.12^a	1.65^a	NA	0.7
La_20_3	0.14^a	0.46^a	0.27	0.265
Li_20	3.56^b	5.94^b	4.6	3.6
MgO	0.77^b	0.12^b	0.11	0.9
MnO_2	0.48^b	0.21^b	0.3	0.36
MoO_3	0.01^a		NA	0.24
Na_20	9.79^a	6.24^a	7.5	8.8
Nd_20_3	$0.16^{a,b}$	0.42^a	(0.17)	0.62
NiO	0.29^a	0.69^a	0.69	0.51
P_20_5	0.55^b	0.88^b	(0.55)	NA
PbO	0.20^a	0.11^a	0.11	NA
ReO_2			0.01	NA
Rh_20_3			NA	NA
$Ru0_2$		0.09^c	0.03	0.2
Sb_20_3			NA	NA
SeO_2			NA	NA
SiO_2	51.97^c	53.09^c	51	53.2
SrO	0.04^a	0.05^a	(0.022)	0.1
TeO_2		0.05^a	0.05	NA
ThO_2			NA	NA
TiO_2	0.12^a	0.08^a	0.059	0.1
Tl_20_3			NA	NA
U_30_8	0.41^d	1.71^a	0.85	NA
V_20_3			0.01	NA
Y				0.05
ZnO	0.05^b	0.02^a	0.02	0.15
ZrO_2	1.99^a	4.42^a	1.9	3.5
$S0_3$	0.40^c	0.32^c		
Total[f]	100.07	100.31	99.4	94.8

Notes:

a. Analysis by ICP-ES (Na_2O_2 and KOH fusion/dissolution) preperation methods.

b. Analysis by ICP-ES (HF digestion) preparation method.

c. Value calculated from washed solids analysis (same method as "a"). Below detection limits in glass.

d. Analysis by fluorescence (typically fluorescence would be used for U; however, the number appended would be a flier when compared to leached uranium in Core 1 and Core 2 and ICP-ES analyses in glass and washed solids.

e. Adjusted value by dividing ICP-ES (Na_2O_2, KOH) result by % yield of constituent determined by ATM-10 and NBS 688 analysis.

f NA means "Not Available"

Table 4.12. Measured Versus Calculated Glass Compositions and Waste-Oxide Loadings

	101-AZ Core #1 Glass Composition (wt% oxides)		101-AZ Core #2 Glass Composition (wt% oxides)		102-AZ Core #1 Glass Composition (wt% oxides)	
	Measured	Calculated	Measured	Calculated	Measured	Calculated
Al_2O_3	5.2	4.6	2.9	2.7	6.2	6.1
B_2O_3	9.7	10.2	7.4	7.5	13.0	13.6
CaO	1.0	1.0	0.4	0.5	0.5	0.3
Fe_2O_3	10.2	10.3	12.3	13.8	11.0	11.9
Li_2O	3.6	3.7	5.9	6.1	4.6	4.8
MgO	0.8	0.8	0.1	0.1	0.1	0.1
Na_2O	9.8	10.6	6.2	6.9	7.5	7.5
SiO_2	52.0	52.2	53.1	53.7	51.0	50.4
ZrO_2	2.0	2.1	4.4	4.8	1.9	2.0
Other	5.9	4.5	7.4	3.9	4.2	3.2
Total	100	100	100	100	100	100
Estimated Waste % Oxide Loading		27%		30%		30%

Results of glass redox as measured by Fe^{+2}/Fe^{+3} are in Table 4.13. All three radioactive samples were well within the prior-planned HWVP acceptable range; the NCAW simulant was at the lower limit. Glass redox resulting from vitrification of NCAW simulant waste has been shown to be somewhat correlated to the amount of formic acid added, initial nitrite, and initial nitrate concentrations.(a) A plot from Merz showing this relationship is provided in Figure 4.15 and includes historical simulant data combined with radioactive data from this study. The radioactive results compared well with simulant data and fall well within the range of simulant results.

4.4.2 Radiochemical Characterization

Radiochemical compositions of the actual waste glasses are provided in Table 4.14 with comparisons to the prior-planned HWVP maximum. Core sample compositions are decay-corrected to 1/1/90, whereas the design limit maximum for the HWVP assumed a plant start-up date of 1999 AD. All radionuclide concentrations were below the maximum, with the exception Co-60, Sr-90, Pu-239+240 and Cm-242. With an additional 9 years of decay, the Sr-90 concentration would decay below the maximum; but the Co-60, Pu-239+240 and Cm-242 would remain above the maximum.

4.4.3 Crystallinity

Samples of each radioactive glass were examined using X-ray diffraction (XRD) to determine the degree of crystallinity and to identify the crystalline phases, if any were present. The degree of crystallinity was low, likely under 1%. No crystalline phases were detected in the glass produced from 101-AZ Core 1. Low concentrations (≤ 1%) of crystalline phases were found in the glass made from 101-AZ Core 2. The presence of ruthenium oxide was tentatively identified, at least one unidentified phase was present. One unidentified peak of low intensity was present in glass made from 102-AZ Core 2. Based on comparative counts of the mount material (Al), a rough estimate of the crystalline concentration was calculated to be between 0.2% and 2%. The low concentrations of crystalline phases were attributed to the fact that each of the glasses was air-quenched before annealing to pass through the devitrification temperature zone (~900°C). Each of the radioactive glasses was formulated to have liquidus temperatures below 900°C.

Table 4.13. Summary of Glass Redox Results

	101-AZ Core 1	101-AZ Core 2	102-AZ Core 1	NCAW Simulant	102-AZ-C1 Simulant	Prior HWVP Acceptable Range
Fe^{+2}/Fe^{+3}	0.026	0.085	0.047	0.005	Not Meas.	0.005 to 0.30

(a) Merz, M. D. 1994. *A Summary Report on Feed Preparation Offgas and Glass Redox Data for Hanford Waste Vitrification Plant*. Letter Report PHTD-C93-03.02L. Pacific Northwest Laboratory, Richland, Washington.

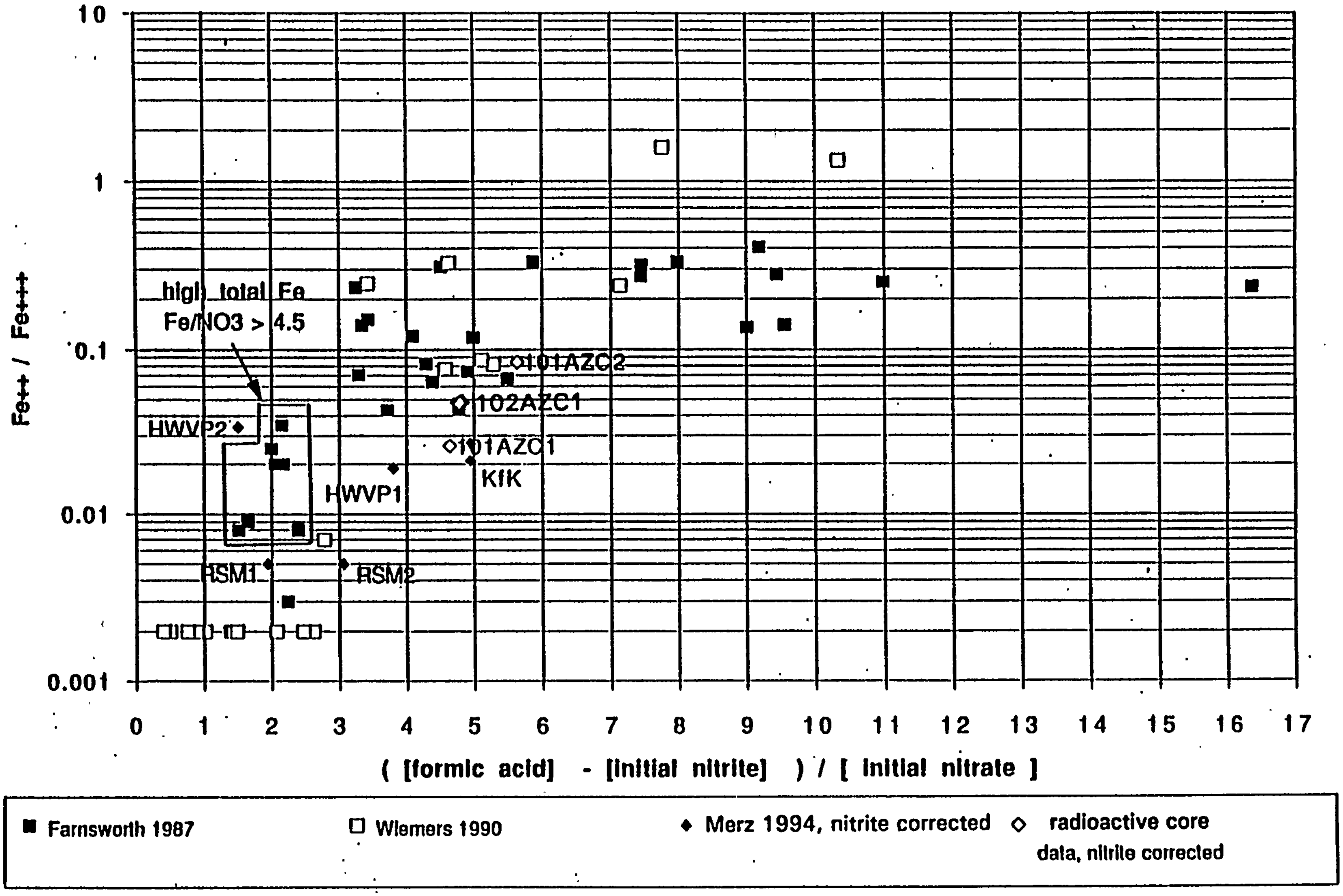


Figure 4.15. Comparison of Simulant Glass Redox to Radioactive Glass Redox

Table 4.14. Radiochemical Composition of Actual Waste Glass

Nuclide	101-AZ Core #1 (uCi/g)[(a)]	101-AZ Core #2 (uCi/g)[(a)]	102-AZ Core #1 (uCi/g)[(a)]	Prior HWVP Maximum (uCi/g)[(b)]
Co-60	7.51E+00	1.96E+01	1.03E+01	2.57E+00
Sr-90	2.03E+04	2.99E+04	1.41E+04	2.50E+04
Tc-99	3.15E-03	2.05E-02	6.73E-03	5.60E+00
Ru-106	7.89E+02	1.80E+03	2.06E+03	2.99E+03
Sb-125	1.45E+02	3.71E+02	2.61E+02	1.05E+03
I-129	< 5.00E-04	< 4.37E-04		9.75E-06
Cs-134	7.35E+00			7.21E+02
Cs-137	6.21E+02	2.81E+02	2.21E+02	3.05E+04
Ce-144	1.33E+03	3.98E+03		1.79E+04
Eu-154	5.71E+01	1.39E+02	5.17E+01	2.01E+02
Eu-155	1.31E+02	3.44E+02	1.89E+02	2.46E+02
Np-237	4.50E-02	5.68E-01		1.19E-01
Pu-238	4.54E-02	8.11E-02	2.00E-01	4.60E-01
Pu-239+240	2.40E-01	4.32E-01	1.73E+00	1.16E+00
Am-241	5.25E+01	1.05E+02	5.56E+01	3.46E+02
Cm-242	6.58E-01	2.14E+00		2.99E-01
Cm-243+244	4.61E-01	1.18E+00		-

Notes:
(a) Decay corrected to 1/1/90.
(b) Decay corrected to 1/1/2000

4.4.4 Physical Characterization

Density of the radioactive glasses was measured with a simple water-displacement test. The density of the cooled glass from 101-AZ Core 1 was 2.56 g/cc; from 101-AZ Core 2, 2.67 g/cc; and from 102-AZ Core 1, 2.54 g/cc.

4.5 Durability

Averaged normalized releases for the major elements and measurable radionuclides with estimated standard deviations and confidence intervals for tests in Table 3.2 are presented here. The methods for calculating normalized releases and the associated statistics are described in Appendix F.2. A review of this appendix section is essential to understanding the limitations of the statistics provided in this section. Model predictions for durability and model statistics are provided in Appendix A, Tables A.20 through A.23. Test leachate analytical data, less radiochemistry, are provided in Appendix A, Tables A.10 through A.19. Leachate radiochemical data for the hot-cell tests only are provided in Appendix B, Tables B.4 through B.6.

The statistics provided throughout this section, excluding model statistics, include only short-term uncertainty estimates. In effect, they represent the degree to which a given laboratory can be expected to reproduce results when using replicate specimens of a given glass under a specific set of conditions. The uncertainties accounted for include short-term analytical uncertainty in the glass, leachates, and blanks; short-term uncertainty introduced from test preparation (e.g., surface area-to-volume ratio); and short-term uncertainty introduced during testing (e.g., temperature gradient within oven, leachate-evaporative losses, test duration). The statistics account neither long-term nor inter-laboratory analytical and test uncertainties nor potential analytical biases in the glass or leachate. By design, the core samples and core-sample simulants were durability tested and analyzed at the same time and under the same conditions to allow comparison without consideration of long-term uncertainties.

Prior to generating statistics for the leach tests, the data were natural log transformed to improve the normality of the distribution and allow comparison with model results, which were generated in the same transformed basis.

4.5.1 Product Consistency Test Elemental Releases

The PCT is a standard test that compares chemical durabilities of various HLW glasses and tests product consistency to assess acceptability of production glass. As discussed in Section 1.2, the WAPS currently specifies the acceptable glass durability criteria for the HLW repository in terms of the PCT (i.e., glass must be at least as durable as EA glass as measured by PCT). Because this preliminary requirement continues to be the primary benchmark for glass acceptability, the PCT remains a critical test for all U.S. producers of HLW glass.

The PCT is a high surface area-to-volume ratio, static dissolution test that results in significant concentrations of glass components in solution. Over a seven day period, the reaction rate changes significantly as the concentration of silicic acid in solution builds up. The initial dissolution rate (or forward rate) is rapid compared to the final rate experienced in the test. Depending on the glass and duration of the test, the fully saturated dissolution rate may be achieved. This rate would be expected to continue indefinitely unless conditions were achieved where silicic acid concentration is depleted by secondary phase formation. Results from PCT cannot be directly used to predict long-term performance in repository conditions.

4.5.1.1 101-AZ Core 1 and Simulant

Results from PCT of 101-AZ Core 1 are provided in Table 4.15. The durability of the glass as measured by boron release is 0.130 g/m^2, which is approximately 50 to 100 times more durable than the EA glass limit. The Na release is comparable to B, while Li is ~70% greater and Si is ~40% less than boron. The reduced level of Si release is typical in PCT results and is due to low solubility of Si in solution. The percent relative standard deviations (RSDs) range from 2.38% for Li and 10.2% for B, which is reasonable considering the difficulties of hot-cell operations. The standard deviations are based on quadruplicate glass, triplicate leachate, and a single leachate blank analyses. Confidence intervals are based on a sample size of three leachates. The natural log-transformed normalized releases and 95% confidence intervals are used in statistical comparisons between simulant and radioactive samples and with model predictions. Exponential or retransformed data are provided to indicate approximate maximum error estimates in untransformed units. Note that the differences between the mean normalized releases and retransformed mean ln normalized releases are insignificant.

Results from PCT of 101-AZ Core 1 simulant are provided in Table 4.16. Separate results are reported for two simulant samples washed, prepared, and durability-tested in the hot cell with the radioactive samples; two simulant samples washed and prepared in the laboratory and tested in the hot cell with the radioactive samples; pooled results from the above four simulant samples; and two simulant samples tested in the laboratory. Note that elemental analysis was not performed on the simulant glass and normalized releases are calculated from the "as-batched" composition. As discussed in Appendix F.2, a conservative estimate of glass analytical variance equivalent to 5% RSD was incorporated into overall RSD and confidence interval calculations. Note also that confidence intervals are strongly influenced by the number of replicate samples. For a given standard deviation, the confidence interval for duplicate samples is three times greater than for triplicate samples and four times greater than for quadruplicate samples. Given the assumed glass analytical uncertainty and limited replicate samples, the confidence intervals for these simulant results are conservative.

The mean normalized releases for the hot-cell simulant samples tested in stainless-steel containers were essentially identical to the laboratory-tested samples in Teflon containers, with the exception of B, which was 0.200 g/m^2 in hot cell tests and 0.233 g/m^2 in laboratory tests. With the exception of B, releases of samples washed and prepared in the laboratory and tested in the hot cell were slightly higher than those tested in the laboratory; releases of samples prepared and tested in the hot cell were slightly lower than those tested in the laboratory. Hot-cell simulant samples were prepared both in the hot cell and in the laboratory to allow for a comparison of results of the two preparation locations. Preparation in this case includes weighing and transferring the glass and leachant into the leach containers, sparging the container head space with argon, sealing the containers, swirling to dislodge bubbles, and removing residual glass from the sides of the containers.

Table 4.15. PCT Leach Test Results for 101-AZ Core #1 (Hot Cell Test #4)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	0.130	10.20	-2.046	-1.792	-2.299	0.129	0.167	0.100
Li	0.226	2.38	-1.488	-1.429	-1.547	0.226	0.240	0.213
Na	0.144	5.70	-1.939	-1.797	-2.080	0.144	0.166	0.125
Si	0.077	3.42	-2.566	-2.481	-2.651	0.077	0.084	0.071

CI = Confidence Interval; Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.05/2)} = 4.303$).
PCT = Product Consistency Test
Exp Transformed = retransformed data from ln form to original form

Table 4.16. PCT Leach Test Results for 101-AZ Core #1 Simulant

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
(Laboratory Test #6 in Teflon - based on sample size of 2)								
B	0.233	5.00	-1.457	-1.008	-1.907	0.233	0.365	0.149
Li	0.246	5.07	-1.401	-0.945	-1.856	0.246	0.389	0.156
Na	0.200	5.00	-1.611	-1.162	-2.060	0.200	0.313	0.127
Si	0.132	5.00	-2.027	-1.578	-2.476	0.132	0.206	0.084
(Hot Cell Test #4 in stainless steel - based on sample size of 2 washed in laboratory)								
B	0.216	5.00	-1.532	-1.082	-1.981	0.216	0.339	0.138
Li	0.263	5.26	-1.335	-0.863	-1.808	0.263	0.422	0.164
Na	0.216	5.48	-1.532	-1.039	-2.024	0.216	0.354	0.132
Si	0.140	5.00	-1.966	-1.517	-2.416	0.140	0.219	0.089
(Hot Cell Test #4 in stainless steel - sample size of 2 washed in hot cell)								
B	0.183	13.79	-1.703	-0.463	-2.942	0.182	0.629	0.053
Li	0.227	9.05	-1.485	-0.672	-2.298	0.226	0.511	0.100
Na	0.182	9.46	-1.707	-0.856	-2.557	0.181	0.425	0.078
Si	0.118	12.14	-2.137	-1.046	-3.228	0.118	0.351	0.040
(Hot Cell Test #4 in stainless steel - combined in-cell results based on sample size of 4)								
B	0.200	12.80	-1.617	-1.439	-1.795	0.198	0.237	0.166
Li	0.245	10.74	-1.410	-1.261	-1.559	0.244	0.283	0.210
Na	0.199	12.03	-1.619	-1.452	-1.786	0.198	0.234	0.168
Si	0.129	12.35	-2.052	-1.880	-2.223	0.129	0.153	0.108

CI = Confidence Interval
PCT = Product Consistency Test
Exp Transformed = retransformed data from ln form to original form

Figure 4.16 is a comparison of elemental releases for 101-AZ Core 1 glass, simulant, and model prediction. General observations of the data are as follows: (1) the core sample and simulant releases for B, Li, and Na were significantly lower than model predictions; (2) the core sample release for B, Na, and Si were lower than corresponding simulant releases; (3) the core sample release for Li was consistent with simulant release; and (4) simulant results in-cell and in-laboratory were within the experimental error. The data with large confidence intervals were those tests with duplicate samples only.

Table 4.17 provides a direct comparison of mean releases between 101-AZ Core 1 and its in-cell tested simulant. Elemental releases from the simulant were 54% greater for B, 8% greater for Li, 38% greater for Na, and 68% greater for Si than corresponding releases in the actual waste glass. Statistical differences were found for B, Na, and Si to a 95% confidence interval between the simulant and core sample. No statistical difference was found for Li, which yielded an alpha error of 0.49. In other words, the confidence interval would have to be reduced to 50% before a statistical difference could be concluded for Li. A beta error, which would estimate the probability of not detecting a difference in the means when one exists, is not calculated for the test.

4.5.1.2 101-AZ Core 2 and Simulant

Results from PCT of 101-AZ Core 2 are provided in Table 4.18. The durability of the glass as measured by boron release was 0.203 g/m^2, which is approximately 20 to 30 times more durable than the EA glass limit. As was observed with the first core, the Li release was greater than the B release (~45%), and Si release was less than the B release (~40%). The percent RSDs ranged from 5% to 7%, except for Li, which has an RSD of 20.7%. The standard deviations are based on quadruplicate glass, triplicate leachate, and a duplicate leachate blank analyses. Confidence intervals were based on a sample size of three leachates.

Results from PCT testing of 101-AZ Core 2 simulant are provided in Table 4.19. Separate results are reported for three simulant samples washed and prepared in the laboratory and tested with the radioactive samples in the hot cell (stainless-steel containers); two simulant samples tested in the laboratory in stainless-steel containers; and two simulant samples tested in the laboratory in Teflon containers. As with 101-AZ Core 1 simulant, elemental analysis was not performed on the simulant glass and normalized releases are calculated from the "as-batched" composition. Assumed glass analytical uncertainty and limited replicate samples resulted in conservative percent RSDs and confidence intervals.

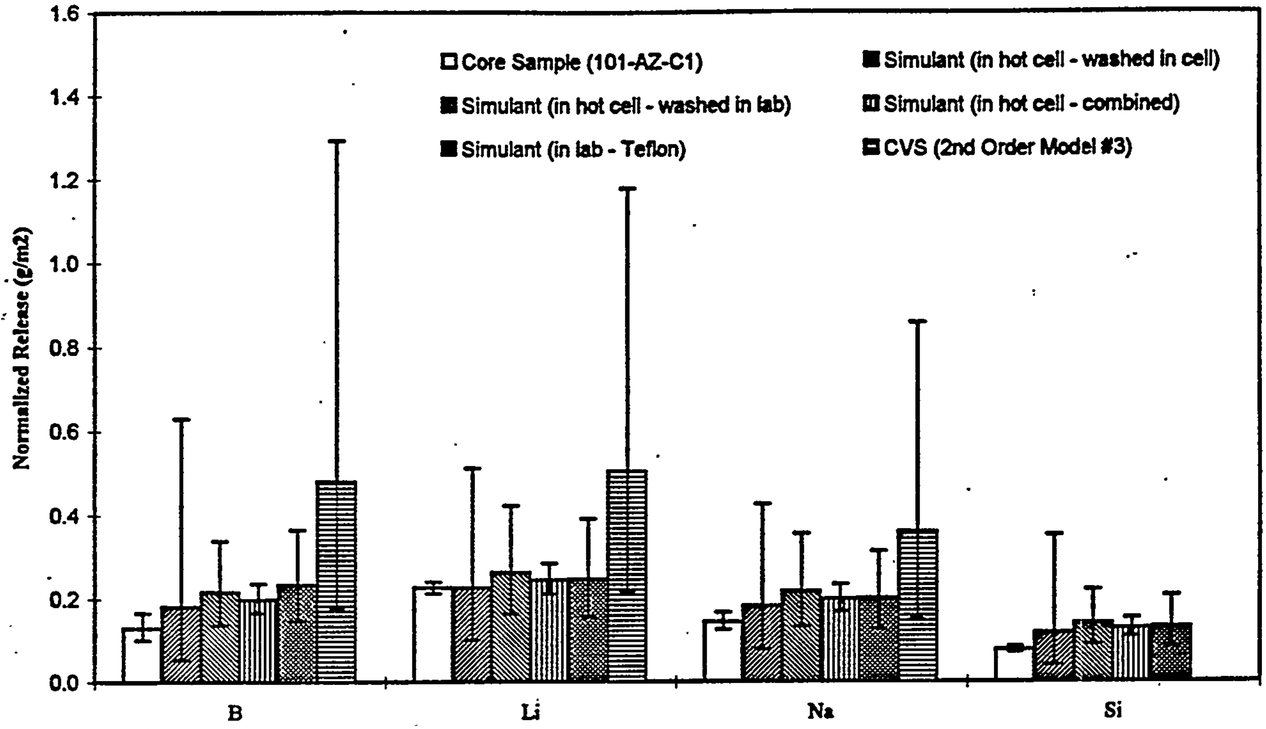


Figure 4.16. PCT Results and Model Predictions for 101-AZ Core #1 and Simulant (error bars = estimated 95% confidence interval)

Table 4.17. PCT Durability Comparison of 101-AZ Core 1 Simulant to 101-AZ Core 1 Radioactive Glass

	Mean Normalized Release (g/m^2)					
	Simulant	Radioactive	Ratio Simulant/ Radioactive	Statistical Difference at 95% CI	Alpha	Degrees of Freedom (n1+n2-2)
B	0.200	0.130	1.54	Yes	0.005	5
Li	0.245	0.226	1.08	No	0.49	5
Na	0.199	0.144	1.38	Yes	0.009	5
Si	0.129	0.077	1.68	Yes	0.0016	5

Notes:
PCT = Product Consistency Test
CI = Confidence Interval
Mean releases become significantly different at a confidence interval of 1-alpha.
n = number of replicate leachate samples.

Releases from hot-cell simulant samples tested in stainless-steel containers were slightly greater than those observed in laboratory-tested samples in Teflon containers, but in all cases differences were less than 10% greater. The differences between releases in hot-cell samples and laboratory samples tested in stainless-steel containers showed no consistent trend, and in all cases the differences were less than 10%. For this simulant, hot-cell samples were prepared in the laboratory, which included weighing and transferring of the glass and leachant into the leach containers, sparging the container head space with argon, sealing the containers, swirling to dislodge bubbles, and removing residual glass from sides of containers.

Figure 4.17 compares elemental releases for 101-AZ Core 2 glass, simulant, and model prediction. General observations of the data are as follows: (1) the simulant releases for B, Li, and Na were slightly lower than model predictions; (2) the core sample releases were lower than corresponding simulant releases; and (3) simulant results in-cell and in-laboratory were within experimental error. The data with large confidence intervals are those tests with duplicate samples.

Table 4.20 compares mean releases between 101-AZ Core 2 and its in-cell tested simulant. Elemental releases from the simulant were 67% greater for B, 26% greater for Li, 136% greater for Na, and 30% greater for Si than were corresponding releases in the actual waste glass. Statistical differences were found for B, Na, and Si to a 95% confidence interval between the simulant and core sample. No statistical difference was found for Li, which yielded an error of 0.082. The confidence interval would have to have been reduced to 92% before a statistical difference could be concluded for Li. A beta error, which would estimate the probability of not detecting a difference in the means being compared when one does exist, is not calculated for the test.

Table 4.18. PCT Leach Test Results for 101-AZ Core #2 (Hot Cell Test #3)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	0.203	5.98	-1.596	-1.448	-1.745	0.203	0.235	0.175
Li	0.293	20.67	-1.241	-0.727	-1.754	0.289	0.483	0.173
Na	0.105	6.89	-2.249	-2.078	-2.421	0.105	0.125	0.089
Si	0.141	4.87	-1.961	-1.841	-2.082	0.141	0.159	0.125

CI = Confidence Interval

Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.05/2)} = 4.303$).

PCT = Product Consistency Test

Exp Transformed = retransformed data from ln form to original form

Table 4.19. PCT Leach Test Results for 101-AZ Core #2 Simulant

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
(Lab Test #8 in Teflon - based on sample size of 2)								
B	0.327	5.00	-1.117	-0.668	-1.566	0.327	0.513	0.209
Li	0.344	5.00	-1.066	-0.617	-1.515	0.344	0.540	0.220
Na	0.227	5.00	-1.484	-1.034	-1.933	0.227	0.355	0.145
Si	0.171	5.00	-1.765	-1.315	-2.214	0.171	0.268	0.109
(Lab Test #10 in stainless steel - based on sample size of 2)								
B	0.371	9.71	-0.994	-0.122	-1.866	0.370	0.885	0.155
Li	0.385	5.27	-0.954	-0.481	-1.428	0.385	0.618	0.240
Na	0.265	10.00	-1.331	-0.433	-2.230	0.264	0.649	0.108
Si	0.185	5.87	-1.686	-1.158	-2.213	0.185	0.314	0.109
(Hot Cell Test #3 in stainless steel - based on sample size of 3)								
B	0.338	6.38	-1.084	-0.925	-1.242	0.338	0.396	0.289
Li	0.369	5.75	-0.998	-0.856	-1.141	0.368	0.425	0.319
Na	0.248	6.63	-1.393	-1.229	-1.558	0.248	0.293	0.211
Si	0.184	5.41	-1.695	-1.560	-1.829	0.184	0.210	0.161

CI = Confidence Interval

PCT = Product Consistency Test

Exp Transformed = retransformed data from ln form to original form

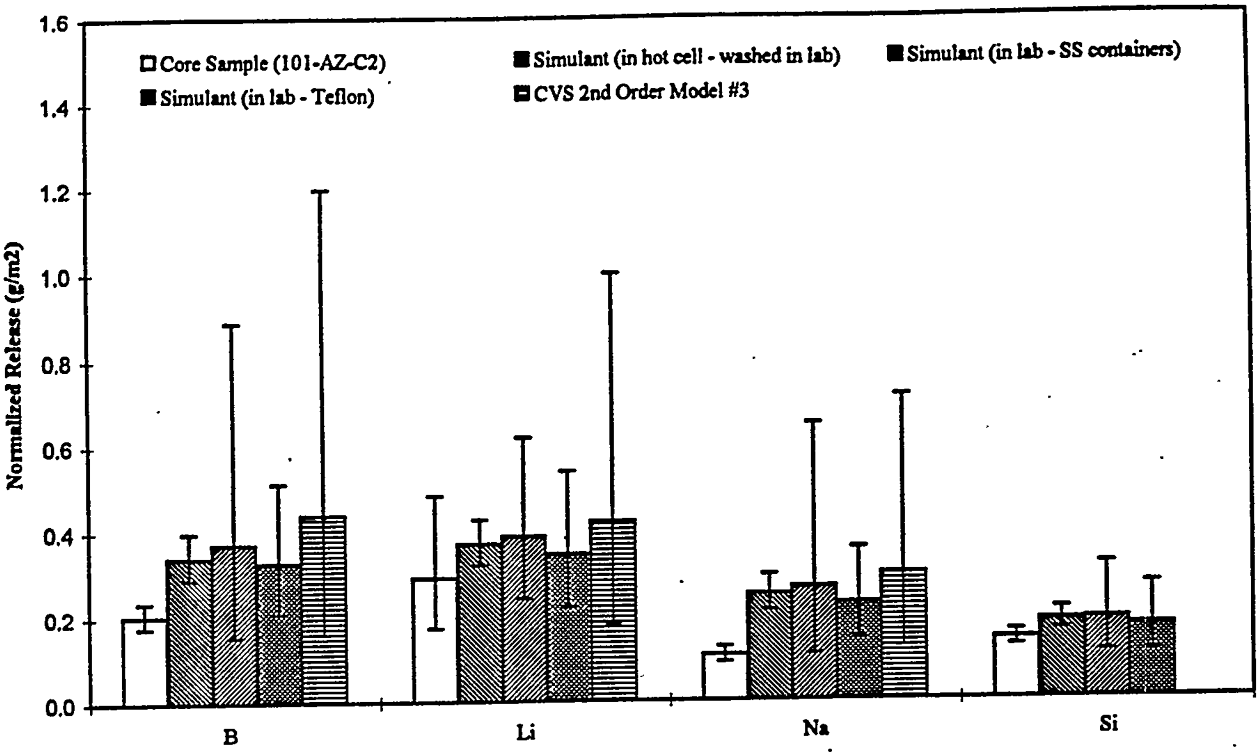


Figure 4.17. PCT Results and Model Predictions for 101-AZ Core #2 and Simulant (error bars = estimated 95% confidence interval)

Table 4.20. PCT Durability Comparison of 101-AZ Core 2 Simulant to 101-AZ Core 2 Radioactive Glass

	Mean Normalized Release (g/m^2)					
	Simulant	Radioactive	Ratio Simulant/ Radioactive	Statistical Difference at 95% CI	Alpha	Degrees of Freedom (n1+n2-2)
B	0.338	0.203	1.67	Yes	0.0012	4
Li	0.369	0.293	1.26	No	0.082	4
Na	0.248	0.105	2.36	Yes	0.0006	4
Si	0.184	0.141	1.30	Yes	0.0038	4

Notes: PCT = Product Consistency Test
CI = Confidence Interval
Mean releases become significantly different at a confidence interval of 1-alpha.
n = number of replicate leachate samples.

4.5.1.3 102-AZ Core 1 and Simulant

Results from PCT of 102-AZ Core 1 are provided in Table 4.21. The durability of the glass as measured by boron release is 0.211 g/m^2, which is approximately 20 to 30 times more durable than the EA glass limit. As was observed with the first two core samples, the Li release was greater than the B release (~43%) and Si release was less than the B release (~25%). The percent RSDs were around 2%, except for Li which had a 9% RSD. The standard deviations were based on duplicate glass, triplicate leachate, and duplicate leachate blank analyses. Confidence intervals were based on a sample size of three leachates.

Results from PCT of 102-AZ Core 1 simulant are provided in Table 4.22. Separate results are reported for two simulant samples crushed, sieved, washed, prepared and tested with the radioactive samples in the hot cell (stainless-steel containers) and two simulant samples tested in the laboratory in Teflon containers. Two additional samples were prepared in the laboratory and tested in the hot cell (see Appendix A, Table A.15), but were not reported. Following the in-cell durability test, it was determined that these two simulant samples were likely unwashed. Elemental analysis was not performed on the simulant glass, and normalized releases were calculated from the "as-batched" composition. Assumed glass analytical uncertainty and limited replicate samples resulted in conservative percent RSDs and confidence intervals.

Releases from hot-cell simulant samples tested in stainless-steel containers were essentially equal to those observed in laboratory-tested samples in Teflon containers. Differences between in-cell and in-laboratory releases were within 2% for all reported elements except Na, which was within 8%.

Figure 4.18 compares elemental releases for 102-AZ Core 1 glass, simulant, and model prediction. General observations of the data are as follows: (1) the simulant releases for B, Li, and Na were lower than model predictions; (2) the core sample releases were slightly lower than corresponding simulant releases; and (3) simulant results in-cell and in-laboratory were essentially identical. The data with large confidence intervals were those tests with only duplicate samples.

Table 4.23 compares mean releases between 102-AZ Core 1 and its in-cell tested simulant. Elemental releases from the simulant were 28% greater for B, 17% greater for Li, 165% greater for Na, and 3% greater for Si than corresponding releases in the actual waste glass. Statistical differences were found for B, Li, and Na, to a 95% confidence interval between the simulant and core sample. No statistical difference was found for Si, which yielded an alpha error of 0.24. The confidence interval would have to have been reduced to 75% before a statistical difference could be concluded for Si. A beta error, which would estimate the probability of not detecting a difference in the means being compared when one exists, was not calculated for the test.

Table 4.21. PCT Leach Test Results for 102-AZ Core #1 (Hot Cell Test #5)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	0.211	2.10	-1.556	-1.504	-1.608	0.211	0.222	0.200
Li	0.301	2.04	-1.201	-1.151	-1.252	0.301	0.316	0.286
Na	0.049	9.04	-3.021	-2.796	-3.245	0.049	0.061	0.039
Si	0.158	2.17	-1.846	-1.792	-1.900	0.158	0.167	0.150

CI = Confidence Interval

Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.05/2)} = 4.303$).

PCT = Product Consistency Test

Exp Transformed = retransformed data from ln form to original form

Table 4.22. PCT Leach Test Results for 102-AZ Core #1 Simulant

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
(Lab Test #11 in Teflon - based on sample size of 2)								
B	0.274	5.16	-1.296	-0.833	-1.759	0.274	0.435	0.172
Li	0.353	5.09	-1.042	-0.585	-1.499	0.353	0.557	0.223
Na	0.136	5.00	-1.992	-1.543	-2.441	0.136	0.214	0.087
Si	0.161	5.03	-1.826	-1.374	-2.278	0.161	0.253	0.103
(Hot Cell Test #5 in stainless steel - based on sample size of 2)								
B	0.269	5.25	-1.313	-0.841	-1.785	0.269	0.431	0.168
Li	0.349	5.02	-1.052	-0.601	-1.504	0.349	0.548	0.222
Na	0.126	5.10	-2.075	-1.616	-2.533	0.126	0.199	0.079
Si	0.165	5.08	-1.799	-1.343	-2.256	0.165	0.261	0.105

CI = Confidence Interval

PCT = Product Consistency Test

Exp Transformed = retransformed data from ln form to original form

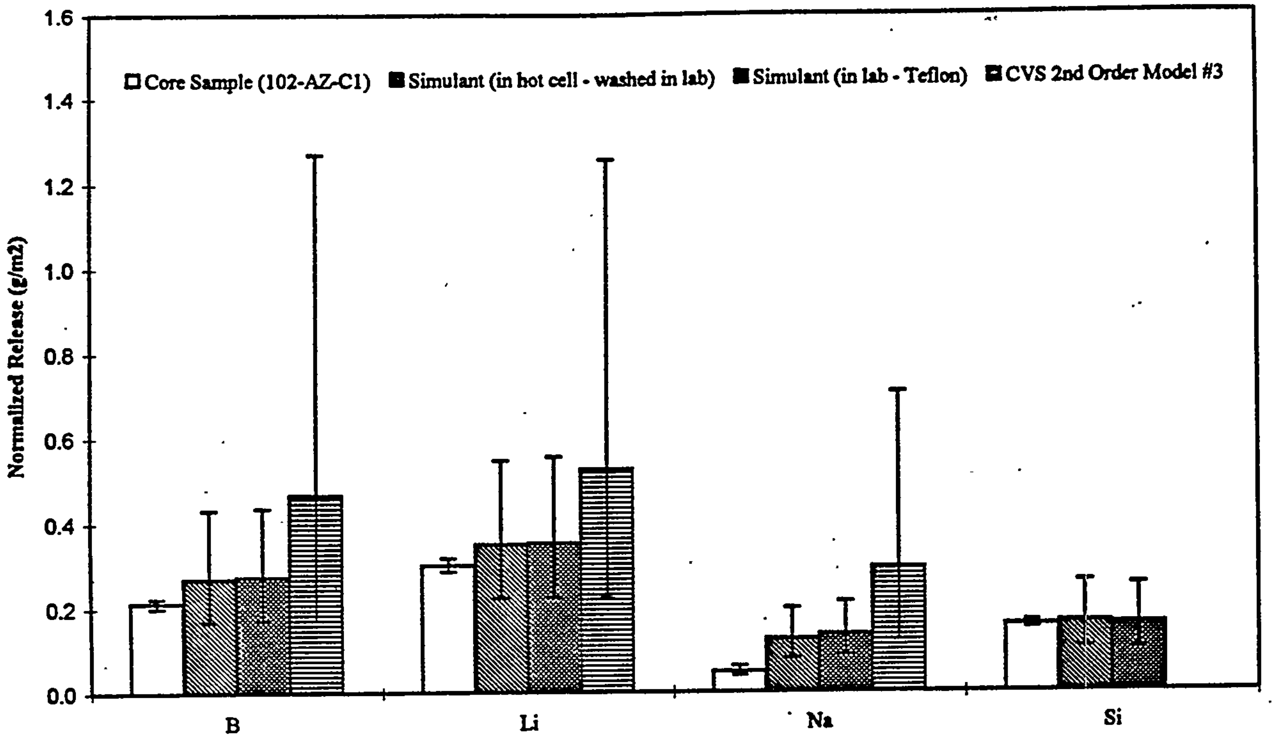


Figure 4.18. PCT Results and Model Predictions for 102-AZ Core #1 and Simulant (error bars = estimated 95% confidence interval)

Table 4.23. PCT Durability Comparison of 102-AZ Core 1 Simulant to 102-AZ Core 1 Radioactive Glass

	Mean Normalized Release (g/m²)					
	Simulant	Radioactive	Ratio Simulant/ Radioactive	Statistical Difference at 95% CI	Alpha	Degrees of Freedom (n1+n2-2)
B	0.271	0.211	1.28	Yes	0.004	3
Li	0.351	0.301	1.17	Yes	0.016	3
Na	0.130	0.049	2.65	Yes	0.0004	3
Si	0.163	0.158	1.03	No	0.24	3

Notes:
PCT = Product Consistency Test
CI = Confidence Interval
Mean releases become significantly different at a confidence interval of 1-alpha.
n = number of replicate leachate samples.

4.5.1.4 Comparison of Core Sample and Simulant PCT Results to Model Predictions

Glass durability models as developed by Hrma, Piepel et al. (1994) were used to generate PCT-normalized release predictions and statistics for each of the core sample glass compositions and the CVS-IS-HW39-4 composition. Model predictions for these compositions are provided in Appendix A, Tables A.20 through A.24. Results from four different models are included as follows: 1st order; 2nd order model #1; 2nd order model #2; and 2nd order model #3. The latter model, 2nd order model #3, provided the best fit for CVS glasses reported by Hrma and Piepel was selected for comparison with core sample results.

Figures 4.19, 4.20, and 4.21 provide a comparison of model predictions to results from actual waste glasses, simulant glasses of same composition, internal standards used in these tests, and environmental assessment glass (upper limit glass). The data are plotted as measured ln mean normalized release to predicted ln normalized release. The diagonal line represents the ideal fit between measured and predicted. The error bars are 95% prediction intervals for the model, and are based on a sample size of two. The prediction intervals for means of larger sample sizes, such as the radioactive glasses with three replicates each are slightly smaller than shown. Boron, Li, and Na results are provided in Figures 4.19, 4.20, and 4.21 respectively. A characteristic of the model, verified during validation, is that it tended to over-predict release in high-durability glasses and under-predict release in low-durability glasses. This was evident in each of these three figures. For B, measured results for all tests shown, except one core sample, were within the 95% prediction intervals of the model. For Li, all measured results were within the 95% prediction intervals of the model. All three core samples and one core sample simulant were outside the 95% prediction intervals of the model for Na. Where not specified, hot-cell tests were performed in stainless-steel containers and laboratory tests were performed in Teflon containers.

A summary by glass type of results fitting within the 95% prediction interval of the model is provided in Table 4.24. With the limited amount of data presented, the actual waste glass mean releases were within the model's 95% prediction interval only 56% of the time and the simulant glass mean releases were within the interval 96% of the time.

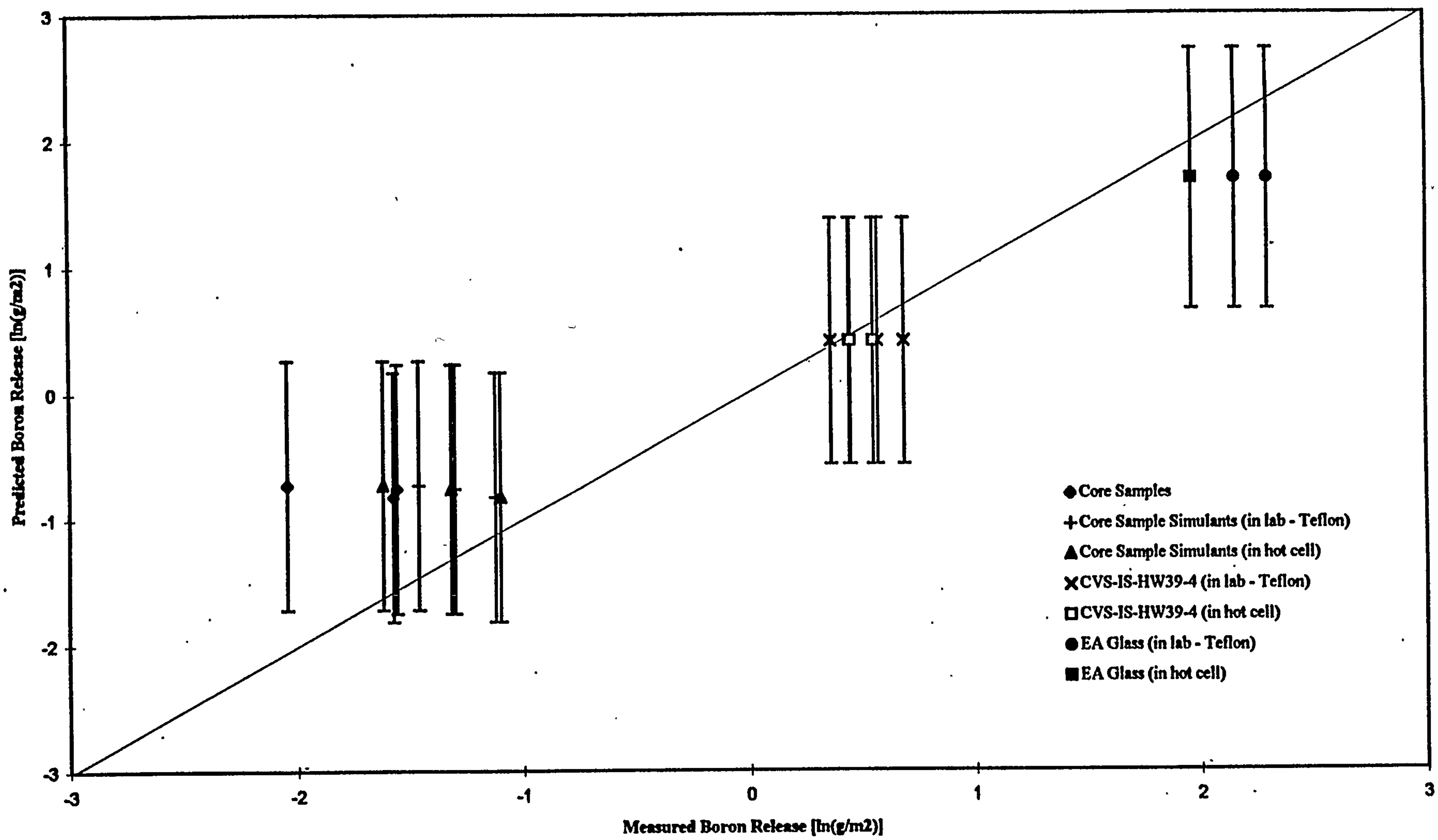


Figure 4.19. Measured Versus Predicted PCT Boron Release for Core Sample, Simulant, and Standard Glasses

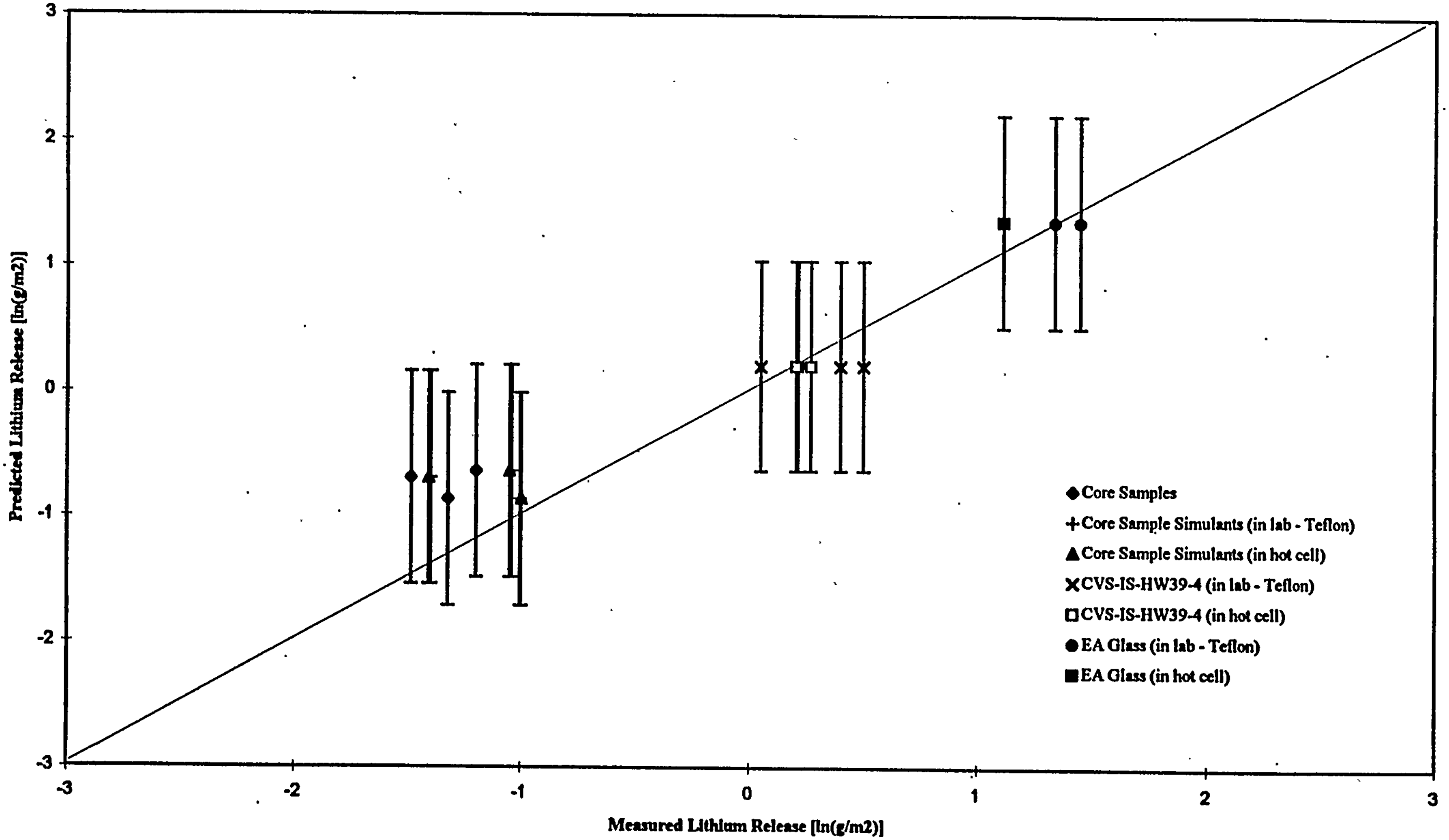


Figure 4.20. Measured Versus Predicted PCT Lithium Release for Core Sample, Simulant, and Standard Glasses

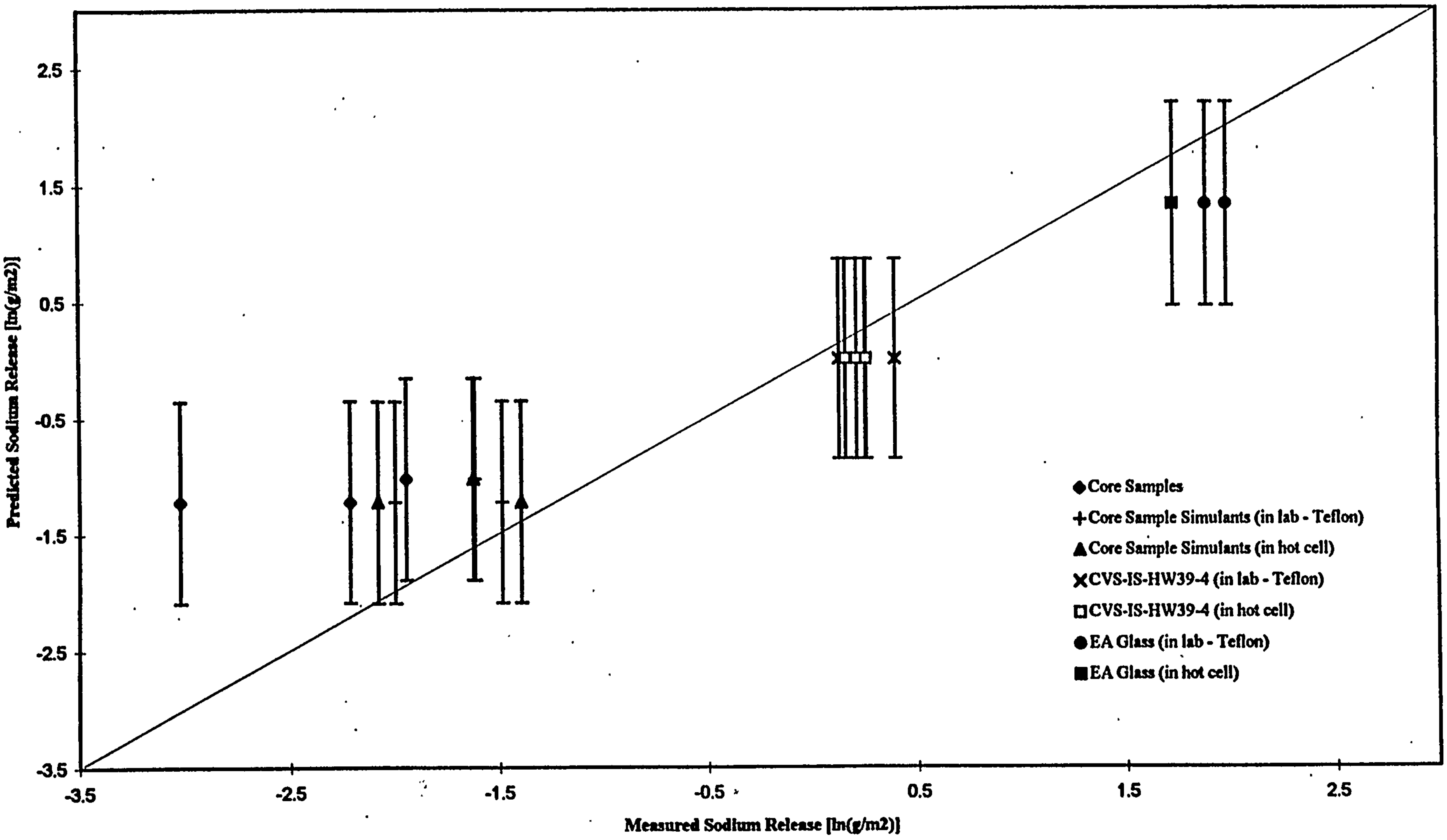


Figure 4.21. Measured Versus Predicted PCT Sodium Release for Core Sample, Simulant, and Standard Glasses

Table 4.24. Summarized Comparison of PCT Model Prediction to Measured

(number of tests within prediction intervals/total number compared)

	Boron	Lithium	Sodium	Total
Core Sample Glasses.	(2/3)	(3/3)	(0/3)	(5/9)
Core Sample Simulants	(3/3)	(3/3)	(2/3)	(8/9)
Reference Glass (CVS-IS-HW39-4)	(3/3)	(3/3)	(3/3)	(9/9)
Environmental Assessment Glass	(3/3)	(3/3)	(3/3)	(9/9)

4.5.1.5 Repeatability of Hot Cell and Laboratory PCTs

Triplicate reference glass samples were tested in each of the hot-cell PCTs to verify consistency between hot-cell tests and to compare them to laboratory tests. The CVS internal standard glass, CVS-IS-HW39-4, was used for this purpose, since considerable comparative laboratory PCT data already exist.

Results from hot-cell tests and one laboratory test in stainless-steel containers are provided in Table 4.25. The durability of the reference glass as measured by B release was around 1.6 g/m^2, which was four to six times more durable than EA glass. Elemental releases for the reference glass in hot-cell tests #3 and test #4 were essentially the same, while hot-cell test #5 yielded slightly higher releases. Each of these tests were performed in stainless-steel containers. The percent RSDs for each of the elements were between 5% and 7.5%, and were based on sample sizes of three. Much of the percent RSD was a contribution from the 5% RSD assumed for glass analytical testing. The in-laboratory test performed in stainless steel yielded slightly higher releases than hot-cell test #5, and resulted in percent RSDs of around 6% to 9%.

Figure 4.22 compares elemental releases from the reference glass for each of the hot-cell PCTs, the in-laboratory test in stainless-steel containers, and prior laboratory tests in Teflon containers (Hrma 1994). Observations from the data are as follows: (1) the three hot-cell tests produced consistent results; (2) the hot-cell tests in stainless-steel containers produced results well within the range set by laboratory tests in Teflon containers; (3) all results were consistent with model predictions; and (4) the laboratory test in stainless-steel containers was well within the range of laboratory tests in Teflon containers. Each of the laboratory tests in Teflon containers (CVS2-19, CVS2-51, CVS2-97) used five replicate glasses. Laboratory test #10 in stainless-steel containers included only two replicate glasses, resulting in large confidence intervals. Confidence intervals for the CVS tests were not readily available and are not included.

Table 4.25. PCT Leach Test Results for CVS-IS-HW39-4

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
(Hot Cell Test #3 in stainless steel - based on sample size of 3)								
B	1.531	5.01	0.426	0.551	0.302	1.531	1.734	1.352
Li	1.227	5.40	0.204	0.338	0.070	1.227	1.403	1.073
Na	1.174	5.80	0.160	0.304	0.016	1.174	1.356	1.016
Si	0.395	5.80	-0.930	-0.786	-1.074	0.395	0.456	0.342
(Hot Cell Test #4 in stainless steel - based on sample size of 3)								
B	1.557	5.92	0.442	0.589	0.295	1.556	1.803	1.343
Li	1.241	6.42	0.215	0.375	0.056	1.240	1.455	1.058
Na	1.233	7.50	0.209	0.395	0.023	1.232	1.485	1.023
Si	0.388	5.82	-0.947	-0.802	-1.091	0.388	0.448	0.336
(Hot Cell Test #5 in stainless steel - based on sample size of 3)								
B	1.731	7.38	0.547	0.731	0.364	1.729	2.077	1.439
Li	1.314	6.81	0.272	0.442	0.103	1.313	1.555	1.109
Na	1.296	7.31	0.258	0.440	0.077	1.295	1.553	1.080
Si	0.431	6.22	-0.842	-0.687	-0.996	0.431	0.503	0.369
(Lab Test #10 in stainless steel - based on sample size of 2)								
B	1.820	9.23	0.598	1.427	-0.232	1.818	4.165	0.793
Li	1.384	8.31	0.324	1.070	-0.423	1.382	2.916	0.655
Na	1.322	8.62	0.278	1.052	-0.497	1.320	2.865	0.608
Si	0.415	6.06	-0.879	-0.335	-1.423	0.415	0.715	0.241

CI = Confidence Interval
PCT = Product Consistency Test
Exp Transformed = retransformed data from ln form to original form

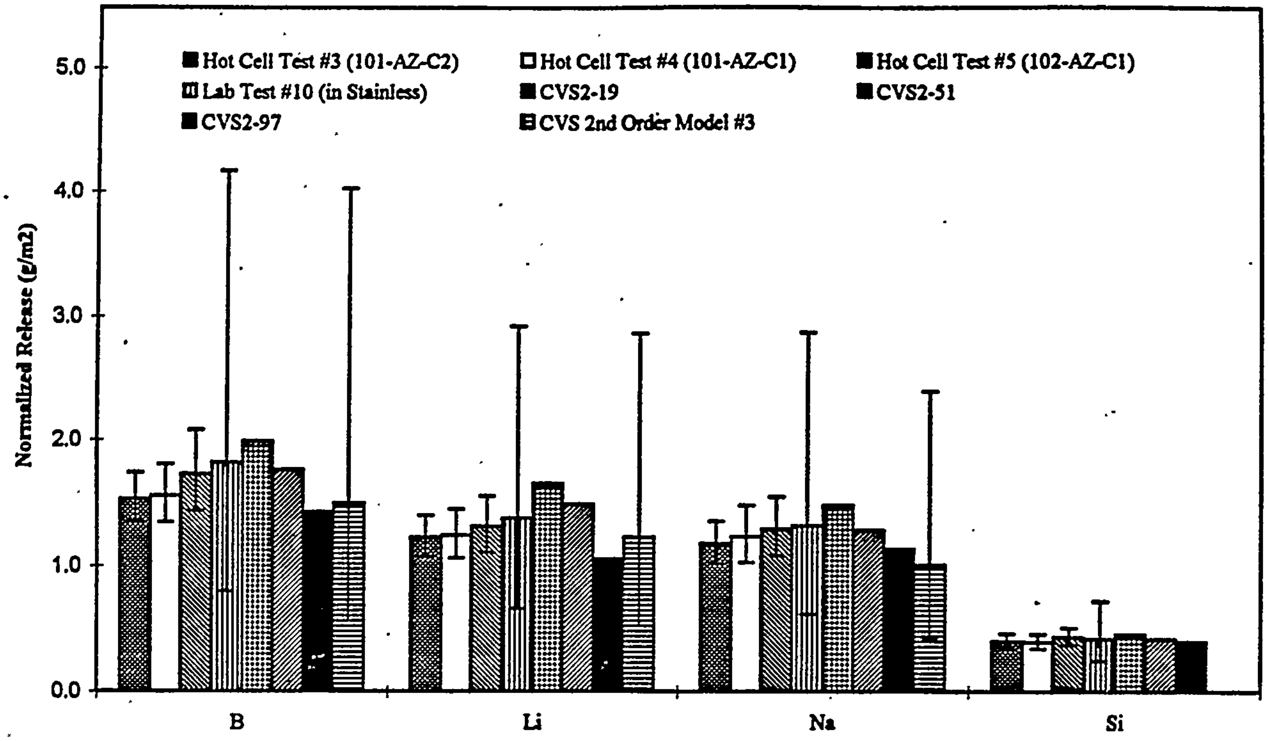


Figure 4.22. PCT Results and Model Predictions for Reference Glass CVS-IS-HW39-4 (Error Bars = Estimated 95% Confidence Interval)

Table 4.26 provides a comparison of mean releases from the reference glasses in the two most widely variant hot-cell tests (tests #3 and #5). Elemental releases in test #5 were 13% greater for B, 7% greater for Li, 10% greater for Na, and 9% greater for Si than corresponding releases in test #3. No statistical differences were found. Comparing the means from these two tests is not technically correct for the following reasons: (1) test-to-test variability contributed to the difference between results in the two tests, but this uncertainty was ignored in calculation of the standard deviations. This factor will increase chances of concluding a statistical difference when one does not exist. (2) Conservative estimates for glass analysis (i.e., 5% RSD) were included in the standard deviation for both tests, but should be ignored because glass samples were drawn from the same batch and a single glass composition was used for calculation of normalized releases. This factor will increase chances of not concluding a statistical difference when one exists. The statistical comparison is presented, however, to show that even when introducing test-to-test variability, the statistical comparison yielded the desired result when using the conservative estimate for glass analytical uncertainty.

4.5.1.6 Comparison of Leach Containers for PCTs

The PCT method (Jantzen 1992) requires use of PFA Teflon or 304L stainless-steel leach vessels. Stainless-steel containers with Teflon gaskets are used for testing nuclear waste glass or testing in radioactive fields. The two containers are different in two notable ways. First, Teflon is permeable to CO_2 and O_2, which equilibrates with the leachate. The stainless-steel container provides a closed system to the atmosphere, except that which is left in the head space. Second, stainless steel

Table 4.26. Comparison of Hot-Cell (HC) Tests #3 and #5 Using Reference Glass CVS-IS-HW39-4

	Mean Normalized Release (g/m^2)					
	CVS-IS-HW39-4 HC Test #3	CVS-IS-HW39-4 HC Test #5	Ratio HC Test #5/ HC Test #3	Statistical Difference at 95% CI	Alpha	Degrees of Freedom (n1+n2-2)
B	1.531	1.731	1.13	No	.072	4

Table 4.27. PCT Leach Test Results for NCAW Simulant Glass

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
(Lab Test #2 in Teflon - based on sample size of 2)								
B	1.920	1.43	0.652	0.780	0.524	1.920	2.183	1.689
Li	1.669	3.55	0.512	0.831	0.193	1.668	2.295	1.213
Na	1.762	0.00	0.566	0.566	0.566	1.762	1.762	1.762
Si	0.493	8.30	-0.708	0.038	-1.454	0.493	1.039	0.234
(Lab Test #5 in stainless steel - based on sample size of 3)								
B	1.698	6.31	0.528	1.096	-0.039	1.696	2.991	0.962
Li	1.435	2.95	0.361	0.626	0.097	1.435	1.870	1.101
Na	1.301	0.00	0.263	0.263	0.263	1.301	1.301	1.301
Si	0.422	7.79	-0.863	-0.164	-1.563	0.422	0.849	0.210
(Lab Test #4 in fused-silica - based on sample size of 2)								
B	1.992	15.80	0.683	2.102	-0.737	1.979	8.182	0.479
Li	1.375	6.15	0.318	0.870	-0.235	1.374	2.387	0.790
Na	1.340	4.04	0.292	0.655	-0.071	1.339	1.925	0.931
Si	0.458	14.65	-0.784	0.532	-2.100	0.457	1.703	0.122
(Hot Cell Test #2 in stainless steel - based on sample size of 2)								
B	1.407	5.39	0.341	0.447	0.235	1.406	1.563	1.265
Li	1.298	4.89	0.260	0.356	0.164	1.297	1.428	1.179
Na	1.149	4.71	0.138	0.231	0.046	1.148	1.259	1.047
Si	0.443	8.49	-0.815	-0.649	-0.982	0.442	0.523	0.375

CI = Confidence Interval
PCT = Product Consistency Test
Exp Transformed = retransformed data from ln form to original form

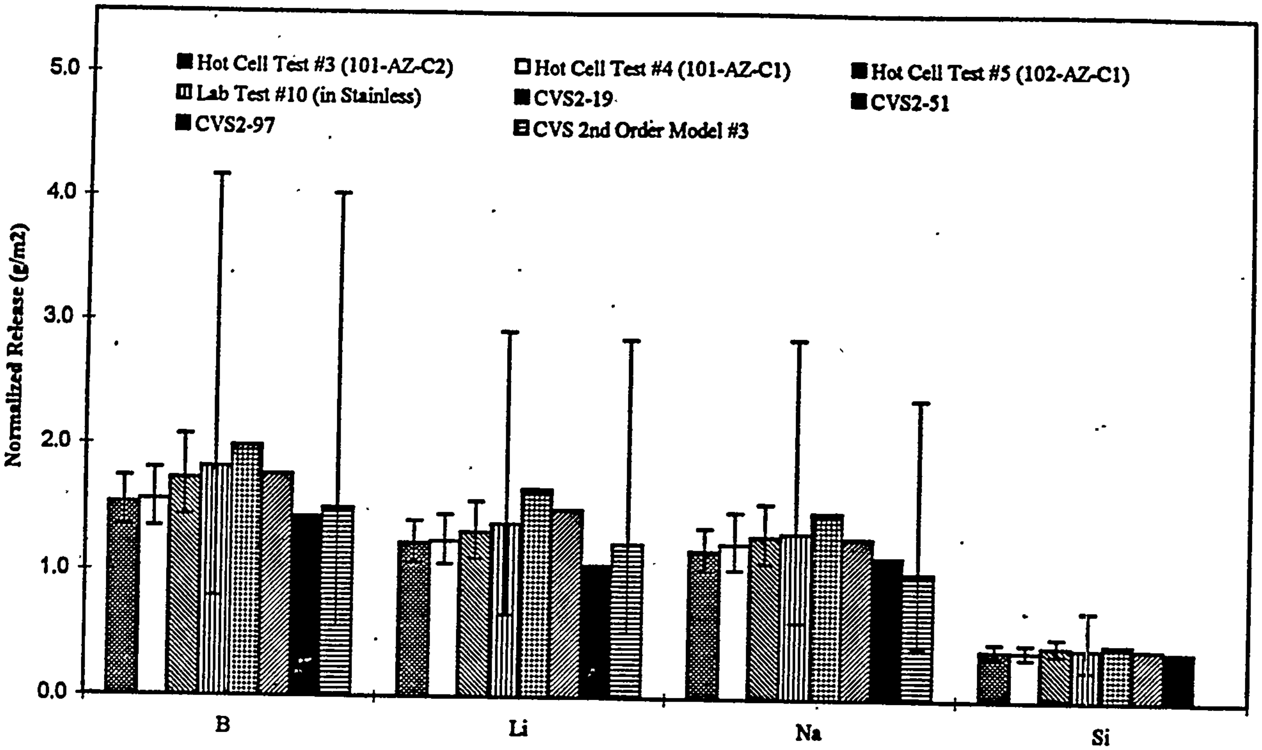


Figure 4.22. PCT Results and Model Predictions for Reference Glass CVS-IS-HW39-4 (Error Bars = Estimated 95% Confidence Interval)

Table 4.26 provides a comparison of mean releases from the reference glasses in the two most widely variant hot-cell tests (tests #3 and #5). Elemental releases in test #5 were 13% greater for B, 7% greater for Li, 10% greater for Na, and 9% greater for Si than corresponding releases in test #3. No statistical differences were found. Comparing the means from these two tests is not technically correct for the following reasons: (1) test-to-test variability contributed to the difference between results in the two tests, but this uncertainty was ignored in calculation of the standard deviations. This factor will increase chances of concluding a statistical difference when one does not exist. (2) Conservative estimates for glass analysis (i.e., 5% RSD) were included in the standard deviation for both tests, but should be ignored because glass samples were drawn from the same batch and a single glass composition was used for calculation of normalized releases. This factor will increase chances of not concluding a statistical difference when one exists. The statistical comparison is presented, however, to show that even when introducing test-to-test variability, the statistical comparison yielded the desired result when using the conservative estimate for glass analytical uncertainty.

4.5.1.6 Comparison of Leach Containers for PCTs

The PCT method (Jantzen 1992) requires use of PFA Teflon or 304L stainless-steel leach vessels. Stainless-steel containers with Teflon gaskets are used for testing nuclear waste glass or testing in radioactive fields. The two containers are different in two notable ways. First, Teflon is permeable to CO_2 and O_2, which equilibrates with the leachate. The stainless-steel container provides a closed system to the atmosphere, except that which is left in the head space. Second, stainless steel

Table 4.26. Comparison of Hot-Cell (HC) Tests #3 and #5 Using Reference Glass CVS-IS-HW39-4

	Mean Normalized Release (g/m^2)					
	CVS-IS-HW39-4 HC Test #3	CVS-IS-HW39-4 HC Test #5	Ratio HC Test #5/ HC Test #3	Statistical Difference at 95% CI	Alpha	Degrees of Freedom (n1+n2-2)
B	1.531	1.731	1.13	No	.072	4
Li	1.227	1.314	1.07	No	.23	4
Na	1.174	1.296	1.10	No	.13	4
Si	0.395	0.431	1.09	No	.085	4

Notes: PCT = Product Consistency Test
CI = Confidence Interval
Mean releases become significantly different at a confidence interval of 1-alpha.
n = number of replicate leachate samples.

controls the system redox and forces a reducing environment. Although the Teflon container is chemically inert, the system is oxidized by virtue of the air atmosphere.

Initially, a redox-inert container was sought for testing radioactive glass in the hot cell, for the following reasons: (1) actinides plate out on the stainless-steel container walls, distorting the actinide release data (i.e., altering the concentration of actinides in solution may affect the rate of certain mineral formations and elemental releases); and (2) the reducing atmosphere may not be representative of the repository environment, which is a function of design. Consequently, alternative container types were considered, including fused-silica and gold-plated.

Results from a model simulation of the NCAW simulant glass reacted in deionized water at 90°C using a fixed O_2 and CO_2 gas fugacity showed a slightly different sequence of secondary mineral formation, compared with the closed-system simulations.[a] Talc, $Mg_3Si_4O_{10}(OH)_2$, which lowers the activity of silicic acid, was predicted to form. Consequently, the reaction rate for the simulant in Teflon containers was predicted to be slightly higher over a finite range of reaction progress for the same glass in stainless-steel containers. This may result in slightly higher PCT releases for Teflon containers, but should have little effect on MCC-1 releases. Model simulations were not performed on the fused-silica containers.

Limited comparative PCT data of Teflon, stainless steel, and fused-silica-lined leach containers were generated to support the decision of which leach container to use in the hot cell. Glass specimens from a single batch of NCAW simulant glass were tested in the laboratory, using all three container types, and in the hot cell using stainless steel. Results from these tests are reported in Table 4.27. Sample sizes for each of the tests were only two, resulting in large statistical confidence intervals. The

---

(a) Memo from Bernard P McGrail to Eugene V. Morrey, Pacific Northwest Laboratory, "Results for Teflon Containers," 2 January 1992.

calculated statistics represent only short-term variability and preclude a meaningful statistical comparison of means for each contains type. The ratio of minimum-measured release to maximum-measured release for the elements are as follows: 0.71 for B, 0.78 for Li, 0.65 for Na, and 0.86 for Si. Similar comparisons of CVS-IS-HW39-4 performed in Teflon containers but at different times (CVS2-19 versus CVS2-97 [see Figure 4.22]) yielded 0.72 for B, 0.64 for Li, 0.77 for Na, and 0.87 for Si. The differences observed between the three leach containers are equivalent in magnitude to long-term differences observed in the internal standard glass. The three leach containers may produce statistically different PCT results, but the limited amount of data generated in this study was insufficient to differentiate between the containers.

Figure 4.23 provides a graphical representation of the NCAW simulant glass tested in the three containers. General observations of the data are that with the exception of B, the normalized releases for fused-silica, stainless steel in the laboratory and stainless steel in the hot cell were essentially identical; Teflon results were slightly higher than the other containers for Li and Na; and fused-silica produced the widest variation in results. Again, each of these observations may or may not be significant, but the current data was insufficient to differentiate between the results. Review of Figures 4.16, 4.17, and 4.18 showed additional comparative data between stainless steel and Teflon containers, except that the stainless-steel tests are also in the hot cell. In these tests, no consistent difference was observed between the two container types.

The decision to use stainless-steel containers in hot-cell testing was made based on several considerations, as follows:

- Uncertainties in the repository design make stainless steel as good of a choice as any. A forced reducing condition is easier to model than a system where the glass determines the redox state. Radioactive data obtained in stainless steel can be used to validate models of this type. Presently, an iron overpack is being considered for use in the repository with special alloys for the canisters, which may drive the system reducing. However, even with the overpack, conservative factors may prevent taking credit for the reducing atmosphere.

- Teflon, with its oxidizing condition, is technically preferable but is susceptible to radiolytic degradation. Expected dose from one sample of glass could be as high as 10^4 rad during a 7-day test.

- To validate simulants, the simulant and radioactive glasses can be tested under the same conditions for direct comparison.

- Results from in-cell PCT in stainless steel yielded low intra-sample variability and comparable results to out-of-cell PCT in stainless.

Table 4.27. PCT Leach Test Results for NCAW Simulant Glass

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
(Lab Test #2 in Teflon - based on sample size of 2)								
B	1.920	1.43	0.652	0.780	0.524	1.920	2.183	1.689
Li	1.669	3.55	0.512	0.831	0.193	1.668	2.295	1.213
Na	1.762	0.00	0.566	0.566	0.566	1.762	1.762	1.762
Si	0.493	8.30	-0.708	0.038	-1.454	0.493	1.039	0.234
(Lab Test #5 in stainless steel - based on sample size of 3)								
B	1.698	6.31	0.528	1.096	-0.039	1.696	2.991	0.962
Li	1.435	2.95	0.361	0.626	0.097	1.435	1.870	1.101
Na	1.301	0.00	0.263	0.263	0.263	1.301	1.301	1.301
Si	0.422	7.79	-0.863	-0.164	-1.563	0.422	0.849	0.210
(Lab Test #4 in fused-silica - based on sample size of 2)								
B	1.992	15.80	0.683	2.102	-0.737	1.979	8.182	0.479
Li	1.375	6.15	0.318	0.870	-0.235	1.374	2.387	0.790
Na	1.340	4.04	0.292	0.655	-0.071	1.339	1.925	0.931
Si	0.458	14.65	-0.784	0.532	-2.100	0.457	1.703	0.122
(Hot Cell Test #2 in stainless steel - based on sample size of 2)								
B	1.407	5.39	0.341	0.447	0.235	1.406	1.563	1.265
Li	1.298	4.89	0.260	0.356	0.164	1.297	1.428	1.179
Na	1.149	4.71	0.138	0.231	0.046	1.148	1.259	1.047
Si	0.443	8.49	-0.815	-0.649	-0.982	0.442	0.523	0.375

CI = Confidence Interval
PCT = Product Consistency Test
Exp Transformed = retransformed data from ln form to original form

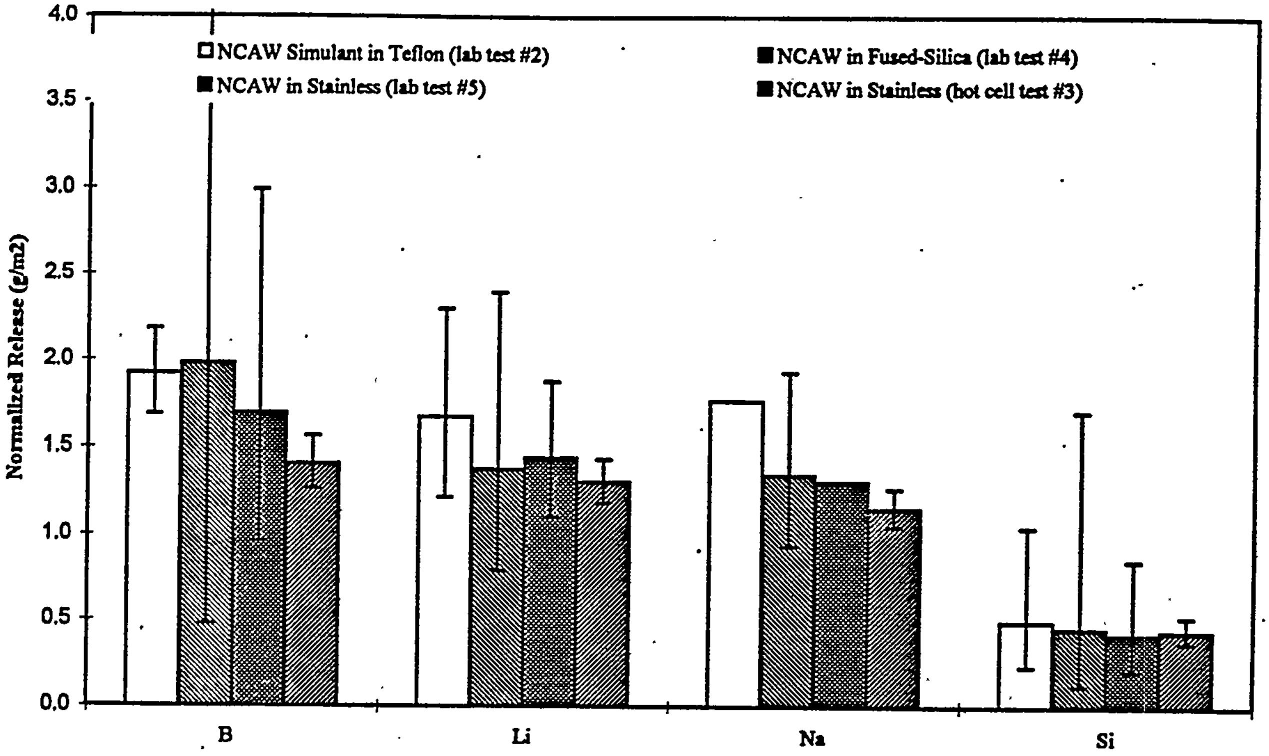


Figure 4.23. Comparison of Alternate Leach Vessels considered for PCT (Error Bars = Estimated 95% Confidence Interval)

4.5.2 Materials Characterization Center (MCC-1) Elemental Releases

The MCC-1 test was developed to compare durabilities of various HLW glasses. An extensive database of MCC-1 results (including CVS) has been created within the HLW glass research community. The ability to compare with this database was a driver for performing MCC-1 analysis on two of the radioactive glasses in this report.

The MCC-1 test is a low surface area-to-volume ratio, static test that can often result in a fairly constant dissolution rate and modest concentrations of silicic acid in solution over a 28-day test period. Results from PCT tests cannot be directly used to predict long-term performance in repository conditions.

4.5.2.1 101-AZ Core 1 and Simulant

Results from MCC-1 testing of 101-AZ Core 1 are provided in Table 4.28. The durability of the glass as measured by MCC-1 boron release was 7.038 g/m^2, which was approximately 7 to 13 times more durable than EA glass. Releases of the four reported elements (B, Li, Na, and Si) were essentially the same, ranging from 7.04 to 6.76 g/m^2. The percent RSDs were quite high, ranging from 28% to 43%. The standard deviations are based on quadruplicate glass, triplicate leachate, and duplicate leachate blank analyses. Confidence intervals were based on a sample size of three leachates.

Note that the differences between the mean normalized releases and retransformed mean ln normalized releases were more significant than the PCT results, because of greater variability in the data, but were still less than 5%.

Results from MCC-1 testing of 101-AZ Core 1 simulant are provided in Table 4.29. Results are reported for two simulant samples tested in the laboratory in Teflon containers. In-cell MCC-1 tests were not performed on the core sample simulants and are therefore not available for direct comparison. The practice of batching core sample simulants and testing them in the same test as the core sample was not begun until after these tests were performed. Elemental analysis was not performed on the simulant glass; normalized releases were calculated from the "as-batched" composition. Assumed analytical glass uncertainties and limited replicate samples resulted in conservative percent RSDs and confidence intervals.

Figure 4.24 compares MCC-1 elemental releases for 101-AZ Core 1 glass, simulant, and model prediction. General observations of the data are as follows: (1) the simulant releases and model predictions matched quite well; (2) the core sample releases were consistently lower than corresponding simulant releases and model predictions; (3) simulant confidence limits were large due to the limited sample size; and (4) core sample confidence limits were large due to variability in the analyzed leachates.

Table 4.30 shows mean releases between 101-AZ Core 1 and its in-laboratory tested simulant. Elemental releases from the simulant were 99% greater for B, 66% greater for Li, 79% greater for Na, and 49% greater for Si than were corresponding releases in the actual waste glass. A statistical difference was found for B, to a 95% confidence interval between the simulant and core sample. No statistical differences were found for Li, Na, and Si, which yielded alpha errors of 0.265, 0.082, and 0.12, respectively. The beta errors, which would estimate the probability of not detecting a difference in the means when one exists, were not calculated for the test. Given the variability in the core sample and the limited sample set for the simulants, the beta error was certainly too high. The statistical comparison provided in Table 4.30 has limited meaning, given that the two tests being compared were performed at different times and under different conditions (i.e., in the hot cell versus in the laboratory, and in fused-silica containers versus in Teflon containers).

Table 4.28. MCC-1 Leach Test Results for 101-AZ Core #1 (Hot Cell Test #1)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	7.038	28.26	1.922	2.624	1.220	6.834	13.790	3.387
Li	7.099	28.73	1.929	2.643	1.215	6.884	14.055	3.372
Na	6.971	43.17	1.877	2.949	0.805	6.534	19.095	2.236
Si	6.760	36.41	1.862	2.767	0.958	6.439	15.911	2.606

CI = Confidence Interval

Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.05/2)} = 4.303$).

MCC-1 = Materials Characterization Center (MCC-1)

Exp Transformed = retransformed data from ln form to original form

Table 4.29. MCC-1 Leach Test Results for 101-AZ Core #1 Simulant (Lab Test #7)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	13.971	6.03	2.637	3.178	2.095	13.967	24.005	8.126
Li	11.794	6.18	2.467	3.022	1.912	11.790	20.537	6.769
Na	12.461	5.52	2.522	3.019	2.026	12.459	20.463	7.586
Si	10.085	5.77	2.311	2.830	1.792	10.083	16.938	6.003

CI = Confidence Interval

Confidence interval is based on a sample size of 2 (i.e., $t_{(2-1,0.05/2)} = 12.706$).

MCC-1 = Materials Characterization Center (MCC-1)

Exp Transformed = retransformed data from ln form to original form

Table 4.30. MCC-1 Durability Comparison of 101-AZ Core 1 Simulant to 101-AZ Core 1 Radioactive Glass

	Mean Normalized Release (g/m^2)					
	Simulant	Radioactive	Ratio Simulant/ Radioactive	Statistical Difference at 95% CI	Alpha	Degrees of Freedom (n1+n2-2)
B	13.971	7.038	1.99	Yes	0.022	3
Li	11.794	7.099	1.66	No	0.265	3
Na	12.461	6.971	1.79	No	0.082	3
Si	10.085	6.760	1.49	No	0.12	3

Notes:
MCC-1 = Materials Characterization Center (MCC-1)
CI = Confidence Interval
Mean releases become significantly different at a confidence interval of 1-alpha.
n = numbers of replicate leachate test samples.

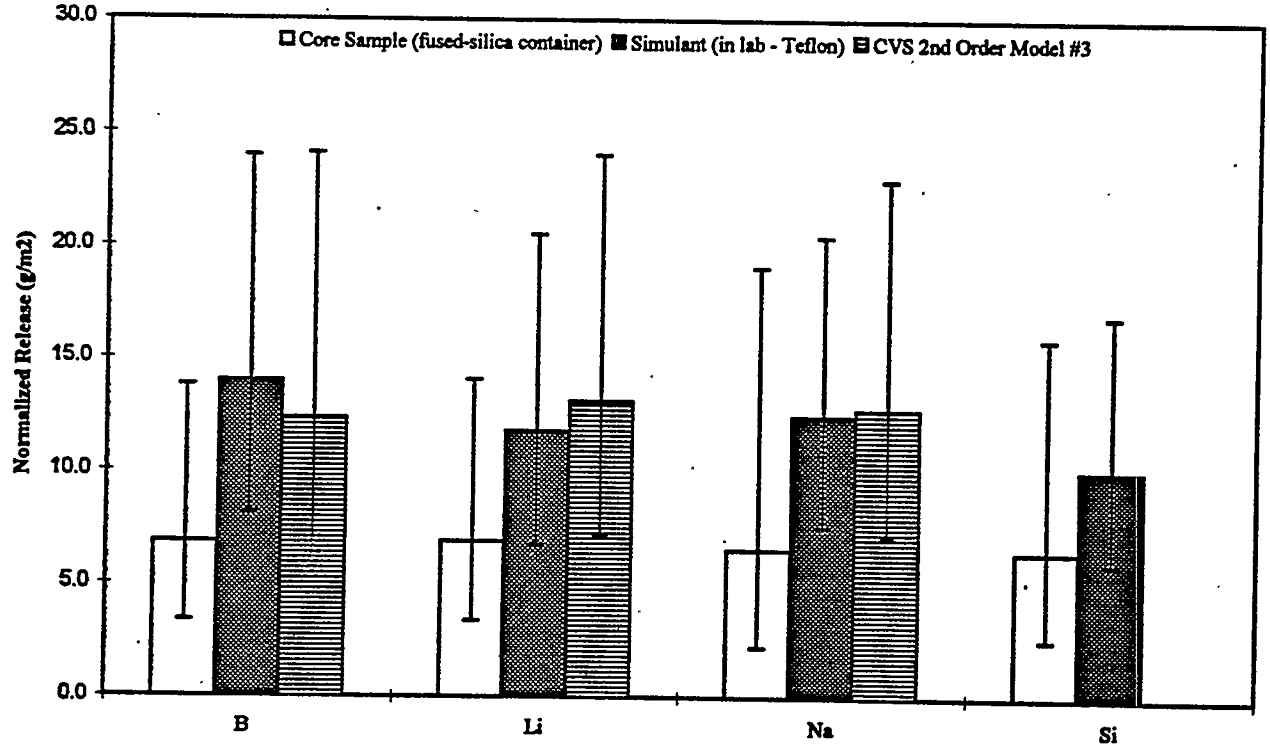


Figure 4.24. MCC-1 Results and Model Predictions for 101-AZ Core 1 and Simulant (Error Bars = Estimated 95% Confidence Interval)

4.5.2.2 101-AZ Core 2 and Simulant

Results from MCC-1 testing of 101-AZ Core 2 are provided in Table 4.31. The durability of the glass as measured by MCC-1 boron release was 6.236 g/m^2, which was approximately 8 to 14 times more durable than EA glass. Normalized releases were similar for B, Li, and Si (ranging from 6.17 g/m^2 to 6.45 g/m^2) and 4.86 g/m^2 for Na. The percent RSDs were quite high, ranging from 14% to 43%. The standard deviations were based on quadruplicate glass, triplicate leachate, and duplicate leachate blank analyses. Confidence intervals were based on a sample size of three leachates.

Results from MCC-1 testing of 101-AZ Core 2 simulant are provided in Table 4.32. Results are for two simulant samples tested in the laboratory in Teflon containers. In-cell MCC-1 tests were not performed on the core sample simulants and were therefore not available for direct comparison. The practice of batching core sample simulants and testing them in the same test as the core sample was not begun until after these tests were performed. Elemental analysis was not performed on the simulant glass; normalized releases are calculated from the "as-batched" composition. Assumed glass analytical uncertainty and limited replicate resulted in conservative percent RSDs and confidence intervals.

Figure 4.25 compares MCC-1 elemental releases for 101-AZ Core 2 glass, simulant, and model prediction. General observations of the data are as follows: (1) the simulant releases and model predictions matched fairly well; (2) the core sample releases were lower than corresponding simulant releases and model predictions; (3) simulant confidence limits were large due to the limited sample size; and (4) core sample confidence limits were large due to variability in the analyzed leachates.

Table 4.33 compares mean releases between 101-AZ Core 2 and its in-laboratory tested simulant. Elemental releases from the simulant were 124% greater for B, 85% greater for Li, 158% greater for Na, and 63% greater for Si than corresponding releases in the actual waste glass. A statistical difference was found for each of the four elements, to a 95% confidence interval between the simulant and core sample. The statistical comparison provided in Table 4.30 has limited meaning given that the two tests being compared were performed at different times and under different conditions (i.e., in the hot cell versus in the laboratory, and in fused-silica containers versus in Teflon containers).

4.5.2.3 102-AZ Core 1 and Simulant

Because of insufficient sample quantities of 102-AZ Core 1 glass, an MCC-1 test was not performed on the actual waste glass. An MCC-1 durability test was performed on the 102-AZ Core 1 simulant glass; the results are provided in Table 4.34. Comparisons with model predictions are discussed in Section 4.5.2.4.

Table 4.31. MCC-1 Leach Test Results for 101-AZ Core #2 (Hot Cell Test #1)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	6.326	17.37	1.835	2.267	1.404	6.266	9.649	4.070
Li	6.452	13.92	1.858	2.204	1.513	6.413	9.062	4.538
Na	4.855	43.25	1.568	2.642	0.493	4.795	14.045	1.637
Si	6.172	20.63	1.811	2.323	1.298	6.114	10.208	3.662

CI = Confidence Interval
Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.05/2)} = 4.303$).
MCC-1 = Materials Characterization Center (MCC-1)
Exp Transformed = retransformed data from ln form to original form

Table 4.32. MCC-1 Leach Test Results for 101-AZ Core #2 Simulant (Lab Test #9 in Teflon)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	14.180	5.45	2.667	2.652	3.142	2.162	14.178	23.141
Li	11.962	5.00	2.482	2.482	2.931	2.033	11.962	18.746
Na	12.529	5.00	2.528	2.528	2.977	2.079	12.529	19.635
Si	10.074	5.00	2.310	2.310	2.759	1.861	10.074	15.787

CI = Confidence Interval
Confidence interval is based on a sample size of 2 (i.e., $t_{(2-1,0.05/2)} = 12.706$).
MCC-1 = Materials Characterization Center (MCC-1)
Exp Transformed = retransformed data from ln form to original form

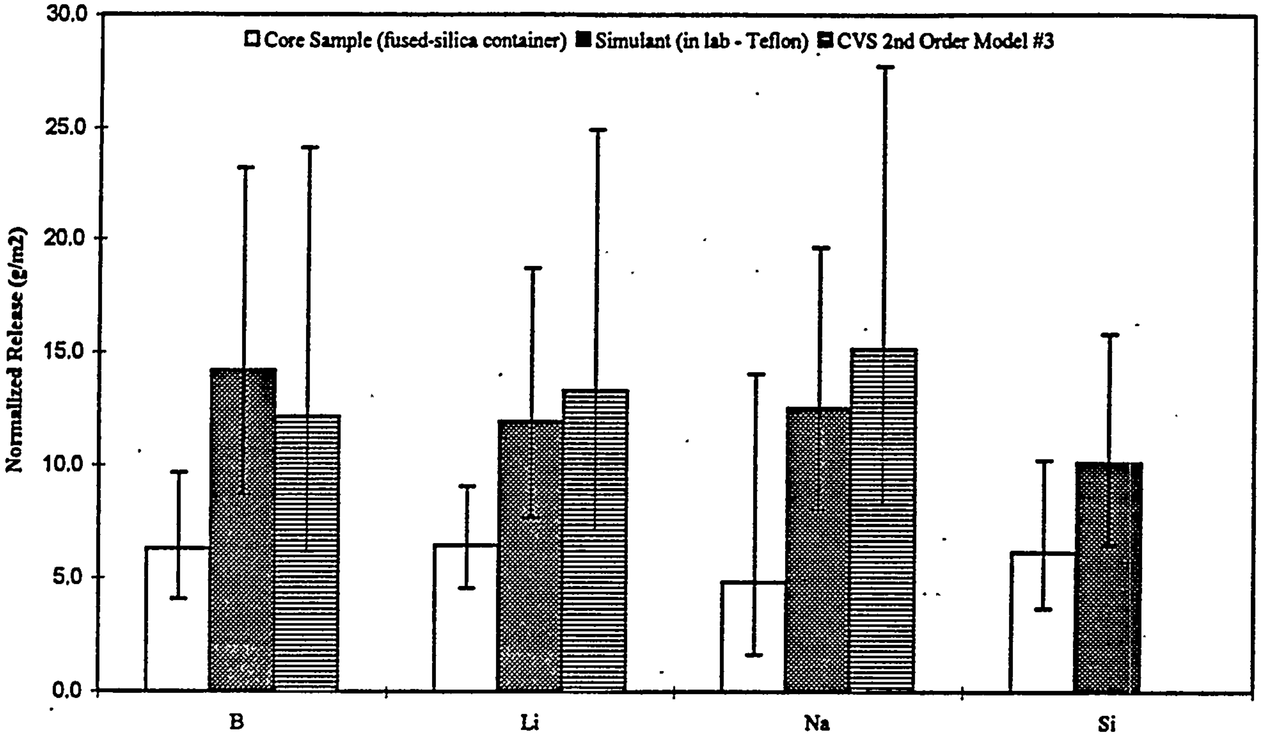


Figure 4.25. MCC-1 Results and Model Predictions for 101-AZ Core 2 and Simulant (Error Bars = Estimated 95% Confidence Interval)

Table 4.33. MCC-1 Durability Comparison of 101-AZ Core 2 Simulant to 101-AZ Core 2 Radioactive Glass

	Mean Normalized Release (g/m^2)					
	Simulant	Radioactive	Ratio Simulant/ Radioactive	Statistical Difference at 95% CI	Alpha	Degrees of Freedom (n1+n2-2)
B	14.180	6.326	2.24	Yes	0.016	3
Li	11.962	6.452	1.85	Yes	0.004	3
Na	12.529	4.855	2.58	Yes	0.030	3
Si	10.074	6.172	1.63	Yes	0.025	3

Notes:
MCC-1 = Materials Characterization Center (MCC-1)
CI = Confidence Interval
Mean releases become significantly different at a confidence interval of 1-alpha.
n = number of replicate leachate samples.

Table 4.34. MCC-1 Leach Test Results for 102-AZ Core 1 Simulant MCC-1 (Lab Test #12 in Teflon)

			------Ln Transformed------			------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Mean Ln Normalized Release ln(g/m2)	Upper Bound (95% CI) ln(g/m2)	Lower Bound (95% CI) ln(g/m2)	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
B	14.829	9.92	2.696	2.890	2.501	14.815	17.995	12.198
Li	14.919	9.24	2.702	2.883	2.521	14.908	17.869	12.437
Na	14.216	9.50	2.654	2.840	2.467	14.204	17.112	11.790
Si	13.025	9.52	2.566	2.753	2.379	13.015	15.685	10.799

CI = Confidence Interval

Confidence interval is based on a sample size of 2 (i.e., $t_{(2-1,0.05/2)} = 12.706$).

MCC-1 = Materials Characterization Center (MCC-1)

Exp Transformed = retransformed data from ln form to original form

4.5.2.4 Comparison of Core Sample and Simulant MCC-1 Results to Model Predictions

Glass durability models as developed by Hrma, Piepel et al. (1994) were used to generate MCC-1 normalized release predictions and statistics for each of the core sample glass compositions and the CVS-IS-HW39-4 composition. Model predictions for these compositions are provided in Appendix A, Tables A.20 through A.24. Results from four different models are included as follows: 1st order; 2nd order model #1; 2nd order model #2; and 2nd order model #3. The latter model, 2nd order model #3, provided the best fit for CVS glasses reported by Hrma and Piepel was selected for comparison with core sample results.

Figures 4.26, 4.27, and 4.28 provide a comparison of model predictions to results from actual waste glasses, simulant glasses of same composition, and environmental assessment glass. The data are plotted as measured ln mean normalized release to predicted ln normalized release. The diagonal line represents the ideal fit between measured and predicted results. The error bars represent 95% prediction intervals for the model and are based on a sample size of two. The confidence intervals for means of larger sample sizes, such as the radioactive glasses with three replicates each, are slightly smaller than shown. Boron, Li, and Na results are provided in Figures 4.26, 4.27, and 4.28 respectively. A characteristic of the model verified during validation is that it tended to overpredict release in highly durable glasses and underpredict release in low durability glasses. For B, measured results for all tests shown, except one core sample test and one EA glass test, were within the 95% prediction intervals of the model. For Li, both core sample tests and one EA glass test were outside the 95% prediction intervals of the model. For Na, both core sample tests and one EA glass tests were outside the 95% prediction intervals of the model. Where not specified, hot-cell tests were performed in fused-silica-lined containers and laboratory tests were performed in Teflon containers.

A summary by glass type of results fitting within the 95% prediction interval of the model is provided in Table 4.35. With the limited amount of data presented, the actual waste glass mean releases were within the models' 95% prediction interval only 17% of the time; the simulant glass mean releases were within the confidence interval 80% of the time.

Table 4.35. Summarized Comparison of PCT Model Prediction to Measured

(# of tests within prediction intervals/total # compared)

	Boron	Lithium	Sodium	Total
Core Sample Glasses	(1/2)	(0/2)	(0/2)	(1/6)
Core Sample Simulants	(3/3)	(3/3)	(3/3)	(9/9)
Environmental Assessment Glass	(1/2)	(1/2)	(1/2)	(3/6)

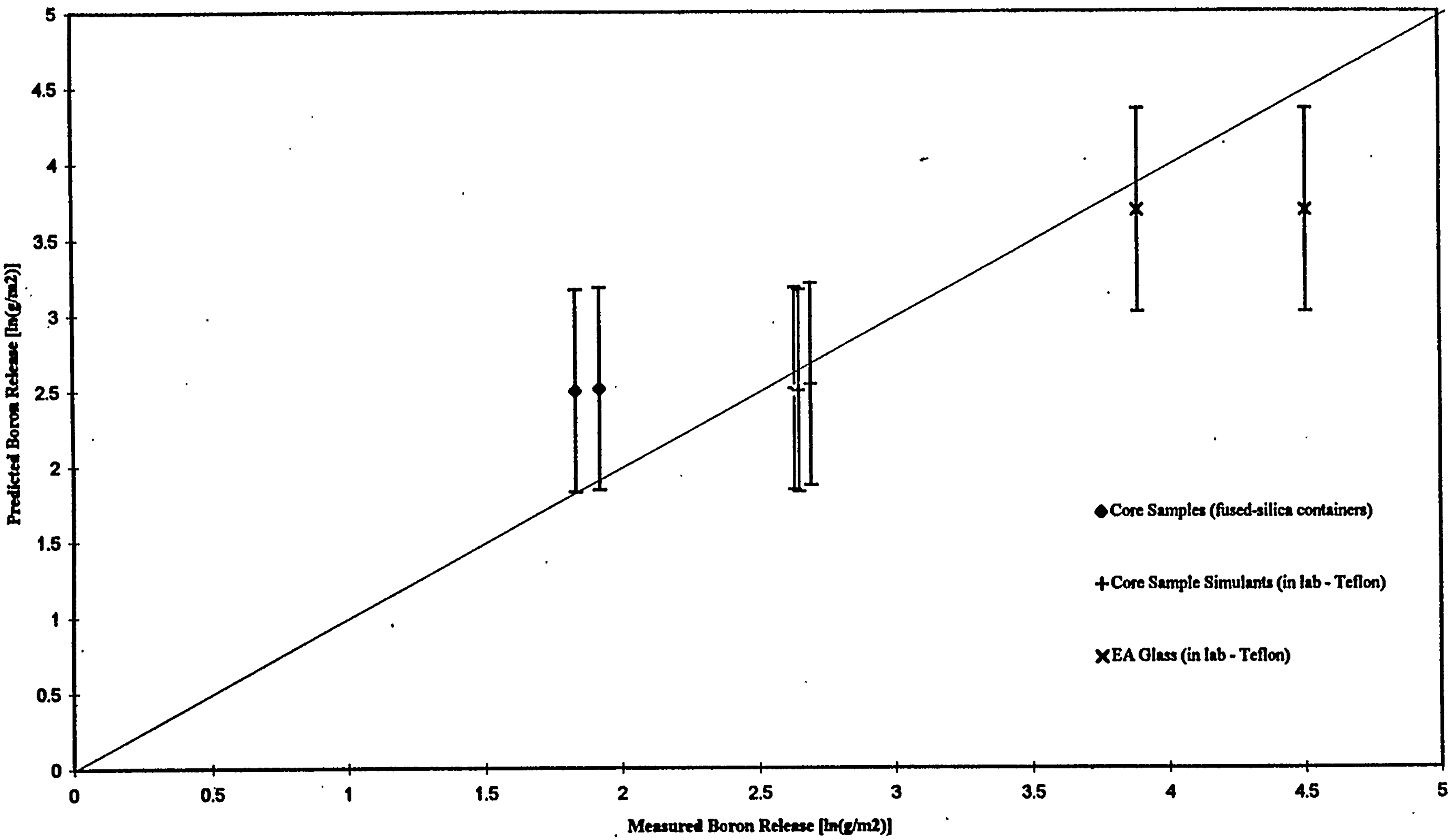


Figure 4.26. Measured Versus Predicted MCC-1 Boron Release for Core Sample, Simulant, and Standard Glasses

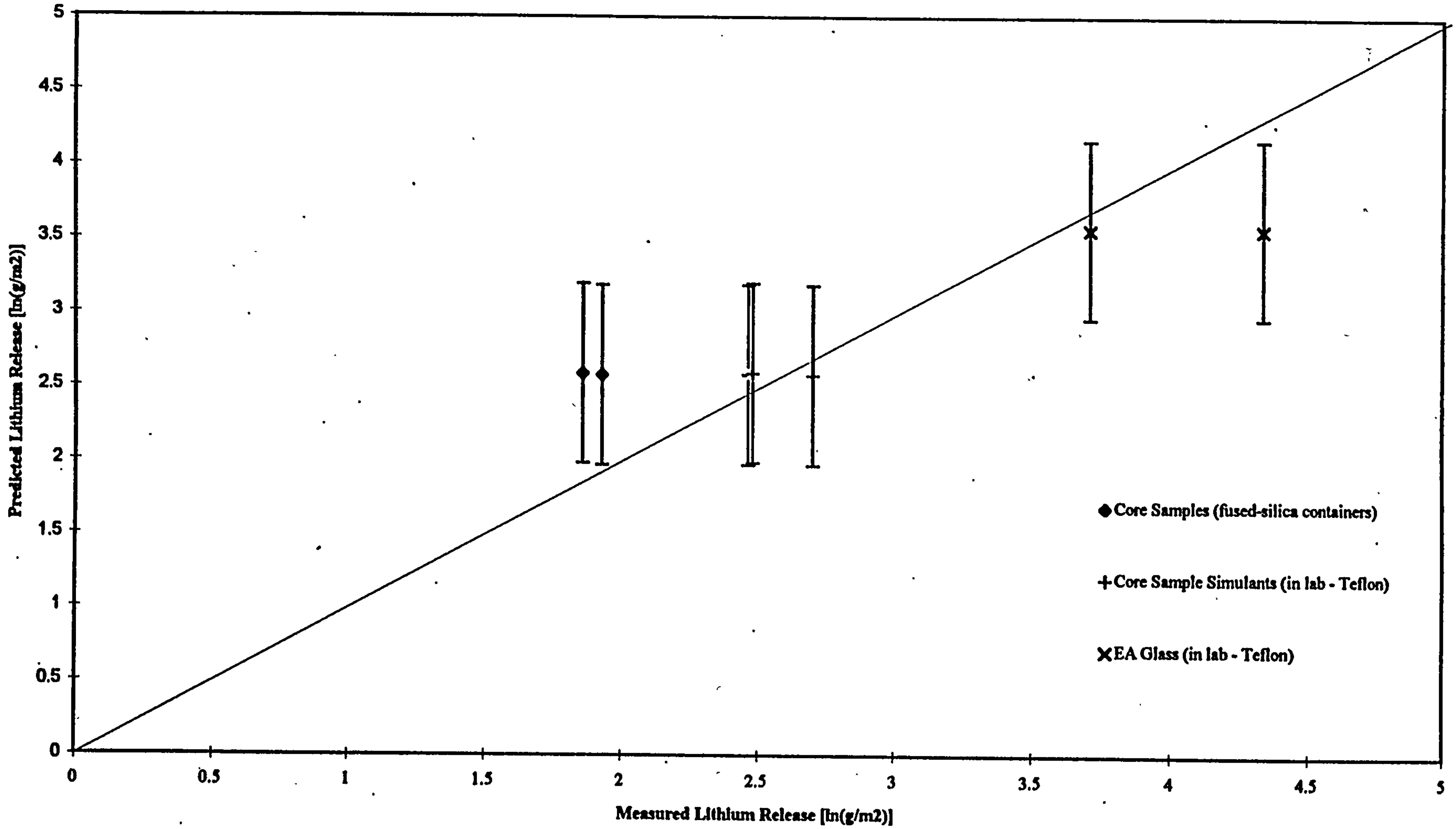


Figure 4.27. Measured Versus Predicted MCC-1 Lithium Release for Core Sample, Simulant, and Standard Glasses

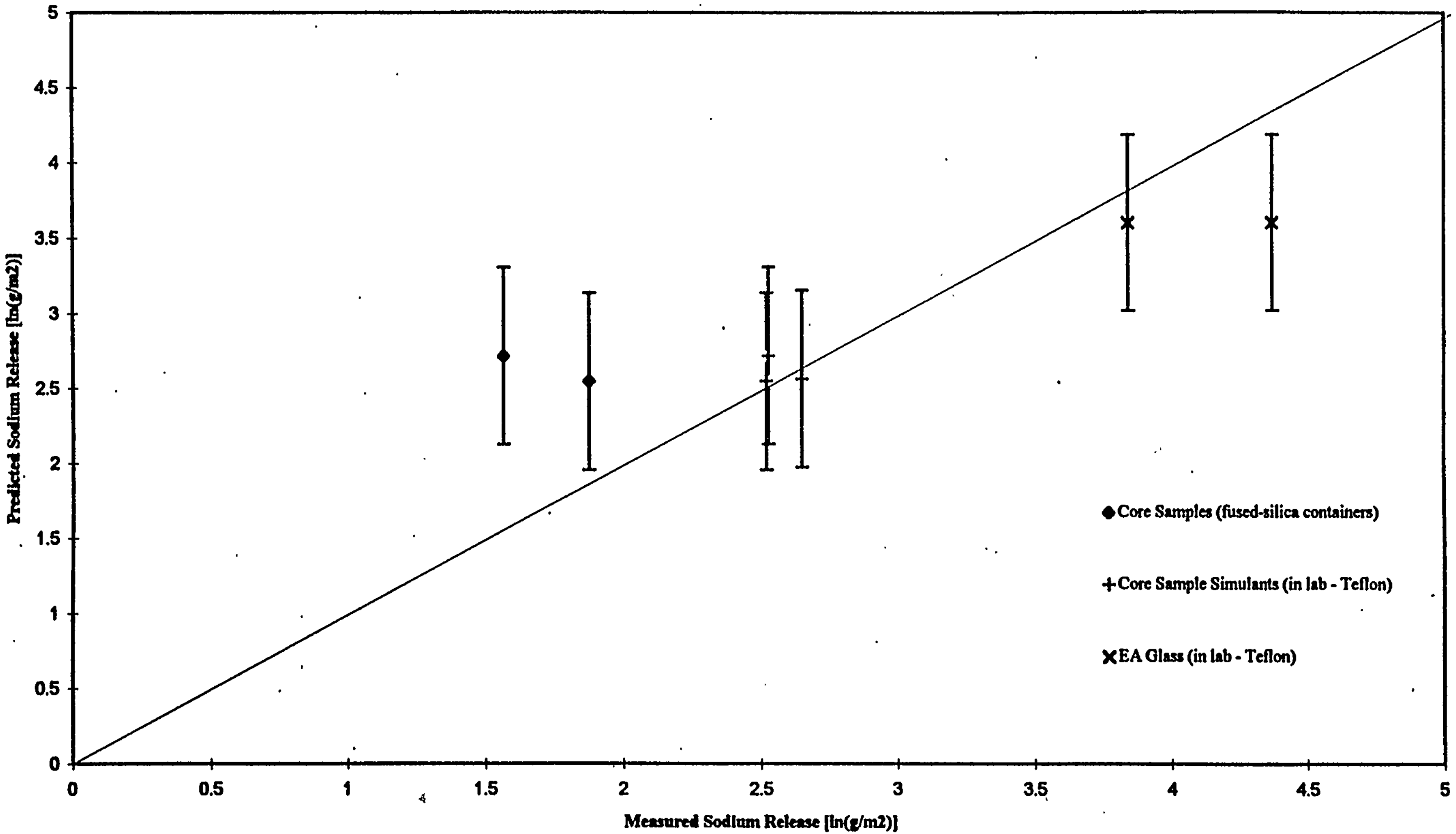


Figure 4.28. Measured Versus Predicted MCC-1 Sodium Release for Core Sample, Simulant, and Standard Glasses

4.5.3 PCT Radiochemical Releases

Radiochemical analysis was performed on each of the triplicate leachates for the three radioactive glasses. Leachates from nonradioactive glass samples (usually the in-cell prepared samples) were analyzed as radiochemical blanks. Following each leach test, acid strips were performed on the radioactive glass leach vessels and radiochemical blank vessels. The vessels were rinsed with deionized water to remove the glass, an amount of 1 vol% HNO_3 equal in volume to the original leachant was added, and the solution was maintained at 90°C for at least 12 hours. Radiochemical analysis was performed on the acid strip solutions to determine quantities of radionuclides precipitated onto the stainless-steel surfaces. Analytical results from these samples are included in detail in Appendix B, Tables B.4 through B.6.

4.5.3.1 101-AZ Core 1

PCT radiochemical releases for 101-AZ Core 1 leachate and acid strip are provided in Table 4.36. The normalized releases are compared by ratio to the B release of 0.130 g/m^2. The normalized boron release is considered to be a good measure of the glass dissolution extent, because B remains in solution and does not precipitate in secondary mineral phases and B is released through dissolution of the glass network although it is generally released to nearly the same depth of alkali metals. Therefore, a ratio of total radionuclide release to B release significantly different than one indicates precipitation of the radionuclide into secondary mineral phases; selective leaching through a diffusion process; retention in the altered layer or analytical error. The estimated percent RSD and 95% confidence intervals for the retransformed mean ln normalized releases are provided as a measure of the estimated minimum and maximum error associated with the measurements. The percent RSD for the leachate ranged from 9.3% for total U to 44.3% for Cm-243/244; the acid strip ranged from 25.3% for Co-60 to 70.1% for Sb-125.

Comparison of the ratios to boron in the leachate, acid strip, and total provide an indication of which radionuclides may have enhanced performance (i.e., mobility retardation) due to secondary phase formation and reduction plate-out. General observations of the data are as follows: (1) U, Tc, Pu, Cm, Sb, and Cs were at least fairly soluble in the leachate (i.e., leachate release within a factor of 3 of B); (2) of these elements, Pu was the only one that plates out on the container in substantial amounts; (3) Sr, Am, Co, Ru, Ce, and Eu were at least an order of magnitude less soluble in the leachate, and (4) only 2% to 6% of the amount of these elements released from the glass was retained in solution. Comparing the results from different isotopes of the same element (i.e., Pu, Cs, and Eu) and isotopes measured by two different techniques (i.e., Am-241) provides an additional indication of accuracy. Redundant measurements of Pu, Eu and Am show excellent agreement. Differences in the Cs results were greater than estimated by the error estimates, but were within a factor of 2.5. The excessive error could have been contributed from the glass or leachate analysis.

Figure 4.29 is a graphical representation of data in Table 4.36. Relative normalized releases in the leachate, acid strip, and total are shown with respect to the normalized B release. Similar observations as identified above are also evident in the figure, with additions. Total U was sufficiently below B to suggest that some of the U was consumed in secondary mineral phases or retained in the glass alteration layer. The discrepancy between Cs-134 and Cs-137 could lead to the conclusion that Cs may or may not be solubility limited. The absence of error bars on Ru-106 is because of the three available samples, only one sample was above detection limits.

Table 4.36. PCT Radionuclide Releases in Solution for 101-AZ Core #1 (Hot Cell Test #4)

	Release Measured in Leachate						Release Measured in Acid Strip						Total Measured Release				
				------Exp Transformed------						------Exp Transformed------					------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)	Mean Normalized Release (g/m2)	%RSD	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)	Mean Normalized Release (g/m2)	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
Total U	0.039	9.3	0.30	0.039	0.049	0.031	0.007	32.7	0.05	0.007	0.015	0.003	0.046	0.354	0.046	0.064	0.034
Tc-99	0.876	74.6	6.74	0.864	5.508	0.135							0.876	6.736	0.864	5.508	0.135
Sr-90	0.004	14.9	0.03	0.004	0.006	0.003	0.004	52.6	0.03	0.003	0.012	0.001	0.008	0.059	0.007	0.018	0.004
Pu-238	0.058	19.2	0.44	0.057	0.092	0.036	0.109	28.7	0.84	0.106	0.217	0.052	0.166	1.278	0.164	0.309	0.088
Pu-239/240	0.058	12.1	0.44	0.058	0.078	0.043	0.099	25.4	0.76	0.097	0.183	0.052	0.156	1.202	0.155	0.260	0.094
Cm-243/244	0.005	44.3	0.04	0.005	0.016	0.002	0.010	51.9	0.08	0.009	0.034	0.003	0.015	0.119	0.015	0.050	0.004
Am-241	0.004	19.7	0.03	0.004	0.007	0.003	0.005	55.0	0.04	0.005	0.018	0.001	0.009	0.071	0.009	0.025	0.004
Co-60	0.008	13.4	0.06	0.008	0.011	0.006	0.009	25.3	0.07	0.009	0.017	0.005	0.017	0.131	0.017	0.028	0.010
Ru-106							0.0005						0.0005	0.0038			
Sb-125	0.102	8.7	0.78	0.101	0.126	0.082	0.002	70.1	0.02	0.002	0.010	0.000	0.104	0.798	0.103	0.136	0.082
Cs-134	0.105	19.0	0.81	0.104	0.167	0.065	0.006	43.4	0.04	0.005	0.015	0.002	0.111	0.851	0.109	0.182	0.067
Cs-137	0.043	6.2	0.33	0.043	0.050	0.037	0.003	66.8	0.02	0.003	0.015	0.001	0.046	0.355	0.046	0.065	0.037
Ce-144							0.005	59.6	0.04	0.004	0.020	0.001	0.005	0.038	0.004	0.020	0.001
Eu-154	0.004	19.7	0.03	0.004	0.006	0.002	0.005	44.6	0.04	0.005	0.015	0.002	0.009	0.071	0.009	0.021	0.004
Eu-155	0.004	17.2	0.03	0.004	0.006	0.003	0.006	47.8	0.04	0.005	0.017	0.002	0.010	0.074	0.009	0.023	0.004
Am-241	0.003	34.9	0.02	0.003	0.007	0.001	0.004	53.5	0.03	0.003	0.012	0.001	0.007	0.052	0.006	0.020	0.002

* Ratio of radionuclide mean normalized release to boron mean normalized release.

CI = Confidence Interval

Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.95/2)}$ = 4.303).

PCT = Product Consistency Test

Exp Transformed = retransformed data from ln form to original form

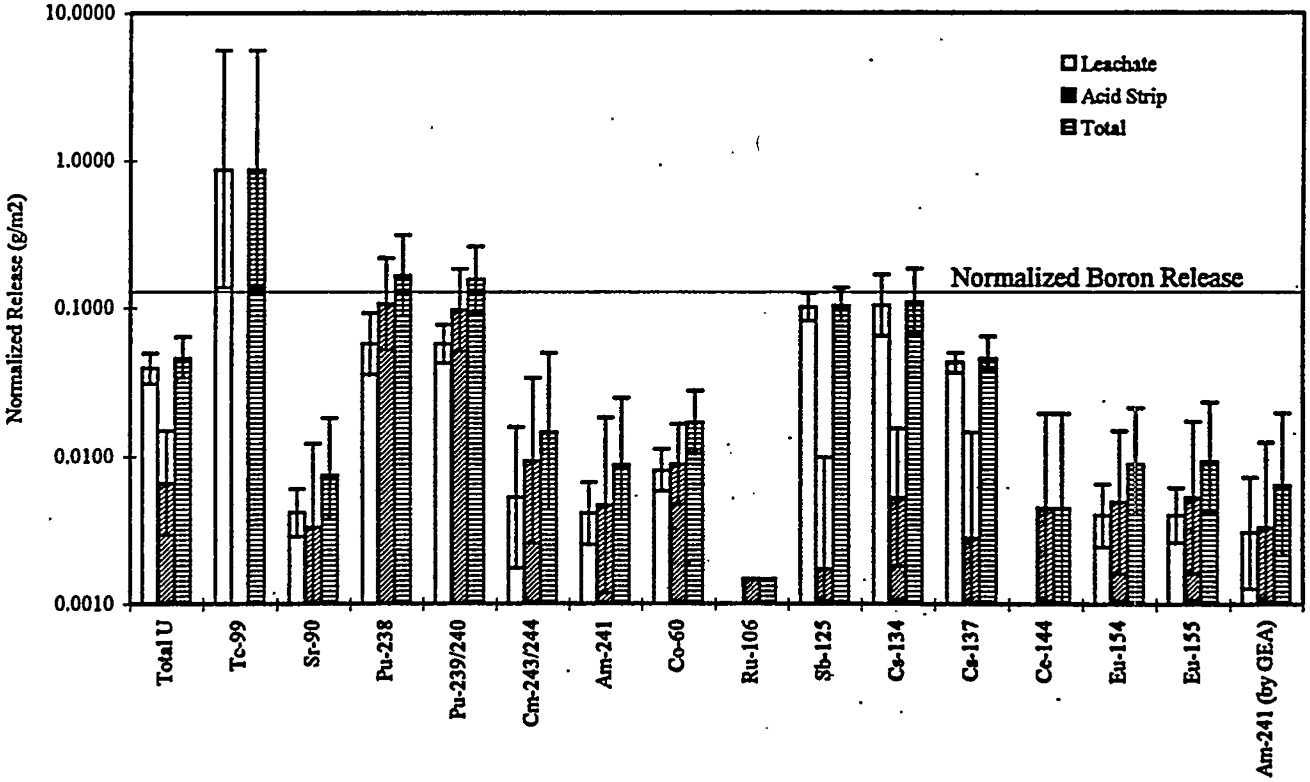


Figure 4.29. PCT Radionuclide Releases for 101-AZ Core 1

4.5.3.2 101-AZ Core 2

PCT radiochemical releases for 101-AZ Core 2 leachate and acid strip are provided in Table 4.37. The normalized releases are compared by ratio to the B release of 0.203 g/m^2. The percent RSD for the leachate ranged from 45.6% for Am-241 to 230% for Pu-238; the acid strip ranged from 10.8% for Pu-239/240 to 90.2% for Cm-243/244. A potential reason for the excessive error was a problem experienced during shipping of the samples. Some of the sample containers used for leachate and acid strip samples leaked during shipping, creating a potential for cross-contamination. Radiochemical blank samples were at least an order of magnitude higher than the blanks in subsequent tests, yet were at least order of magnitude lower than the leachates for most analytes. In addition, due to suspect data acid strip sample #2 was omitted from the calculations. Acid strip #2 was approximately 2 orders of magnitude higher for the soluble radionuclides and 4 orders of magnitude higher for the less soluble radionuclides. The most likely reason for this discrepancy is that not all of the glass was removed before the acid strip of sample #2. Special precautions were taken to prevent these two problems in hot-cell tests #4 and #5.

Comparison of the ratios to boron in the leachate, acid strip, and total provide an indication of which radionuclides may have enhanced performance (i.e., mobility retardation). General observations of the data are as follows: (1) U, Tc, Pu, Cm, Sb, and Cs were at least fairly soluble in the leachate (i.e., same order of magnitude as B); (2) of these elements, Pu was the only one that plates out on the container in substantial amounts; (3) Sr, Am, Co, Ru, Ce, and Eu were at least an order of magnitude less soluble in the leachate than B; and (4) only 0.2% to 6% of the amount released from the glass for these elements was retained in solution. Comparing the results from different isotopes of the same element (i.e., Pu, and Eu) and isotopes measured by two different techniques (i.e., Am-241) provides an additional indication of accuracy. Redundant measurements of Eu and Am show very good agreement, whereas the Pu results for leachates differed by a factor of 6.

Figure 4.30 represents data in Table 4.37. Relative normalized releases in the leachate, acid strip, and total are shown with respect to the normalized B release. Similar observations as identified previously are also evident in the figure, with a few additions. U, Cm, Sb, and Cs were sufficiently below B to suggest reduced solubility. Variability in the Pu results would suggest that Pu may or may not be solubility limited. The absence of error bars on certain radionuclides is because only one sample was above detection limits.

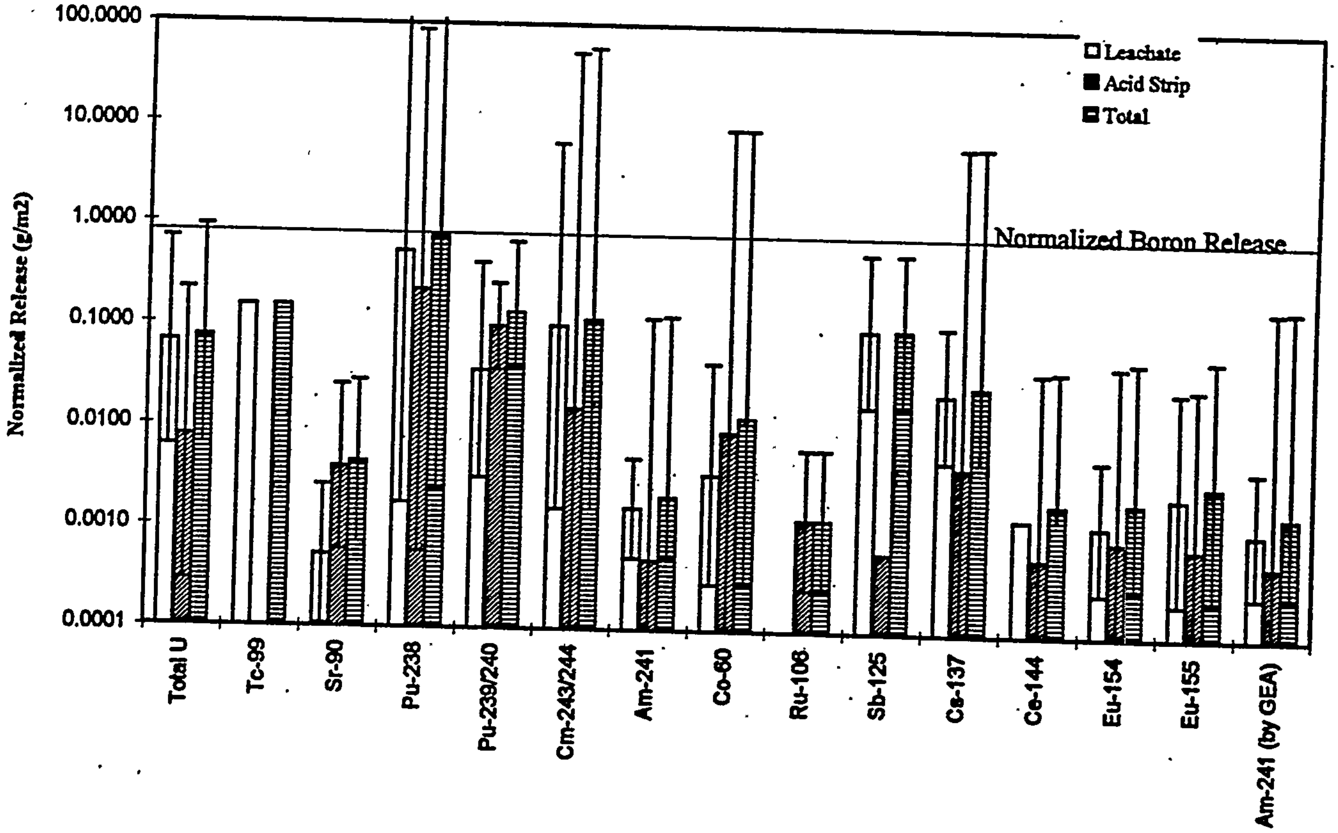


Figure 4.30. PCT Radionuclide Releases for 101-AZ Core 2

Table 4.37. PCT Radionuclide Releases in Solution for 101-AZ Core #2 (Hot Cell Test #3)

	Release Measured in Leachate						Release Measured in Acid Strip						Total Measured Release				
				------Exp Transformed------						------Exp Transformed------					------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)	Mean Normalized Release (g/m2)	%RSD	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)	Mean Normalized Release (g/m2)	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
Total U	0.101	95.2	0.50	0.066	0.705	0.006	0.005	37.0	0.03	0.008	0.219	0.00028	0.106	0.52	0.074	0.924	0.007
Tc-99	0.050		0.25										0.050	0.25			
Sr-90	0.001	64.2	0.00	0.001	0.003	0.000	0.003	21.0	0.01	0.004	0.026	0.00059	0.003	0.016	0.004	0.028	0.001
Pu-238	0.291	230.0	1.43	0.531	160.977	0.002	0.168	66.3	0.83	0.224	86.801	0.00058	0.459	2.26	0.755	247.778	0.002
Pu-239/240	0.046	97.8	0.23	0.036	0.413	0.003	0.066	10.8	0.32	0.099	0.261	0.03725	0.112	0.55	0.135	0.674	0.040
Cm-243/244	0.056	167.3	0.28	0.101	6.448	0.002	0.013	90.2	0.07	0.016	51.208	0.00000	0.069	0.34	0.116	57.656	0.002
Am-241	0.002	45.6	0.01	0.002	0.005	0.001	0.000	61.1	0.00	0.001	0.121	0.00000	0.002	0.010	0.002	0.126	0.001
Co-60	0.008	101.2	0.04	0.004	0.044	0.000	0.007	76.5	0.04	0.009	9.106	0.00001	0.015	0.08	0.013	9.150	0.000
Ru-106							0.0009	17.9	0.004	0.001	0.007	0.00026	0.001	0.004	0.001	0.007	0.000
Sb-125	0.134	69.5	0.66	0.098	0.551	0.017	0.0002		0.001				0.134	0.66	0.099	0.552	0.018
Cs-134																	
Cs-137	0.027	61.3	0.13	0.023	0.107	0.005	0.004	80.9	0.02	0.004	6.351	0.00000	0.031	0.15	0.028	6.458	0.005
Ce-144	0.0005		0.002				0.000	47.0	0.00	0.001	0.039	0.00001	0.001	0.004	0.002	0.041	0.001
Eu-154	0.001	60.6	0.01	0.001	0.006	0.0003	0.001	44.0	0.00	0.001	0.046	0.00002	0.002	0.010	0.002	0.052	0.000
Eu-155	0.002	96.9	0.01	0.002	0.027	0.0002	0.001	40.4	0.00	0.001	0.029	0.00002	0.002	0.011	0.003	0.056	0.000
Am-241	0.001	57.4	0.01	0.001	0.005	0.0003	0.000	64.6	0.00	0.001	0.173	0.00000	0.002	0.008	0.002	0.178	0.000

* Ratio of radionuclide mean normalized release to boron mean normalized release.

CI = Confidence Interval

Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.05/2)}$ =4.303) for leachate release and 2 (i.e., t(2-1,0.05/2) =12.706) for acid strip release.

Acid strip sample #2 was believed to have been contaminated by residual glass left in the container, due to excessive levels of radionuclides. This data was not included in acid strip release calculations.

PCT = Product Consistency Test

Exp Transformed = retransformed data from ln form to original form

4.5.3.3 102-AZ Core 1

PCT radiochemical releases for 102-AZ Core 1 leachate and acid strip are provided in Table 4.38. The normalized releases are compared by ratio to the B release of 0.211 g/m^2. The estimated percent RSD and 95% confidence intervals for the retransformed mean ln normalized releases are provided as a measure of the estimated minimum and maximum error associated with the measurements. The percent RSD for the leachate ranged from 3.2% for Eu-155 to 34.4% for Tc-99 and for the acid strip ranged from 29.2% for Total U to 119.4% for Co-60.

General observations of the data are as follows: (1) U, Tc, Pu, Sb, and Cs were at least fairly soluble in the leachate (i.e., same order of magnitude as B); (2) of these elements, Pu was the only one that plates out on the container in substantial amounts; (3) Sr, Am, Co, and Eu were at least an order of magnitude less soluble in the leachate than B; and (4) only 2% to 7% of the amount released from the glass for these elements was retained in solution. Comparing the results from different isotopes of the same element (i.e., Pu and Eu) provides an additional indication of accuracy. Redundant measurements of Pu and Eu show excellent agreement.

Figure 4.31 is a graphical representation of data in Table 4.38. Relative normalized releases in the leachate, acid strip, and total are shown with respect to the normalized B release. Similar observations as identified previously are also evident in the figure, with one addition. U, Pu, and Cs were sufficiently below B to suggest that each of these elements were involved in secondary phase precipitations. The absence of error bars on the Tc-99 acid strip is because only one sample was above detection limits.

4.5.4 MCC-1 Radiochemical Releases for 101-AZ Core 1 and 101-AZ Core 2

Radiochemical analysis was performed on a single MCC-1 leachate sample from 101-AZ Core 1 and 101-AZ Core 2 and on one blank from the in-cell test. Acid strips were not performed on the fused-silica leach containers. Analytical results from these samples are included in detail in Appendix B, Tables B.4 through B.6.

Table 4.38. PCT Radionuclide Releases in Solution for 102-AZ Core #1 (Hot Cell Test #5)

	Release Measured in Leachate						Release Measured in Acid Strip						Total Measured Release				
				------Exp Transformed------						------Exp Transformed------					------Exp Transformed------		
	Mean Normalized Release (g/m2)	%RSD	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)	Mean Normalized Release (g/m2)	%RSD	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)	Mean Normalized Release (g/m2)	Ratio to Boron*	Mean Ln Normalized Release (g/m2)	Upper Bound (95% CI) (g/m2)	Lower Bound (95% CI) (g/m2)
Total U	0.083	8.4	0.39	0.083	0.102	0.067	0.010	29.2	0.05	0.010	0.020	0.005	0.093	0.44	0.093	0.122	0.072
Tc-99	0.224	34.4	1.06	0.222	0.522	0.095	0.006		0.03				0.230	1.09	0.239	0.539	0.112
Sr-90	0.004	10.5	0.02	0.004	0.005	0.003	0.002	44.5	0.01	0.001	0.004	0.000	0.005	0.02	0.005	0.009	0.003
Pu-238	0.030	13.1	0.14	0.030	0.041	0.022	0.021	32.5	0.10	0.020	0.045	0.009	0.051	0.24	0.050	0.086	0.031
Pu-239/240	0.028	11.3	0.13	0.027	0.036	0.021	0.016	44.6	0.08	0.015	0.047	0.005	0.044	0.21	0.043	0.083	0.026
Co-60	0.015	9.4	0.07	0.015	0.019	0.012	0.003	119.4	0.01	0.003	0.060	0.000	0.018	0.09	0.018	0.078	0.012
Sb-125	0.170	3.8	0.80	0.170	0.186	0.154	0.0003		0.001				0.170	0.80	0.170	0.187	0.155
Cs-137	0.023	12.1	0.11	0.023	0.031	0.017							0.023	0.11	0.023	0.031	0.017
Eu-154	0.013	5.7	0.06	0.013	0.016	0.012	0.002	63.8	0.01	0.002	0.008	0.000	0.015	0.07	0.015	0.023	0.012
Eu-155	0.013	3.2	0.06	0.013	0.014	0.012	0.002	60.0	0.01	0.001	0.006	0.000	0.015	0.07	0.015	0.021	0.012
Am-241	0.013	4.9	0.06	0.013	0.015	0.012	0.001	103.6	0.00	0.001	0.009	0.000	0.014	0.07	0.014	0.024	0.012

* Ratio of radionuclide mean normalized release to boron mean normalized release.

CI = Confidence Interval

Confidence interval is based on a sample size of 3 (i.e., $t_{(3-1,0.05/2)}$ = 4.303).

PCT = Product Consistency Test

Exp Transformed = retransformed data from ln form to original form

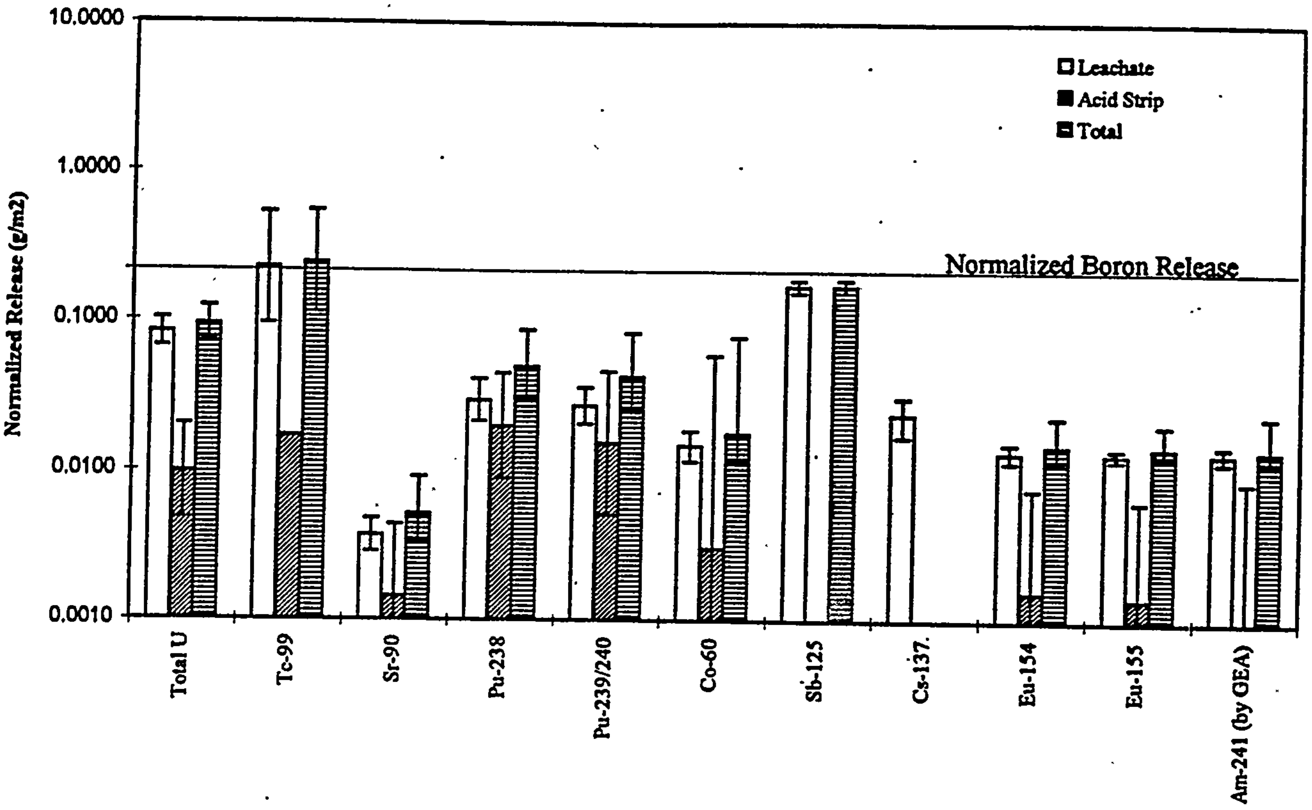


Figure 4.31. PCT Radionuclide Releases for 102-AZ Core 1

Actual and relative MCC-1 radiochemical releases for 101-AZ Core 1 and 101-AZ Core 2 leachates are provided in Table 4.39. The normalized releases are compared by ratio to the B release of the particular samples analyzed (i.e., 4.87 g/m^2 for sample #3 of 101-AZ Core 1 and 7.59 g/m^2 for sample #1 of 101-AZ Core 2). A ratio of radionuclide release to B release significantly different than one indicated the following: (1) retention of the radionuclide in the glass alteration layer; (2) selective leaching through a diffusion process; (3) absorption of silicate forming radionuclide onto the fused-silica liner; in secondary phase precipitation and/or (5) analytical error. The 28-day MCC-1 tests are typically well undersaturated and may be close to the forward reaction rate for glass dissolution.

General observations of the data are that Pu, Sb, and Cs had releases similar to or greater than B; U, Sr, and Np had releases less than B but within the same order of magnitude; and Am, Co, and Cm had releases at least an order of magnitude less than B. Comparing the results from different isotopes of the same element (i.e., Pu and Cs) provides an additional indication of accuracy. Two of the three pairs of data were within 30% and one differed by a factor of 3.5. Graphical representations of the data showing the same observations are provided in Figures 4.32 and 4.33.

Table 4.39. Summary of MCC-1 Radionuclide Releases Compared to Boron

	101-AZ Core #1		101-AZ Core #2	
	Mean Normalized Release (g/m2)	Ratio to Boron*	Mean Normalized Release (g/m2)	Ratio to Boron*
Total U	1.21	0.25	1.79	0.24
Sr-90	3.35	0.69	0.82	0.11
Pu-238	22.67	4.66	5.50	0.72
Pu-239/240	6.37	1.31	7.80	1.03
Cm-243/24	0.30	0.06	0.01	0.001
Am-241	0.02	0.003	0.01	0.001
Np-237	2.36	0.48	0.53	0.07
Co-60	0.46	0.09		
Sb-125	4.81	0.99	7.25	0.95
Cs-134	7.03	1.44		
Cs-137	4.87	1.00	7.07	0.93

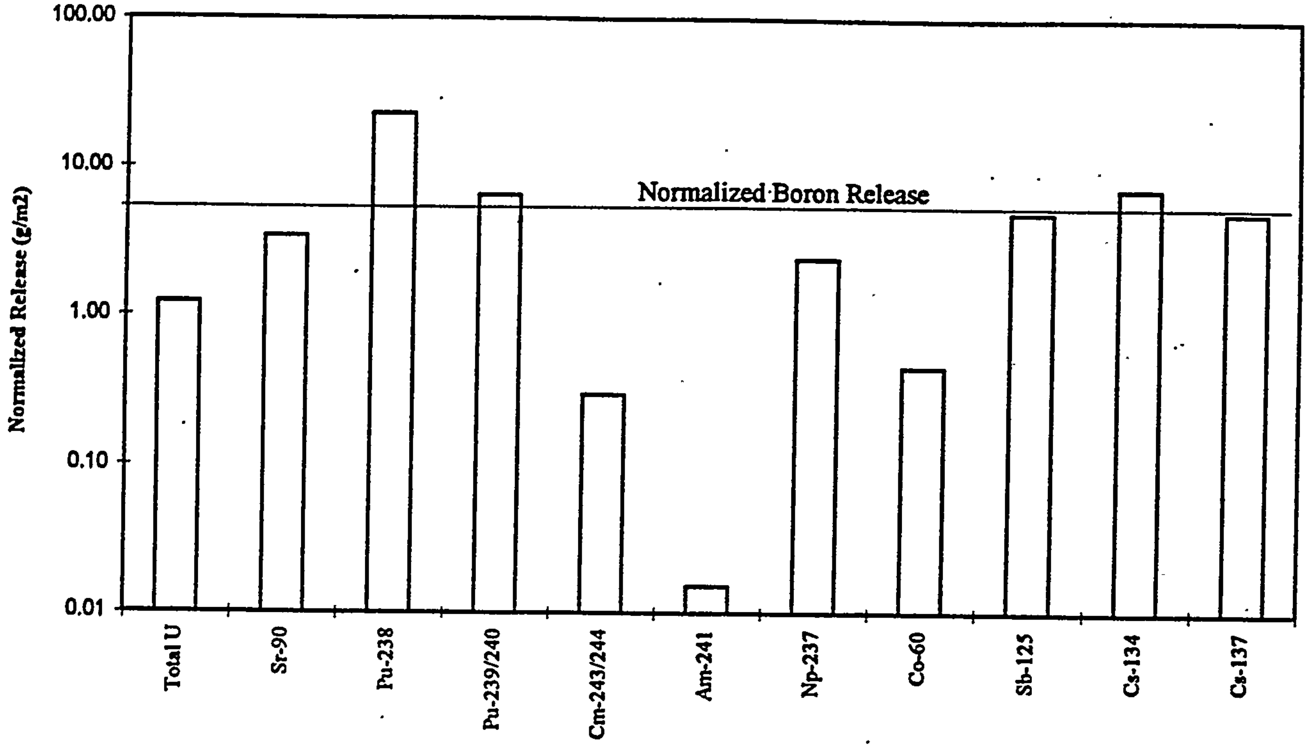


Figure 4.32. MCC-1 Radionuclide Normalized Releases for 101-AZ Core 1

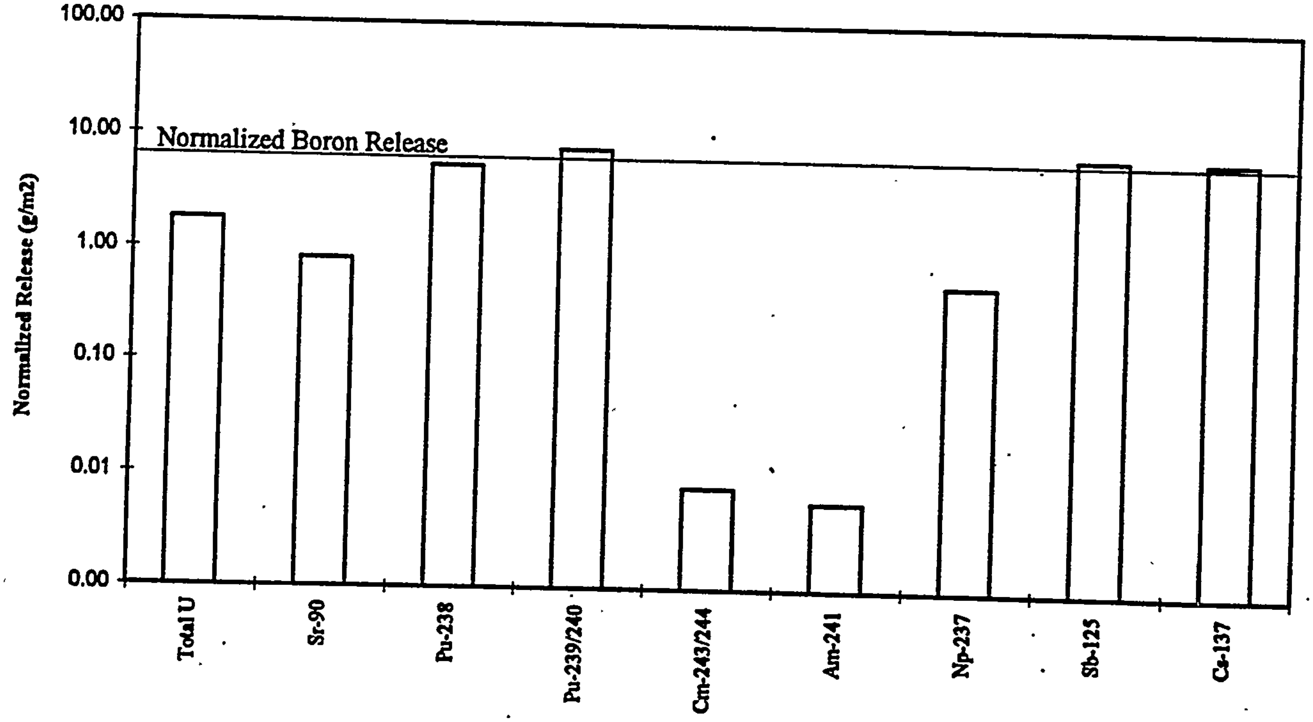


Figure 4.33. MCC-1 Radionuclide Normalized Releases for 101-AZ Core 2

4.5.5 PCT Anion Releases and pH

Leachates from all in-cell PCTs were analyzed for anions by IC. Complete PCT IC results are provided in Appendix Tables A.10 through A.18. Summarized results from these tests are provided in Table 4.40 and include averaged results from the core sample glasses, simulants and the reference glass from each test. Observations of the data are as follows: (1) F^- was present in minor quantities (2.5 ppm - 2.8 ppm) in the radioactive glasses but was not detected in simulants; (2) NO_2^-, NO_3^-, and Br^- were not present in appreciable amounts in any of the glasses; (3) PO_4^{-3} and SO_4^{-2} were present in leachates from core samples and their simulants in somewhat comparable amounts; (4) Cl^- was greater in the core samples than in their simulants; and (5) the reproducibility as indicated by comparison of the reference samples was very good.

Table 4.40. PCT Anion and pH Results for Core Sample and Simulant Glasses

			(μg/ml)					
Glass Type	pH	F^-	Cl^-	NO_2^-	Br^-	NO_3^-	PO_4^{-3}	SO_4^{-2}
101-AZ Core 1	7.49	2.5	3.0	<0.5	<0.25	<0.5	1.5	2.1
101-AZ Core 1 Simulant	9.90	<0.25	0.3	<0.5	<0.25	<0.5	2.6	1.8
101-AZ Core 2	9.85	NA	NA	NA	NA	NA	NA	NA
101-AZ Core 2 Simulant	10.06	NA	NA	NA	NA	NA	NA	NA
102-AZ Core 1	9.26	2.8	1.2	<0.5	<0.25	--	2.7	0.3
102-AZ Core 1 Simulant	9.61	<0.25	0.5	<0.5	<0.25	<0.5	3.2	0.7
CVS-IS-HW39-4-3	10.14	NA	NA	NA	NA	NA	NA	NA
CVS-IS-HW39-4-4	10.24	<2	1.1	0.5	0.25	<0.5	7.8	25.1
CVS-IS-HW39-4-5	10.23	<0.25	0.7	<0.5	<0.25	<0.5	7.6	24.4

Notes:

1. Values reported are blank-corrected averaged values.
2. NA = Not available. Due to problems in shipping, IC samples in hot cell test #3 were contaminated with HNO_3.
3. A NO_3^- value for 102-AZ Core 1 is not reported since the only measured value above blank levels appeared to be an outlier.
4. The suffix number appended to each CVS-IS-HW39-4 glass indicates the hot cell test number.

Table 4.41. Simulant-to-Radioactive Comparisons for P and S Releases

	Relative Simulant/Radioactive Ratio for Sulfur Release	Relative Simulant/Radioactive Ratio for Phosphorus Release
101-AZ Core 1	0.6	1.1
101-AZ Core 2	NA	NA
102-AZ Core 1	1.8	0.9

Notes: 1. Relative ratios equal simulant-to-radioactive ratio for analyte release in leachate divided by same ratio for boron.

2. NA = not available

These results were consistent with "as-batched" simulant glass compositions. Additions of P and S were made to the simulant glasses to match measured compositions of the radioactive glasses; however, additions of Cl and F were not made to the simulant glasses. As would be expected, PO_4 and SO_3 leachate levels were somewhat comparable between simulant and radioactive glasses; Cl and F were easily measurable in the radioactive leachate samples and near detection levels in the simulant leachate samples (the detection level for Cl is 0.25 μg/ml). Because the radioactive glasses are slightly more durable than their corresponding simulants, the leachate releases in the radioactive glass should be somewhat lower for P and S.

Table 4.41 compares relative anion results (i.e., simulant-to-radioactive release ratios) to relative B results. A relative ratio near 1.0 indicates that the ratio of simulant-to-radioactive release for the analyte is equal to that same ratio for B, or that the concentration of the analyte in the simulant glass matches well with that in the radioactive glass. The Table 4.41 results indicate that the P concentrations in the simulant glass matched well with the radioactive glass, at least for two glasses. Review of the ICP leachate data for the PCT and MCC-1 indicates that the P concentrations are matched well for all three radioactive glasses. The S composition of 101-AZ Core 1 simulant may be too low; the 102-AZ Core 1 simulant may be too high.

Minor component influences, including Cl, F, P, and S at and above solubility limits, were studied in LLW borosilicate glass[a]. Fluorine was shown to increase durability and lower pH in the PCT. This effect was quite dramatic at F concentrations above solubility limits in the glass, but was minor in the range of F levels found in the radioactive glasses. Chlorine and P were shown to decrease durability in the PCT, although the difference attributed to Cl may be within experimental error. Sulfur had minor effects on durability at concentrations near or below solubility. The effect of each minor component can be attributed to more than one competing cause. For example, F was shown to decrease durability in a dynamic flow through leach test, which is attributed to weakening of the network bond strength (when F is substituted for oxygen in Si-O-Si). In the PCT, however, glass dissolution is affected by pH, which is buffered by F. Table 4.42 shows results from the Hong study

---

(a) Hong, L., J.D. Darab, P.A. Smith, X. Feng, and D.K. Peeler. 1995. "Chemical Durability of Low-Level Simulated Radioactive Waste Glasses with High-Concentrations of Minor Components." Pacific Northwest Laboratory. Richland, Washington.

Table 4.42. Effects of Minor Components on PCT Durability

	Base Glass Composition (wt %)	Minor Component Glass Comp. (wt %)	Leachate Concentration Difference (μg/ml)	Change in PCT B Release	Change in PCT Na Release	Change in PCT Si Release
Cl	.09	0.57	2.2	+13.2%	+12.2%	+ 8.7%
F	0.21	0.92	4.4	- 2.2%	- 9.4%	- 6.6%
P_2O_5	1.19	2.1	0.7	+29.4%	+19.5%	+26.7%
SO_3	0.32	0.75	1.0	+ 4.0%	+ 7.6	+ 2.1%

indicating potential causes for differences between the core samples and simulants. The increased level of Cl^- in the core sample leachates would tend to decrease durability in the radioactive glass slightly. The F^- measured in the core samples would tend to increase durability slightly of the radioactive glasses relative to their simulants. The consistent levels of P in the radioactive and simulant glasses should not affect the durability of one over the other; however, as can be seen in the table, small differences in P can influence durability significantly. The magnitude of the measured anion differences (radioactive versus simulant glass) does not appear to be significant enough to account for observed differences in radioactive and simulant durability. However, the durability models, which do not account for independent variation in these minor components, may be difficult to use in predicting actual waste glass and simulant durability where the minor components do not conveniently match the "minor components mix" used in simulant preparation for model development.

4.5.6 MCC-1 Anion Releases and pH

Results of the IC analysis of the hot-cell MCC-1 leachates are shown in Table 4.43. The samples analyzed included one of the blanks, two of the leachates from Core 1, two of the leachates from Core 2, one leachate from the nonradioactive simulant, and one leachate from the ATM-10 glass. The results indicated only slightly elevated levels of Cl^- in the radioactive samples. A 10x dilution on samples made before analysis decreased the sensitivity of the analysis. In subsequent tests, the researchers specifically requested no dilution of the samples, regardless of limited sample volumes.

Table 4.43. MCC-1 Anion and pH Results for Core Sample and Simulant Glasses

Glass Type	pH	F^- (μg/mL)	Cl^- (μg/mL)	NO_2^- (μg/mL)	Br^- (μg/mL)	NO_3^- (μg/mL)	PO_4^{-3} (μg/mL)	SO_4^{-2} (μg/mL)
101-AZ Core #1	8.06	<2	2.5	<4	<2	<4	<4	<4
102-AZ Core 1	8.26	<2	2.5	<4	<2	<4	<4	<4
NCAW Simulant	8.73	<2	<2	<4	<2	<4	<4	<4
ATM-10	8.91	<2	<2	<4	<2	<4	<4	<4

Note: Values reported are blank-corrected averaged values.

4.5.7 Reasons for Differences Between Radioactive and Simulant Durabilities and Comparison with Work by Others

Radionuclide decay in glass can affect durability by radiolysis of air and water in contact with the glass and damage to the glass itself. Irradiation of water, dissolved gases, and air creates several free radicals and new molecules that affect the pH and redox potential of leachant solutions. This in turn affects the durability of glass. Predominate species generated under gamma irradiation include hydrated electrons (e^-_{aq}), hydrogen ions (H^+), hydroxyl ($\cdot OH$), and hydrogen atoms ($H\cdot$), and under alpha irradiation include hydroperoxyl ($HO_2\cdot$), molecular hydrogen (H_2), and hydrogen peroxide (H_2O_2) (Mendel 1984). Both gamma and alpha irradiation are known to reduce the pH of deionized water due to the radiolysis of air and formation of nitric acid (Cunnane 1994). Damage to the glass structure occurs through processes of displacement and ionization. Volume increases in HLW glass due to displacement damage is only expected after 1000 years in the repository and ionization damage may occur in the first 10 years after fabrication (Cunnane 1994). The glasses in this study were leach tested in argon atmospheres using deaerated and deionized water at times between 15 and 50 months following fabrication, thus minimizing radiation affects.

The evaluation, prediction, and comparison of radiation effects on glass corrosion are complex because of the number of important variables and complex interactions (e.g., amount and type of radioactivity, composition of headspace and dissolved gas, duration of test, extent of glass corrosion reaction, composition of dissolved glass in solution, type of leachant). Results from several irradiated leach test studies using nonradioactive and doped, simulant glasses are referenced below.

Gamma-irradiated tests using PNL 76-78 glass in deionized and deaerated water at 20 m^{-1} and deaerated brines at 10, 100, and 1000 m^{-1} showed an increase in pH and release rates over unirradiated tests (McVay, 1981). Gamma irradiated tests using borosilicate glasses in deionized and deaerated water by several researchers showed three- to five-fold increase in B, alkalis, Si, and actinides (Cunnane 1994). Irradiated tests in groundwater equilibrated with tuff by several researchers showed no change to pH and glass dissolution rates, except in a few cases where dissolution rates decreased (Cunnane 1994). In irradiated tests designed to separately evaluate the effect of water radiolytic products (vacuum-degassed and Ar-sparged systems to remove nitric and carboxylic acid production), the increase in glass dissolution was about half that observed in similar tests in air (McVay 1981; Pederson 1983). In companion tests where nitric acid was added but the tests were not irradiated, the increase in glass dissolution was again about half that observed in irradiated tests in air.

Studies comparing fully radioactive, actual waste glass and simulant waste glasses report both increased and decreased dissolution rates for the actual waste glass. Results from static tests in deionized water and Ar atmosphere at 1100m^{-1} showed a 50% increase in B and Si release in the radioactive glass (JSS/A) when compared to the simulant glass (ABS-118) during 91 and 180 days (Werme 1990; JSS 1988). This difference was within systematic error and was not observed at a duration of one year. Tests, which included 1 g of magnetite, showed release rates of the radioactive glass to be similar or significantly less than the simulant glass. Differences in radionuclide (Tc, Np, Pu, and Am) release between actual waste glasses with Magnox and THORP compositions and doped glasses of similar compositions showed a release increase between two for Tc and 75 times for Am in the fully radioactive glass (Hall 1988; Boult 1991; Marples 1991). Three Savannah River fully radioactive glasses, SRL 131/11, 165/42, and 200, were tested in EJ-13 solution at 340, 2,000, and 20,000m^{-1}, respectively, and at various durations to evaluate differences with simulant glasses (Feng 1993). In all three glass pairs the radioactive glasses yielded leachates with lower pHs, which was attributed to radiolytic generation of nitric acid from air and other acids. Two of the radioactive glasses exhibited decreased release that was attributed to a dominant corrosion mechanism of network hydrolysis, which is favored by higher solution pH. The most durable of the glass pairs exhibited the opposite behavior of increased radioactive glass release. This was attributed to a dominant corrosion mechanism of ion exchange, which is favored by low pH. Release rate differences between simulant and actual waste glasses were all within a factor of two to three for periods up to 182 days.

As mentioned above, the prediction and explanation of radiolytic effects on glass dissolution are complex. The results from this study showed a modest reduction in dissolution rates (22 to 40% for B in 7-day PCT) for fully radioactive glasses when compared to simulant glasses of the same composition. This difference was equal to or lower than differences observed by others, which is consistent with removing part of the radiolytic effect. To the extent argon back-filling of the leach containers was effective, the effect of radiolytic generation of nitric acid was eliminated. Based on the type of test performed and the relative durability of the glasses in this study, the dominant corrosion mechanism was expected to be network hydrolysis, which is favored under higher pHs. With the absence of air in the system, it was not clear whether radiolytic affects should have increased or decreased corrosion.

Release of radionuclides in leach test conditions is strongly influenced by solubility. Solubilities can be influenced by redox, presence of complexants, equilibrium with solid forms, and presence of competing elements for sorption. Generalizations can be made but changes to the system such as leachant composition and redox can change solubilities significantly. In HLW glass, Tc, U, Np, and Cs are generally more soluble than Pu or Am under pH basic conditions of most repositories and leach tests (Cunnane 1994). Laboratory testing of several glasses has shown higher releases for Tc, U, Np, and Cs than for the less-soluble Pu and Am. Testing of R7T7 glass in static leach tests at 90°C in distilled water showed relative releases of actinides as U > Np > Pu > Am (Vernaz 1992). Normalized releases of U and Np were roughly 10% of the normalized B release whereas Pu and Am were 3% and 0.3%, respectively.

Results from this study were mostly consistent with results from Vernaz (1992) and other laboratory tests referenced by Cunnane (1994). Figure 4.34. provides a summary of radiochemical releases for PCT and MCC-1 tests for selected radionuclides. The radionuclide-to-B ratio is the inverse of retention and indicates the fraction of corroded constituent that is released into solution. A log of radionuclide-to-B ratios near 0 indicates completely soluble constituents; -1 indicates normalized releases near 10% of B; -2 indicates normalized releases near 1% of B; and so forth. As with prior studies, Am had normalized releases significantly lower than B, ranging from 0.1% to 6% of B. Also consistent with prior studies, Tc, U, Np, and Cs were generally more soluble than Am (i.e., ≥ 10% of B normalized release). Not consistent with prior studies, Pu exhibited significant normalized releases near B. Strontium, which should behave similar to Ca, was soluble in MCC-1 tests and insoluble in PCT.

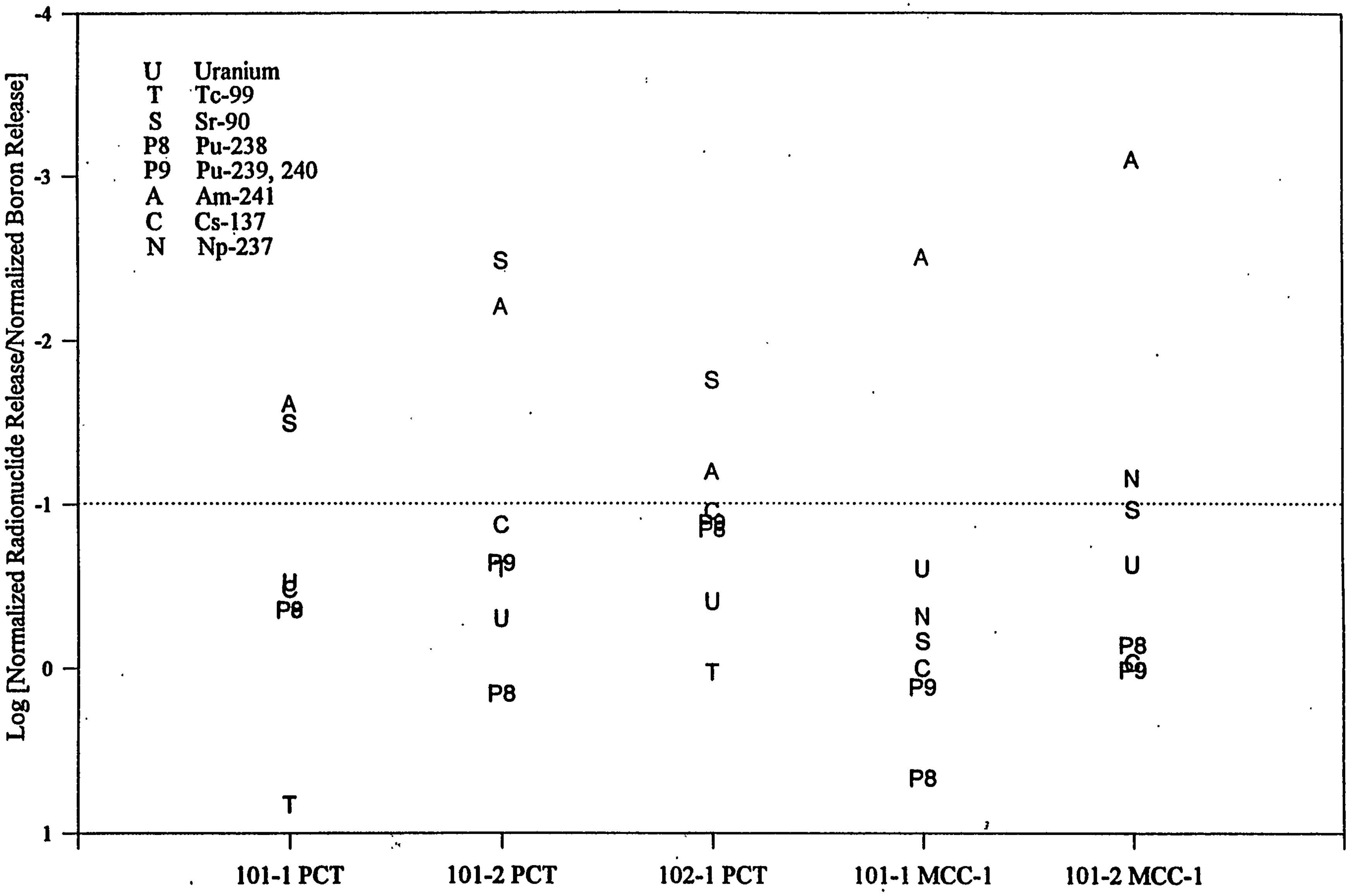


Figure 4.34. Summary of Radionuclide Releases Ratioed to Boron for PCT and MCC-1

4.6 Mass Balances

The elemental composition of each of the process slurries and glass for each core sample and the two simulants are shown in Figures 4.35 through 4.39. The concentrations of the analytes increase throughout the process because the solid to liquid ratio is increased at each step along the process. The K concentration in the formated slurry from 101-AZ Core 2 increases drastically during the formating process. This increase occurred when the pH probe broke into the sample during the formating process and a significant quantity of KCl was released into the sample. For each of the samples, a significant increase in the concentrations of the frit components is observed between the formated slurry and melter feed.

In Figures 4.40 through 4.42, decay corrected specific activities of the major radionuclides present in the three core samples are plotted for each of the process slurries and the glass. The percent recoveries observed for the radionuclides are also reported in Tables 4.44 through 4.46. Mass balances were performed on each core sample throughout the HWVP process including washed solids, formating, frit addition, and glass. Mass balances for the washing process were reported in the characterization reports for the three core samples(a), (b)(Peterson, et. al. 1989). Mass balances were performed on the major cations and radionuclides.

The mass balance results from all of these samples indicate that the analytical subsamples from the melter feeds are not representative of the bulk composition. The majority of the undissolved solids in the melter feeds settle very quickly making it difficult to obtain representative subsamples unless the sample is being stirred. Even when the sample is stirred, the frit tends to settle to the bottom and collect along the edge of the container; therefore, nonrepresentative samples are often obtained even when the sample is being stirred.

This radionuclide data indicates that Tc is volatilized during the melt. This result is consistent with previous results observed in simulant studies. The core sample results indicate that approximately 98% of the Tc in the sample is volatilized during the melting processes. There also appears to be some Cs volatility as indicated by the recoveries from 101-AZ Core 1 and 102-AZ Core 1. The recoveries of Cs do not appear to correlate well with the chloride concentrations in the sample as was postulated from simulant studies. Volatilization as high as 40% of the original Cs activity were observed, but no significant volatilization of Cs was observed in 101-AZ Core 2 which was expected to have the highest chloride concentrations. The radionuclide mass balances also indicate that ^{129}I may have been lost during the formating process. The data is not definitive, but it appears that some of the iodine may have been lost during this process.

(a)Gray, W. J., M.E. Peterson, R.D. Scheele, and J.M. Tingey *Characterization of the Second Core Sample of Neutralized Current Acid Waste From Double-Shell Tank 101-AZ*, Letter Report to Westinghouse Hanford Company, September 1993.

(b)Gray, W. J., M. E. Peterson, R. D. Scheele, and J. M. Tingey *Characterization of the first Core Sample of Neutralized Current Acid Waste From Double-Shell Tank 102-AZ*, Letter Report to Westinghouse Hanford Company, September 1993.

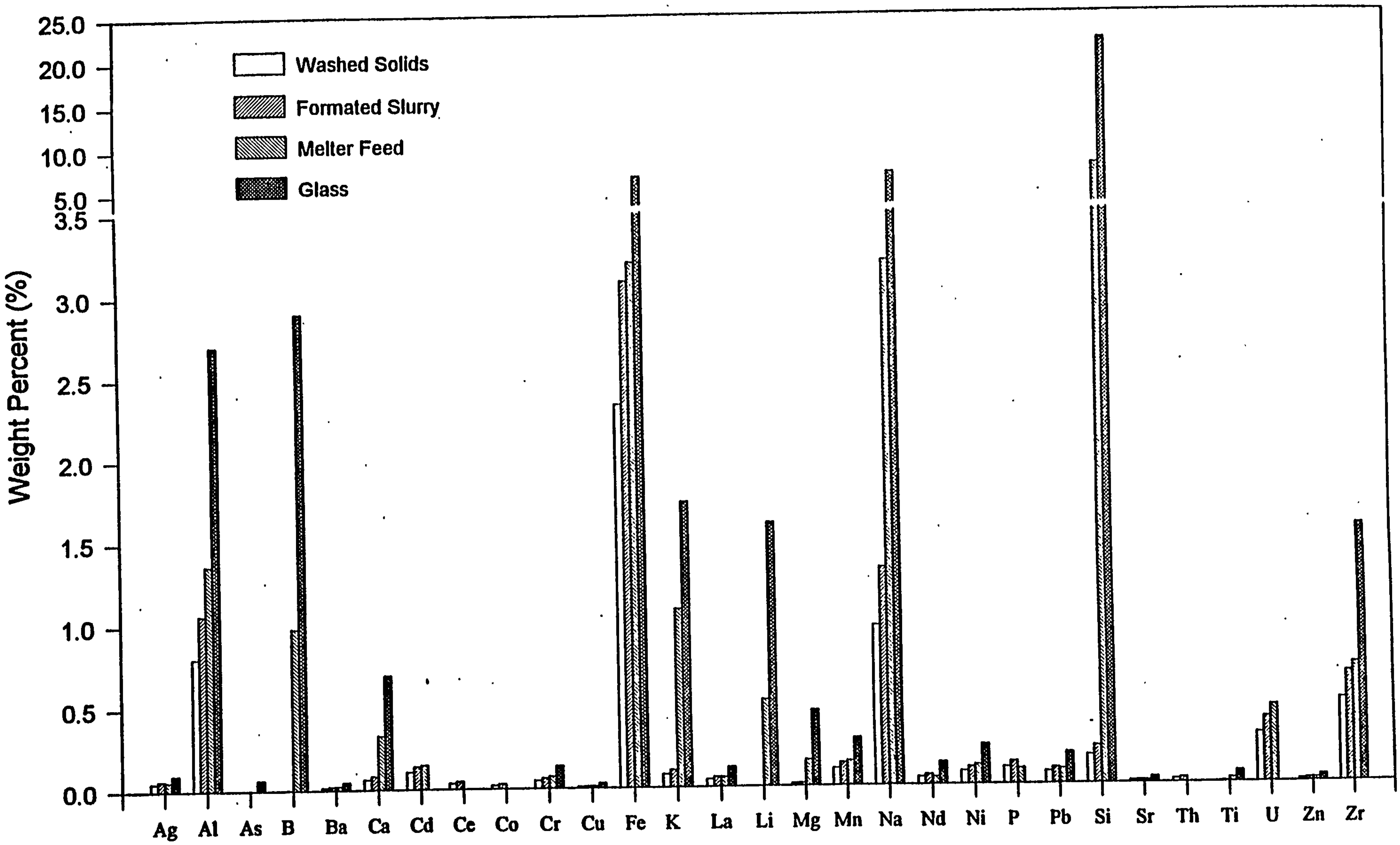


Figure 4.35. Chemical Composition of 101-AZ Core 1

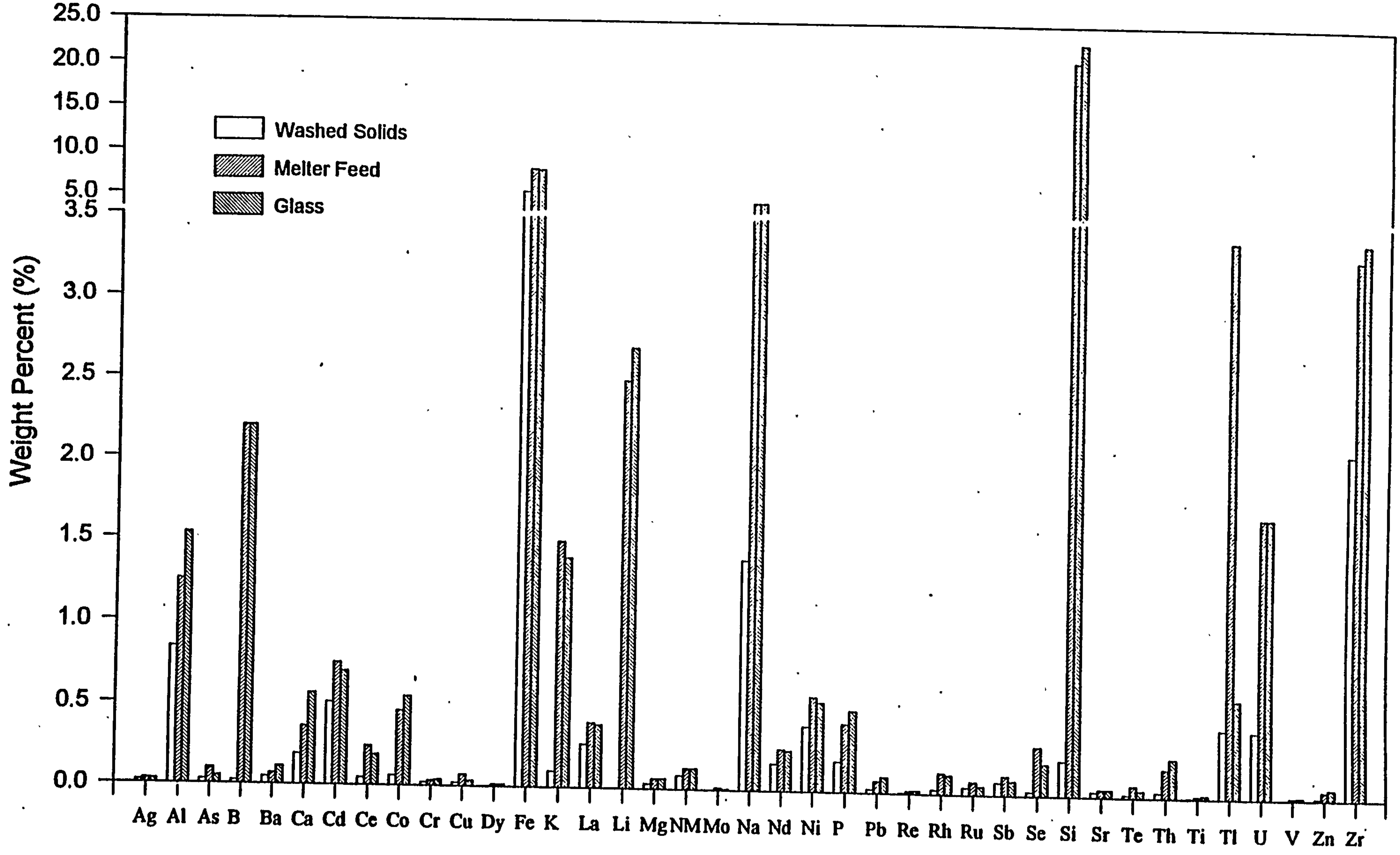


Figure 4.36. Chemical Composition of 101-AZ Core 2

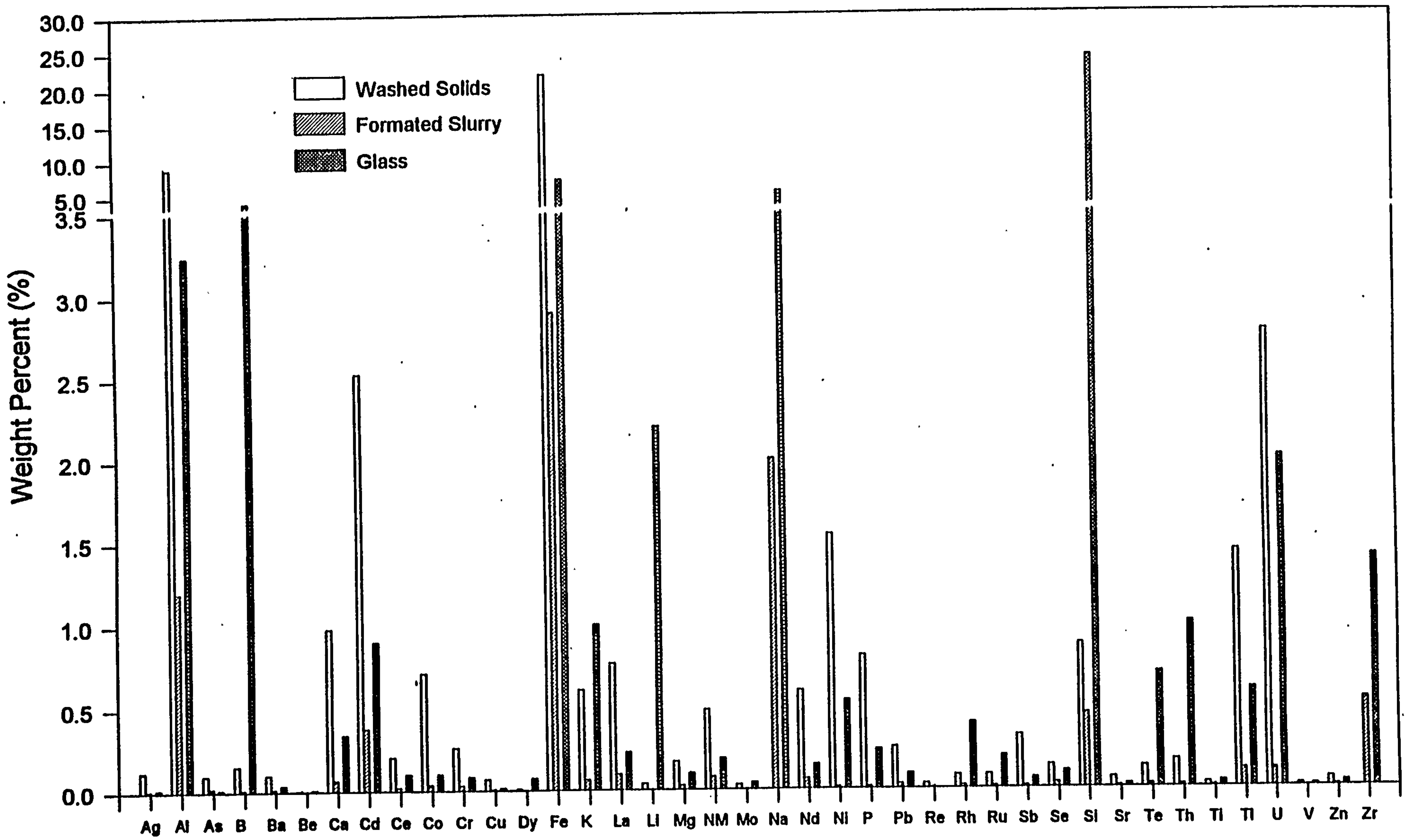


Figure 4.37. Chemical Composition of 102-AZ Core 1

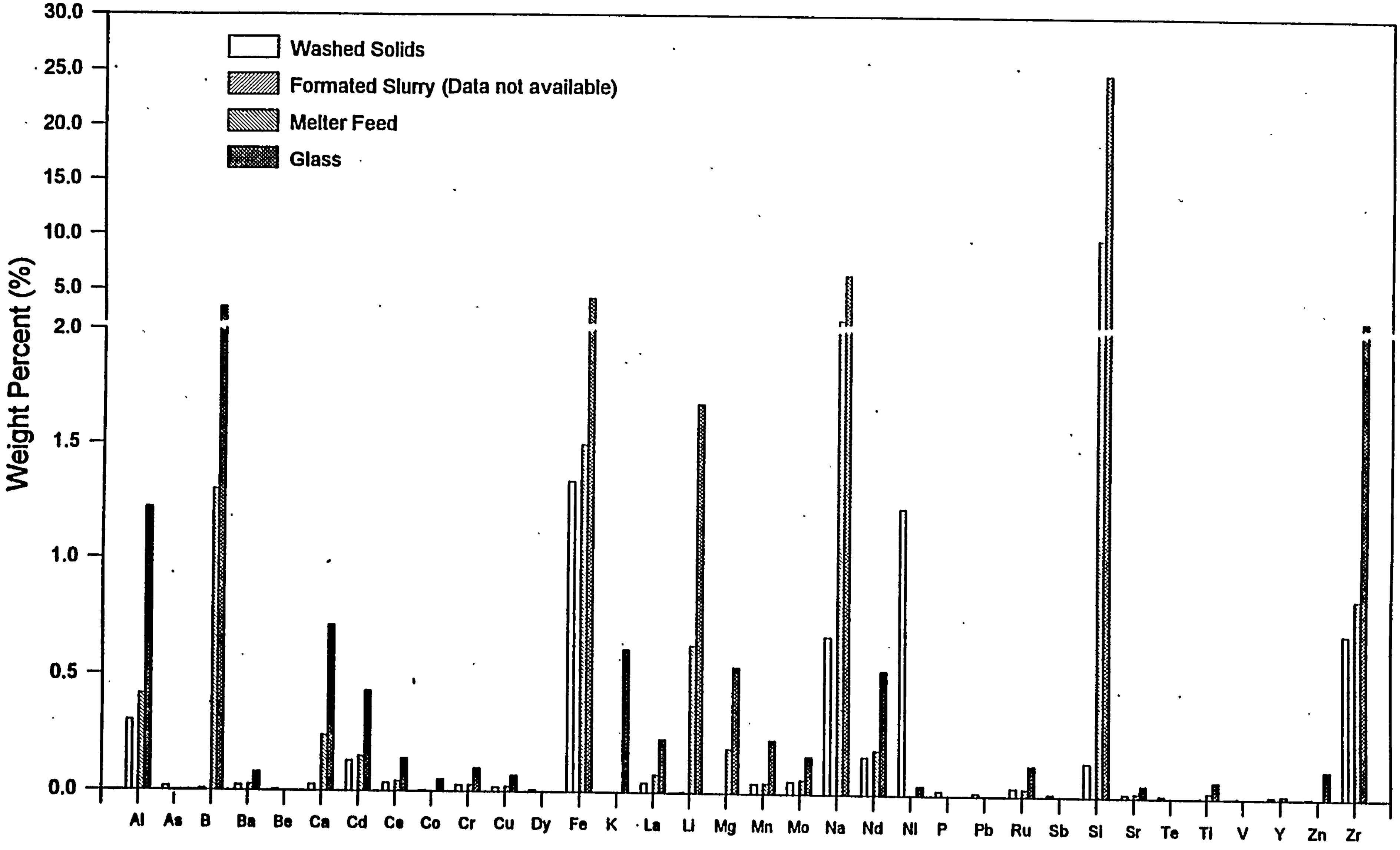


Figure 4.38. Chemical Composition of NCAW Simulant

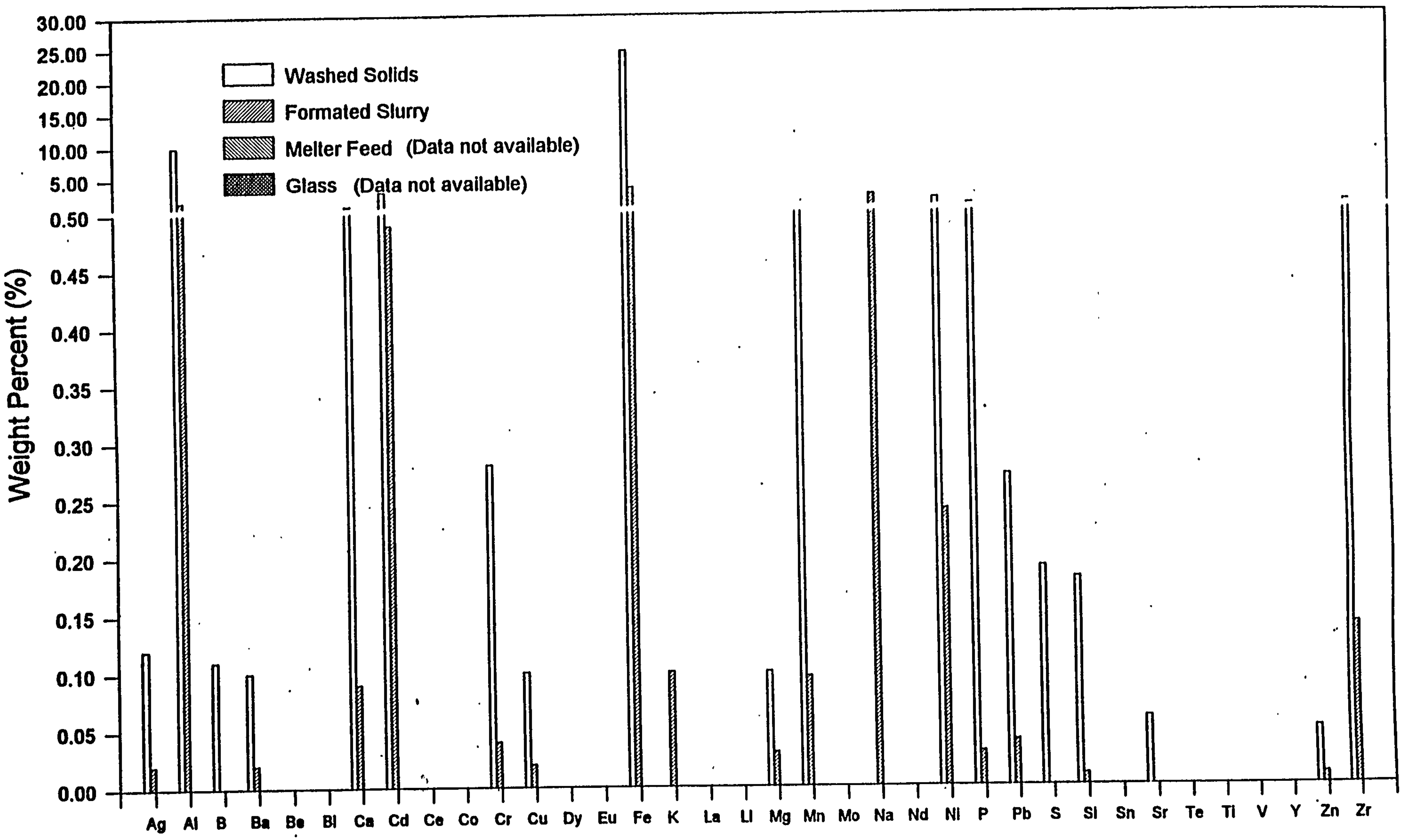


Figure 4.39. Chemical Composition of 102-AZ Core 1 Simulant

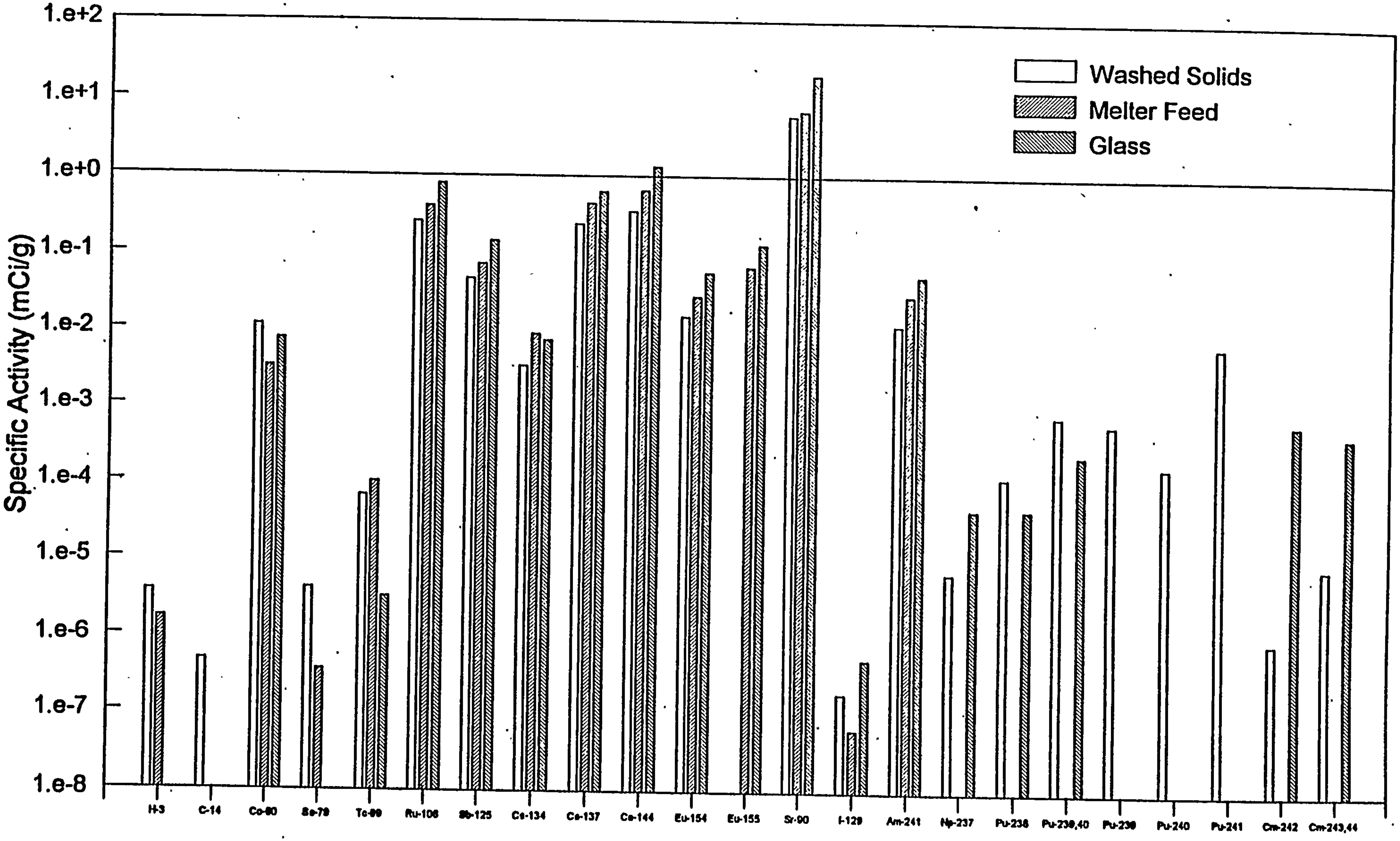


Figure 4.40. Specific Activity of Radionuclides for 101-AZ Core 1

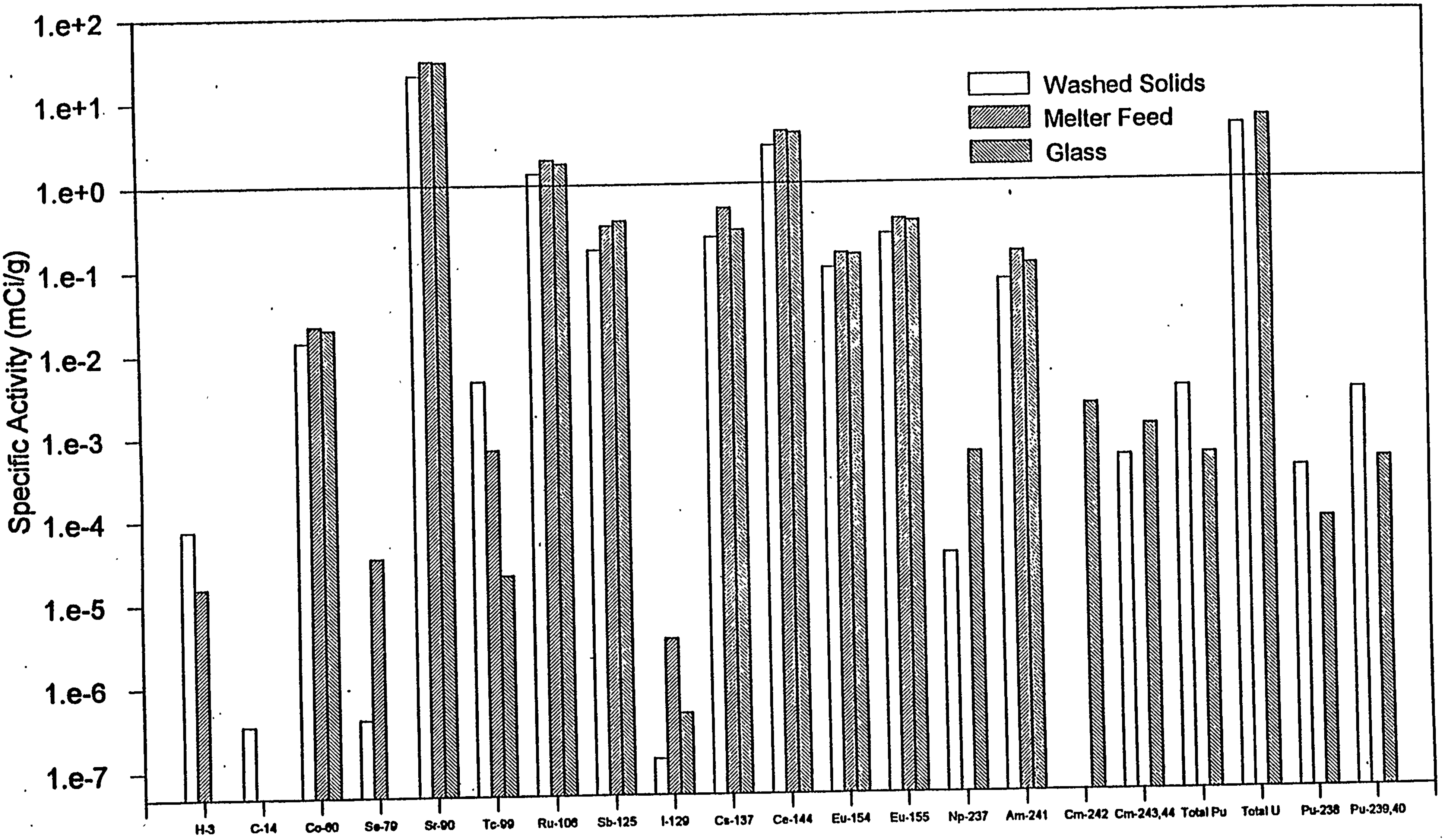


Figure 4.41. Specific Activity of Radionuclides for 101-AZ Core 2

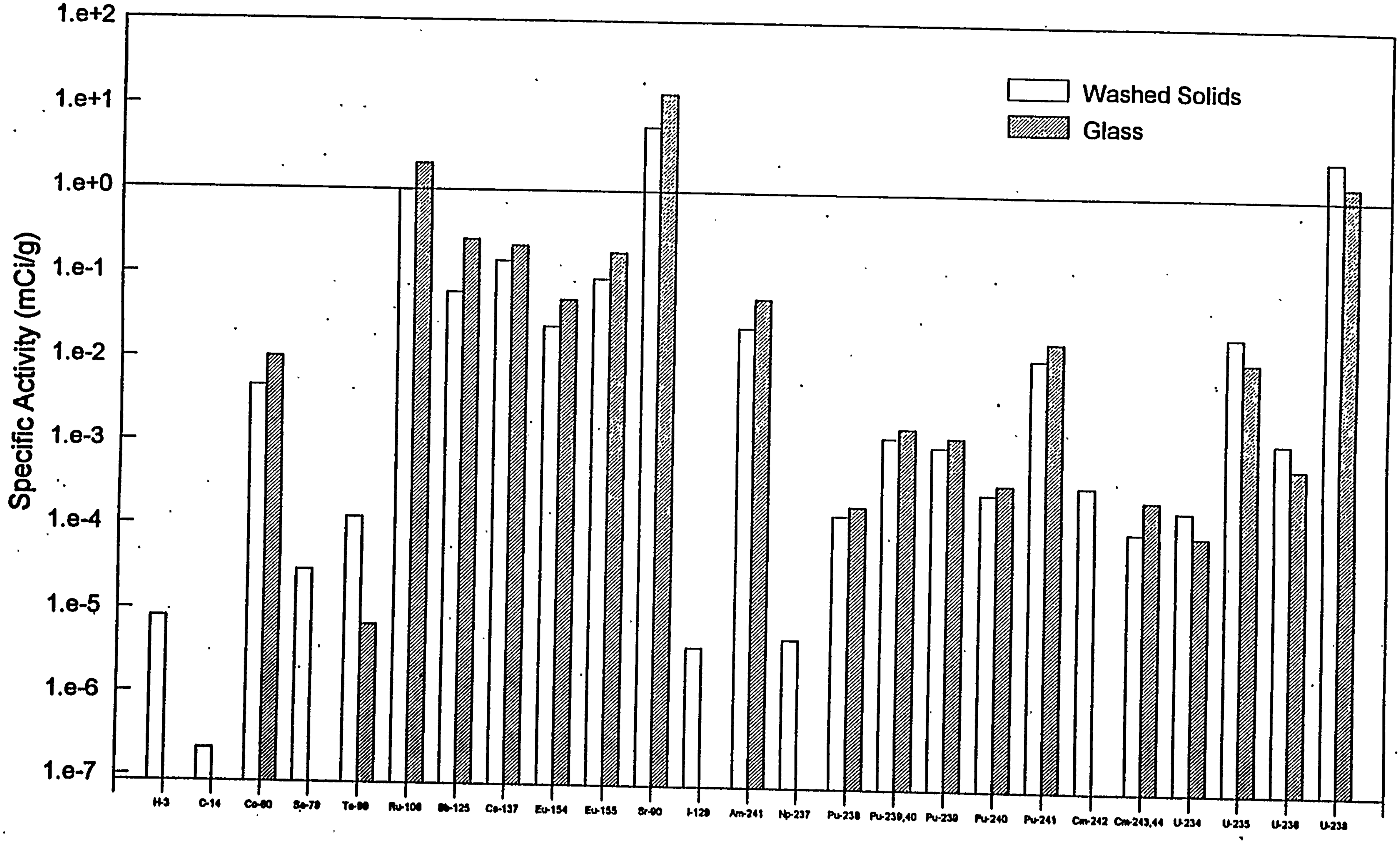


Figure 4.42. Specific Activity of Radionuclides for 102-AZ Core 1

Table 4.44. Decay Corrected Specific Activity and Recovery Percent for 101-AZ Core #1

	Specific Activity (mCi/g) - Decay Corrected to 1/1/90				Recovery (%)		
Nuclide	Washed Solids	Melter Feed	Melter Feed Supernate	Glass	Melter Feed From Washed Solids	Glass	Glass (based on Melter Feed)
H-3	< 4.5E-07	1.66E-06			59		
C-14	4.70E-07						
Co-60	1.13E-02	3.19E-03		7.51E-03	37	35	94
Se-79	< 4.00E-06	< 3.57E-07					
Tc-99	6.60E-05	1.00E-04		3.15E-06		3	3
Ru-106	2.55E-01	4.03E-01	3.65E-03	7.89E-01	209	164	78
Sb-125	4.71E-02	7.29E-02		1.45E-01	205	163	79
Cs-134	3.33E-03	8.91E-03	1.14E-03	7.35E-03	354	117	33
Cs-137	2.37E-01	4.41E-01	7.66E-02	6.21E-01	246	138	56
Ce-144	3.49E-01	6.55E-01		1.33E+00	248	201	81
Eu-154	1.54E-02	2.82E-02		5.71E-02	242	196	81
Eu-155		6.72E-02		1.31E-01		78	78
Sr-90	6.04E+00	7.07E+00		2.03E+01	155	178	115
I-129	< 1.80E-07	< 6.14E-08		< 5.00E-07	45		
Am-241	1.20E-02	2.92E-02		5.25E-02	322	232	72
Np-237	6.70E-06			4.50E-05		355	355
Pu-238	1.20E-04			4.54E-05		20	20
Pu-239+240	7.90E-04			2.40E-04		16	16
Pu-239							
Pu-240							
Pu-241							
Cm-242	8.82E-07			6.58E-04		39459	39459
Cm-243+244	8.36E-06			4.61E-04		2915	2915

Table 4.45. Decay Corrected Specific Activity and Recovery Percent for 101-AZ Core #2

	Specific Activity (mCi/g) - Decay Corrected to 1/1/90				Recovery (%)		
Nuclide	Washed Solids	Melter Feed	Melter Feed Supernate	Glass	Melter Feed from Washed Solids	Glass from Melter Feed	Glass from Washed Solids
H-3	< 7.45E-05	1.54E-05					
C-14	3.34E-07						
Co-60	1.38E-02	2.13E-02		1.96E-02	311	37	114
Se-79	< 3.90E-07	< 3.38E-05					
Sr-90	2.04E+01	3.05E+01		2.99E+01	302	39	118
Tc-99	< 4.63E-03	< 3.76E-04		2.05E-05			
Ru-106	1.38E+00	1.99E+00	3.65E-03	1.80E+00	291	36	105
Sb-125	1.68E-01	3.23E-01		3.71E-01	387	46	177
I-129	1.27E-07	< 3.42E-06		< 4.37E-07			
Cs-134			1.14E-03				
Cs-137	2.31E-01	5.14E-01	7.66E-02	2.81E-01	449	22	98
Ce-144	2.81E+00	4.17E+00		3.98E+00	299	38	114
Eu-154	9.70E-02	1.45E-01		1.39E-01	302	38	115
Eu-155	2.43E-01	3.65E-01		3.44E-01	303	38	114
Np-237	3.45E-05			5.68E-04			1322
Am-241	6.71E-02	1.45E-01		1.05E-01	437	29	126
Cm-242				2.14E-03			
Cm-243,4	4.93E-04			1.18E-03			193
Total Pu	3.38E-03			5.13E-04			12
Total U	4.50E+00			5.60E+00			100
Pu-238	3.40E-04			8.11E-05			19
Pu-239,240	3.04E-03			4.32E-04			11

Table 4.46. Decay Corrected Specific Activity and Recovery Percent for 102-AZ Core #1

Nuclide	Specific Activity (mCi/g) - Decay Corrected to 1/1/90 Washed Solids	Glass	Recovery (%) Glass from Washed Solids
H-3	7.50E-07		
C-14	2.10E-07		
Co-60	4.60E-03	1.03E-02	95
Se-79	< 4.20E-06		
Tc-99	1.25E-04	6.73E-06	2
Ru-106	9.89E-01	2.06E+00	89
Sb-125	6.18E-02	2.61E-01	181
Cs-137	1.46E-01	2.21E-01	65
Eu-154	2.49E-02	5.17E-02	89
Eu-155	9.17E-02	1.89E-01	88
Sr-90	5.75E+00	1.41E+01	105
I-129	3.00E-08		
Am-241	2.51E-02	5.56E-02	95
Np-237	5.16E-06		
Pu-238	8.77E-06	2.00E-04	974
Pu-239+240	1.83E-02	1.73E-03	4
Cm-242	2.06E-04		
Cm-243+244	1.07E-04	2.62E-04	104
U	2.95E+00	1.49E+00	
U-234	1.96E-04	9.88E-05	22
U-235	2.31E-02	1.17E-02	22
U-236	1.32E-03	6.66E-04	22
U-238	2.93E+00	1.48E+00	22

5.0 References

Boult, K.A., J.T. Dalton, A. Hough, J.A.C. Marples, G.P. Robertson, and R.I. Wilkins. 1991. *Radionuclide Release from Solidified High-Level Waste.* EUR-13604, Commission of European Communities.

Cunnane, J.C., ed. 1994. *High-Level Waste Borosilicate Glass: A Compendium of Corrosion Characteristics.* DOE-EM-0177, Vol. 2, U.S. Department of Energy, Washington, D.C.

Feng, X., J.K. Bates, E.C. Buck, C.R. Bradley, and M. Gong. 1993. "Long-Term Comparison of Dissolution Behavior Between Fully Radioactive and Simulated Nuclear Waste Glasses." *Nuclear Technology* **104**:193-206.

Hall, A.R., A. Hough, and J.A.C. Marples. 1988. *Leach Testing of Fully Active MW Glass.* AERE-R-13071, Harwell Laboratory, Oxfordshire, United Kingdom.

Hrma P.R., G.F. Piepel, M.J. Schweiger, D.E. Smith, D.S. Kim, P.E. Redgate, J.D. Vienna, C.A. LoPresti, D.B. Simpson, D.K. Peeler, and M.H. Langowski. 1994. *Property/Composition Relationships for Hanford High-Level Waste Glasses Melting at 1150°C.* PNL-10359, Pacific Northwest Laboratory, Richland, Washington.

Jantzen, C.M., N.E. Bibler, D.C. Beam, W.G. Ramsey, and B.J. Waters. 1992. *Nuclear Waste Glass Product Consistency Test (PCT) Version 5.0(U).* WSRC-TR-90-539, Rev.2, Westinghouse Savannah River Company, Aiken, South Carolina.

Japanese Swiss Swedish (JSS) Project. 1988. *JSS-Project Phase V: Final Report, Testing, and Modelling of the Corrosion of Simulated Nuclear Glass Powders in a Waste Package Environment.* JSS-88-02, Japanese Swiss Swedish Project.

Marples, J.A.C., N. Godon, F. Lanza, and P. Von Iseghem. 1991. "Radionuclide Release from High Level Waste Forms Under Repository Conditions in Clay or Granite." In *Radioactive Waste Management and Disposal*, L. Cecille, ed. pp.287-301. Elsevier, Amsterdam, The Netherlands.

Materials Characterization Center. 1984. *MCC-1P Static Leach Test Method.* Pacific Northwest Laboratory, Richland, Washington.

McVay, G.L., W.J. Weber, and L.R. Pederson. 1981. "Effects of Radiation on the Leaching Behavior of Nuclear Waste Forms." *Nucl. Chem. Waste Mgmt.* **2**:103-108.

Mendel, J.E., compiler. 1984. *Final Report of the Defense High-Level Waste Leaching Mechanisms Program.* PNL-5157, Pacific Northwest Laboratory, Richland, Washington.

Pederson, L.R., and G.L. McVay. 1983. "Influence of Gamma Irradiation on Leaching of Simulated Nuclear Waste Glass: Temperature and Dose Rate Dependence in Deaerated Water." *J. Am. Ceram. Soc.* **66**:863-867.

Peterson, M.E., R.D. Scheele, and J.M. Tingey. 1989. *Characterization of the First Core Sample of Neuatralized Current Acid Waste From Double-Shell Tank 101-AZ.* PNL-7758, Pacific Northwest Laboratory, Richland, Washington.

Tingey, J.M., M.L. Elliott, and E.V. Morrey. 1991. "Laboratory Characterization and Vitrification of Hanford High-Level Waste." *Ceramic Transactions Nuclear Waste Management IV.* E.G. Wicks, D.F. Bickford, ed., Vol. 23, pp. 15.

Vernaz, E.Y. 1992. "Leaching of Actinides from Nuclear Waste Glass: French Experience." *Mat. Res. Soc. Symp. Proc.* **257**:37-48.

Werme, L., I.K. Bjorner, G. Bart, H.U. Zwicky, B. Grambow, W. Lutze, R.C. Ewing, and C. Magrabi. 1990. "Chemical Corrosion of Highly Radioactive Borosilicate Nuclear Waste Glass under Simulated Repository Conditions." *J. Mater. Res.* **5**:1130-1146.

APPENDIX A

Chemical Data

Table A.1. Chemical Composition of 101-AZ Core #1 Process Solutions and Product Glass

	Washed Solids			Melter Feed			MF Supernate		Glass		
	(wt%)	(wt% oxide)	(reproducibility)	(wt%)	(wt% oxide)	(reproducibility)	(ug/ml)	(reproducibility)	(wt%)	(wt% oxide)	(reproducibility)
Ag	(0.048)	0.58	6%	0.056	0.2	10%	4.8	8%	0.091	0.098	4%
Al	0.802	17	7%	1.36	6.7	1%	22	13%	2.7	5.2	1%
As	< 0.038	<1	NM	<3.0E-02	<0.1	NM	4.0	NM	0.063	0.084	NM
B	2.0E-03	0.073	<0.5%	0.98	8.2	39%	150	8%	2.9	9.3	<0.5%
Ba	1.5E-02	0.19	9%	0.022	0.1	4%	2.7	113%	0.091	0.1	1%
Be	0.0E+00	<.00	NM	0.00035	0.0	NM	0.06	NM	7.0E-04	0	14%
Ca	6.2E-02	0.98	NM	0.33	1.2	66%	240	11%	1.02	1.65	NM
Cd	0.10	1.3	7%	0.149	0.4	3%	590	5%		0.30	<0.5%
Ce	0.034	0.47	27%	<4.0E-02	<0.1	NM	5.9	NM	<0.19	<.2	NM
Co	0.021	0.33	54%	<7.4E-02	<0.3	NM	6.3	NM	<5.5E-02	<.8	NM
Cr	0.047	0.77	6%	0.073	0.3	<0.5%	29	1%	0.136	0.2	1%
Cu	8.0E-03	0.11	<0.5%	0.010	0.0	5%	5.0	28%	0.029	0.04	6%
Dy	1.5E-03	0.019	67%	<2.6E-03	<0.01	NM	0.87	NM	<1.3E-02	<.02	NM
Fe	2.3	38	7%	3.2	12	1%	3.4	12%	6.8	9.7	<0.5%
K	0.080	1.09	NM	1.08	3.4	NM	17000	3%	1.73	2.1	NM
La	0.045	0.60	9%	0.060	0.18	3%	9.2	5%	0.12	0.14	2%
Li	0.0	<.01	NM	0.53	3.0	37%	115	5%	1.6	3.15	<0.5%
Mg	0.013	0.24	15%	0.16	0.68	33%	63	3%	0.46	0.76	<0.5%
Mn	0.10	1.86	8%	0.15	0.63	4%	400	4%	0.29	0.47	1%
Mo	2.0E-03	0.034	<0.5%	<3.3E-03	<0.01	NM	0.30	NM	0.0033	<.003	NM
Na	0.97	15	NM	3.2	11	NM	29000	6%	7.14	9.6	NM
Nd	0.044	0.58	18%	0.046	0.14	16%	4.5	NM	(0.11)	0.13	9%
Ni	0.080	1.15	NM	0.12	0.41	NM	119	3%	0.24	0.30	NM
P	0.10	2.7	32%	(9.3E-02)	0.55	7%	14	NM	<0.5	<1.1	NM
Pb	0.073	0.88	7%	0.091	0.25	8%	4.2	NM	(0.19)	0.21	10%
Re	0.0	<.01	NM	<3.7E-03	<0.01	NM	0.49	NM	<2.1E-02	<.03	NM
Rh	0.0	<.08	NM	<2.1E-02	<0.07	NM	6.4	NM	<0.12	<.2	NM
Ru	0.0	<.08	NM	<9.2E-03	<0.03	NM	3.6	NM	<5.3E-02	<.07	NM
Sb	0.0	<.6	NM	<0.18	<0.6	NM	5.3	NM	<8.9E-02	<.1	NM
Se	0.0	<.2	NM	<5.1E-02	<0.2	NM	9.4	NM	<0.18	<.3	NM
Si	0.17	4.2	8%	7.9	44	38%	120	26%	22	48.1	0%
Sr	0.011	0.14	10%	0.014	0.0	18%	36	4%	0.033	0.039	4%
Te	0.0	<.1	NM	<1.8E-02	<0.06	NM	2.6	NM	<1.7E-02	<.02	NM
Th	(0.019)	0.24	27%	<2.4E-02	<0.07	NM	<0.7	NM	<0.24	<.3	NM
Ti	4.0E-03	0.076	<0.5%	0.024	0.10	37%	0.23	NM	0.069	0.12	2%
Tl	0.0	<1.4	NM	<0.43	<1.3	NM	21	NM	<0.6	<.7	NM
U	0.30	3.9	24%	0.47	1.4	NM	670	1%	<1.4	<1.6	NM
V	0.0	<.01	NM	<1.7E-03	<0.01	NM	0.67	35%	<1.3E-02	<.02	NM
Zn	0.014	0.19	7%	0.020	0.066	8%	1.7	66%	0.0921	0.115	1%
Zr	0.51	7.7	NM	0.72	2.5	NM	1.0	45%	1.5616	2.1	NM

	Washed Solids			Formated Slurry			Melter Feed		
	(wt%)	(g/100g Oxide)	(reproducibility)	(wt%)	(g/100g Oxide)	(reproducibility)	(wt%)	(g/100g Oxide)	(reproducibility)
NO_2^-	0.40	6.09	NM	0.156	1.45	NM	0.253	0.62	NA
NO_3^-	0.41	6.24	NM	0.633	5.90	NM	0.594	1.46	NA
F	0.013	0.20	NM	0.74	6.90	NM	0.933	2.29	NA
Cl^-	0.003	0.05	NM	0.78	7.27	NM	0.933	2.29	NA
SO_4^{-2}	0.106	1.61	NM	0.136	1.27	NM	0.191	0.47	NA
PO_4^{-3}	0.02	0.24	NA	<0.039	<.36	NM	<0.0038	<0.01	NA
TIC	0.13	1.97	NM	< 0.00008	<0.0008	NM	0.037	0.09	25%
TOC	0.64	9.70	NM	0.0066	0.06	NM	0.84	2.06	7%
TC	0.77	11.67	NM	0.0067	0.06	NM	0.87	2.13	8%
pH		12.6			5.3			5.8	

	Washed Solids	Formated Slurry	Melter Feed	MF Supernate	Glass
density (g/mL)	1.04	1.1	1.47	1.09	2.56
g TO/L (meas)	69	118	600	NM	2560
g TO/L (by ICP)	98	NM	560	19	2380
Fe(II)/Fe(III)					0.026

Notes:
1. "wt%" = g analyte/100 g sample; "wt% oxide" = g analyte oxide/100 g total oxide; "g/100 g oxide" = g analyte/100 g sample.
2. "wt% oxides" are calculated using total oxides as measured by elemental analysis except for in glass where sample weight is used for total oxides.
3. "reproducibility" = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
4. TIC = Total inorganic carbon; TOC = total organic carbon; TC = total carbon; NM = not measured.
5. "g TO/L (meas)" = g total oxides/liter measured by calcined weight; "g TO/L (calc)" = g total oxide/liter calculated from elemental analysis data.
6. The glass ICP data as reported in this table is from the set 1 analysis More detailed analysis can be found in Table A.6.

Table A.2. Chemical Composition of 101-AZ Core #2 Process Solutions and Product Glass

	Washed Solids			Melter Feed			MF Supernate	Glass		
	(wt%)	(wt% oxide)	(reproducibility)	(wt%)	(wt% oxide)	(reproducibility)	(µg/ml)	(wt%)	(wt% oxide)	(reproducibility)
Ag	0.017	0.11	31%	(0.029)	0.04	24%	<2.2	(0.026)	(0.028)	NM
Al	0.84	9.0	18%	1.25	2.7	•••	<7	1.53	2.9	0.2%
As	<0.024	<.18	NA	<0.098	<0.1	NA	<11	(0.047)	(0.06)	43%
B	<0.020	<.36	NA	2.2	7.5	7%	680	2.2	7.1	•••
Ba	0.044	0.28	20%	0.067	0.086	1%	0.30	0.11	0.12	1.3%
Be	4.1E-03	0.064	156%	1.20E-03	<6.4E-4	•••	0	9.0E-04	0.002	44%
Ca	0.19	1.5	58%	0.36	0.22	NA	8.4	0.5638	1.0	NM
Cd	0.51	3.3	20%	0.75	1.0	1%	<1.1	0.69965	0.80	•••
Ce	<0.046	<.32	NA	<0.24	<3	NA	<26	<0.19	<2	NA
Co	<0.059	<.47	NA	<0.46	<7	NA	<51	<0.55	<8	NA
Cr	0.018	0.15	16%	(0.032)	(0.054)	6%	<3.3	(0.037)	(0.05)	33%
Cu	0.019	0.14	24%	0.07	0.07	59%	<2.2	0.034	0.04	6%
Dy	<2.8E-03	<.018	NA	<0.015	<.020	NA	<2.2	<0.013	<.02	NA
Fe	5.6	46	20%	8.2	13	2%	(2.3)	8.1	12	•••
K	<0.10	<.71	NA	(1.5)	(2.0)	NA	6900	(1.4)	(1.7)	NA
La	0.27	1.81	20%	0.40	0.53	2%	<2.2	0.39	0.46	2%
Li	<3.1E-03	<.038	NA	2.5	6.0	1%	1400	2.7	5.7	3%
Mg	0.032	0.30	22%	0.062	0.087	5%	1.4	0.065	0.11	2%
Mn	0.085	0.76	18%	0.13	0.24	10%	0.20	0.13	0.20	5%
Mo	<2.1E-03	<.017	NA	<0.014	<.025	NA	(2.5)	(4.4E-3)	6.6E-03	NA
Na	1.4	11	NA	4.6	6.8	NA	1300	4.6	6.2	NA
Nd	0.17	1.1	19%	(0.26)	(0.34)	4%	<11	0.25	0.29	28%
Ni	0.40	2.9	NA	0.58	0.79	NA	<2.2	0.55	0.70	NA
P	<0.19	<2.4	NA	<0.42	<1.1	NA	<46	<0.5	<1.1	NA
Pb	(0.022)	0.14	35%	<0.074	<.092	NA	<7.7	<0.1	<1	NA
Re	<4.3E-03	<.029	NA	<0.017	<.023	NA	<2.2	<0.021	<.03	NA
Rh	<0.027	<.19	NA	<0.13	<.19	NA	<14	<0.12	<2	NA
Ru	(0.045)	(0.34)	23%	<0.078	<.12	NA	<8.8	<0.053	<.07	NA
Sb	<0.079	<.53	NA	<0.12	<.24	NA	<13	<0.089	<1	NA
Se	<0.026	<.21	NA	0.30	0.49	NA	<13	<0.2	<3	NA
Si	0.22	2.7	21%	21	50	10%	170	23	48	•••
Sr	0.031	0.21	23%	0.046	0.06	11%	1.2	0.046	0.054	11%
Te	<0.017	<.12	NA	<0.072	<.10	NA	<7.7	(0.043)	0.054	NA
Th	<0.033	<.21	NA	<0.18	<.24	NA	<20	<0.24	<3	NA
Ti	(3.8E-03)	0.036	37%	0.014	0.027	NA	<1.1	(0.02)	0.03	NA
Tl	<0.42	<2.7	NA	<3.4	<4.4	NA	<370	<0.6	<7	NA
U	(0.41)	2.7	34%	(1.7)	(2.3)	25%	(370)	(1.7)	2.0	NA
V	<2.4E-03	<.020	NA	<0.012	<.020	NA	<1.1	<0.013	<.02	NA
Zn	0.011	0.079	4%	0.05	0.07	71%	<1.1	0.066	0.1	7%
Zr	2.1	16	NA	3.3	5.2	NA	(1.7)	3.4	4.6	NA
density (g/ml)		1.10			1.20		1.04		2.67	
g TO/l (meas)		122			479		NM		2670	
g TO/l (calc)		112			1080				2450	
Fe(II)/Fe(III)									0.058	

	Washed Solids			Formated Slurry			MF Supernate	Melter Feed		
	(wt%)	(g/100g Oxide)	(reproducibility)	(wt%)	(g/100g Oxide	(reproducibility)	(µg/ml)	(wt%)	(g/100g Oxide)	(reproducibility)
NO2-	0.61	3.68	NM	0.22	2.40	NM	0.14	0.14	NM	NM
NO3-	0.31	1.87	NM	0.071	0.77	NM	0.20	0.20	NM	NM
F-	0.026	0.16	NM	0.12	1.42	NM	0.23	0.23	NM	NM
Cl-	4.0E-03	0.02	NM	0.54	5.88	NM	1.2	1.2	NM	NM
SO4-2	0.15	0.90	NM	0.084	1.00	NM		0.22	NM	NM
PO4-3	0.031	0.19	NM	<0.004	<0.01	NM		0.028	NM	NM
TIC	0.076	0.46	12%	0.078	0.85	5%		NM		
TOC	0.042	0.25	31%	0.31	3.38	8%		NM		
TC	0.12	0.72	19%	0.39	4.25	9%		NM		
pH		12.7			4.0		NM		9.4	

Notes:
1. "wt%" = g analyte/100 g sample; "wt% oxide" = g analyte oxide/100 g total oxide; "g/100 g oxide" = g analyte/100 g sample.
2. "wt% oxides" are calculated using total oxides as measured by elemental analysis except for in glass where sample weight is used for total oxides.
3. Reproducibility = (| analysis 1 - analysis 2 | / (analysis 1 + analysis 2)) / 2 x 100%
4. TIC = Total inorganic carbon; TOC = total organic carbon; TC = total carbon; NM = not measured.
5. "g TO/L (meas)" = g total oxides/liter measured by calcined weight; "g TO/L (calc)" = g total oxide/liter calculated from elemental analysis data.
6. "• • • • •" indicates that the reproducibility was less than 0.5%.
7. The glass ICP data as reported in this table is from the set 1 analysis. More detailed analyses can be found in Table A.7.

Table A.3. Chemical Composition of 102-AZ Core #1 Process Solutions and Product Glass

	Washed Solids			WS Condensate	Formated Slurry			FS Supernate		MF Supernate	Glass		
	(wt%)	(wt% oxide)	(reproducibility)	µg/ml	(wt%)	(wt% oxide)	(reproducibility)	µg/ml	(reproducibility)	(ug/ml)	(wt%)	(wt% oxide)	(reproducibility)
Ag	0.017	0.16	1%	<8.1E-03	8.3E-03	0.074	6%	NA	NA	<8.0E-03	(0.014)	(0.016)	23%
Al	1.275	21.5	3%	<0.19	1.2	18	2%	110	26%	<0.18	3.24	6.2	1%
As	< 0.01	<0.14	NA	<0 11	<0.020	<0.22	NA	NA	NA	(0.11)	<0.01	NA	NA
B	< 0.02	<0.54	NA	(0.10)	(0.012)	(0.34)	NA	12	0%	220	4.1	13	0.3%
Ba	0.014	0.14	5%	(0.01)	0.015	0.13	3%	3.9	0%	0.03	0.033	0.037	2%
Be	4.13E-04	0.010	3%	<3.3E-03	<6.2E-4	<0.014	NA	0.2	0%	<3.0E-03	<7E-03	NA	NA
Ca	0.113	1.4	43%	0.60	0.066	0.76	5%	470	0.02%	0.72	0.34	0.48	30%
Cd	0.361	3.7	4%	<7.8E-03	0.38	3.6	2%	2600	0.15%	0.33	0.9	1	0.9%
Ce	< 0.025	<0.26	NA	<0.11	<0.020	<0.21	NA	NA	NA	<0.10	<0.1	0.08	NA
Co	< 0 009	<0.11	NA	<0.19	<0.037	<0.43	NA	NA	NA	<0.19	<0.1	NA	NA
Cr	0.036	0.45	0%	<0 012	0 030	0.37	3%	2.8	0%	<0.011	0.083	0.12	2%
Cu	0.016	0.18	81%	<9.2E-03	(4.9E0-3)	(0.05)	20%	0.8	0%	<9.E-03	(0.015)	(0.019)	9%
Dy	< 0.001	<0 01	NA	<5.5E-03	<1.0E-03	<9.9E-03	NA	NA	NA	<5.0E-03	<0.07	NA	NA
Fe	3.122	40	4%	<.010	2.9	35	3%	2.1	0%	0.16	7.3	11	1.8%
K	< 0.08	<0.79	NA	<0.33	<0.062	<0.63	NA	260	2%	20	<1	NA	NA
La	0.109	1.14	3%	<0 013	0 097	0.94	2%	2.6	0%	<0.012	0.23	0.27	0.9%
Li	< 0.005	<0 089	NA	<7 7E-03	<1.5E-03	<0 026	NA	NA	NA	220	2.2	4.6	0.9%
Mg	0 025	0 36	1%	0 09	0 024	0 32	14%	180	0 04%	2.3	<0.1	0.11	NA
Mn	0 070	0 99	5%	0 01	0 081	1.1	2%	590	0.2%	0.05	0.19	0.3	9%
Mo	< 0 003	<0 038	NA	<0 013	<2.5E-03	<0 031	NA	NA	NA	(0.02)	<0.04	NA	NA
Na	1.011	12	NM	3 41	2 0	22	2%	22000	0.3%	2000	5.5	7.5	NA
Nd	0.083	0 9	2%	<0.052	0 062	0 61	2%	NA	NA	<0 05	(0.15)	(0 17)	4%
Ni	0.215	2.4	NM	0 11	<0 012	2 0	NA	620	0 07%	0 97	0 54	0.69	NA
P	< 0 12	<2.09	NA	<0 64	<9 8E-03	<2 3	NA	NA	NA	<0 61	(0 24)	(0 55)	0%
Pb	0.036	0.35	1%	<0 087	(0 028)	(0 25)	2%	NA	NA	<0 083	<0 09	0 11	NA
Re	< 0.004	<0 038	NA	<0 018	<3 4E-03	<0 033	NA	NA	NA	<0 017	NA	0 01	NA
Rh	<0 012	<0 12	NA	<0 086	<0 016	<0 17	11%	NA	NA	<0 082	<0 4	NA	NA
Ru	< 0 012	<0 13	NA	<0 036	(0 010)	(0 10)	2%	NA	NA	0 45	0 2	0 03	NA
Sb	< 0.042	<0 42	NA	<0 050	<9 4E-03	<0 093	NA	42	0%	<0 048	0 06	NA	NA
Se	< 0 018	<0.21	NA	<0 16	<0 030	<0 35	NA	NA	NA	<0 152	<0 1	NA	NA
Si	0.124	2.4	2%	3.2	0 454	8 1	3%	44	4%	24	24	51	1.3%
Sr	8.10E-03	0 09	6%	<4 0E-4	7 1E-03	0 070	3%	38	0 3%	0.12	(0 019)	(0 022)	23%
Te	< 0.018	<0 19	NA	<0 099	<0 019	<0.19	NA	NA	NA	<0 095	<0 7	0 05	NA
Th	< 0.021	<0.20	NA	<0 076	<0 014	<0 14	NA	NA	NA	<0.073	<1	NA	NA
Ti	0.004	0.053	3%	<5 6E-03	(4.5E-03)	(0 062)	9%	NA	NA	<5.E-03	0 035	0.059	7%
Tl	< 0.18	<1.73	NA	<0.57	<0 11	<1.0	NA	NA	NA	<0.55	<0.6	NA	NA
U	0.300	3.2	NM	<0.56	<0 11	<1.0	NA	210	0.05%	17	<2	0.85	NA
V	< 0 002	<0 026	NA	<9 4E-03	<1.8E-03	<0.022	NA	NA	NA	<9.E-03	<01	0.01	NA
Zn	0.010	0.11	47%	(0 01)	8.8E-03	0 091	3%	4.1	6%	(00.01)	<0.03	0.02	NA
Zr	0.528	6	NM	<7.9E-03	0.538	6.0	7%	NA	NA	(0.02)	1.4	1.9	NA

	Washed Solids		WS Condensate	Formated Slurry			Melter Feed
	(wt%)	(g/100g Oxide)	(µg/ml)	(wt%)	(g/100g Oxide)	(reproducibility)	(wt%)
NO_2	0.208	1.89	10.6	0.085	0.65	1.7%	NM
NO_3	0.076	0.69	7.5	0.168	1.28	<0.5%	NM
F^-	0.008	0.07	<0.25	<0.12	<0 9	NA	NM
Cl^-	0.002	0.02	<0.25	<0.024	<0.2	NA	NM
SO_4^{-2}	0 070	0.64	<0.5	0.013	0.10	7.1%	NM
PO_4^{-3}	0.021	0.19	<0.5	<0.005	<.04	NA	NM
TIC	0.305	2.78	NM	0 007	0.05	<0.5%	NM
TOC	0.108	0.98	NM	1.11	8.46	11.2%	NM
TC	0.305	2.78	NM	1.12	8.53	11.2%	NM
pH	NM		NM		5.0		8.8

	Washed Solids	Formated Slurry	FS Supernate	Melter Feed	MF Supernate	Glass
density (g/mL)	1.11	1.12	1 03	1 34	1 03	2.54
g TO/L (meas)	122	147	NM	515	NM	2540
g TO/L (by ICP)	124	134	36	NM	4	2530
Fe(II)/Fe(III)						0 047

Notes: 1. "wt%" = g analyte/100 g sample; "wt% oxide" = g analyte oxide/100 g total oxide; "g/100 g oxide" = g analyte/100 g sample
2. "wt% oxides" are calculated using total oxides as measured by elemental analysis except for as glass where sample weight is used for total oxides
3. "reproducibility" = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
4. TIC = Total inorganic carbon; TOC = total organic carbon; TC = total carbon; NM = not measured.
5. "g TO/L (meas)" = g total oxides/liter measured by calcined weight, "g TO/L (calc)" = g total oxide/liter calculated from elemental analysis data.
6. Washed Solids composition was determined by analysis adjusted for known, subsequent additions of NaNO3, NaNO2, and CaNO3.

Table A.4. Chemical Composition of NCAW Simulant Process Solutions and Product Glass

	Washed Solids		Melter Feed Slurry (Run 2)			Melter Feed Slurry		Melter Feed Supernate	Glass	
	(wt%)	(wt%oxide)	(wt%)	(wt%oxide)	reproducibility	(wt%)	(wt%oxide)	(µg/ml)	(wt%oxide)	(reproducibility)
Ag		<DL	NA	NA	NA		NA	NA	NA	NA
Al	0.302	10	0.61	3.2	0.8%	0.415	2.2	<0.6	2.3	0%
As	<0.016	<DL	<DL	<DL	NA	NA	NA	<1.6	NA	NA
B	0.0049	0.28	1.3	12	4%	1.3	12	240	11	2%
Ba	0.0224	0.44	0.041	0.13	2%	0.027	0.085	1.31	0.085	12%
Be	<4.E-03	<DL	NA	NA	NA	NA	NA	<0.04	NA	NA
Ca	0.0251	0.62	0.27	1.05	15%	0.24	0.93	11	1.0	NA
Cd	0.129	2.6	0.22	0.71	3%	0.15	0.48	<0.08	0.49	0%
Ce	0.035	0.76	0.061	0.21	0.7%	0.043	0.15	<0.8	0.17	12%
Co	<2.E-03	<DL	<DL	<DL	NA	NA	NA	<0.2	0.07	NA
Cr	0.026	0.68	0.042	0.17	9%	0.029	0.12	<0.4	0.15	40%
Cu	0.0172	0.38	0.034	0.12	0.9%	0.022	0.076	1.9	0.09	0%
Dy	<8.E-03	0	NA	NA	NA	NA	NA	<0.08	NA	NA
Fe	1.34	34	2.3	9.1	3%	1.5	6.0	2.2	6.2	8%
K	NM	<DL	0.4	1.34	NA	NA	NA	100	0.7	NA
La	0.0406	0.85	0.12	0.38	0.9%	0.077	0.25	<0.16	0.265	4%
Li	0.0028	0.11	0.68	4.1	8%	0.63	3.8	290	3.6	0%
Mg	(0.017)	0.50	0.2	0.94	4%	0.19	0.88	45	0.9	2%
Mn	0.0416	1.17	0.067	0.30	3%	0.045	0.20	0.27	0.36	83%
Mo	0.052	1.4	0.096	0.40	1.5%	0.06	0.25	450	0.24	0%
Na	0.674	16	2.7	10	NA	2.4	9.1	13000	8.8	NA
Nd	0.162	3.4	0.3	0.96	1.4%	0.19	0.64	<0.4	0.62	6%
Ni	1.23	2.8	0.2	0.72	NA	NA	NA	<0.4	0.51	NA
P	<0.02	<DL	NA	NA	NA	NA	NA	<2	NA	NA
Pb	<0.012	<DL	<DL	<DL	NA	NA	NA	<01.2	NA	NA
Re	NA	<DL	<DL	<DL	NA	NA	NA	NA	NA	NA
Rh	NA	<DL	<DL	<DL	NA	NA	NA	NA	NA	NA
Ru	(0.035)	0.82	0.056	0.20	17%	0.031	0.11	4.6	0.2	30%
Sb	<0.010	<DL	<DL	<DL	NA	NA	NA	<1.0	NA	NA
Se	NA	<DL	<DL	<DL	NA	NA	NA	NA	NA	NA
Si	0.145	5.5	8.2	49	42%	9.9	60	48	53.2	11%
Sr	0.0149	0.31	0.027	0.089	0%	0.018	0.06	3.4	0.1	0%
Te	<0.01	<DL	<DL	<DL	NA	NA	NA	<1.2	NA	NA
Th	NA	<DL	<DL	<DL	NA	NA	NA	NA	NA	NA
Ti	0.0023	0.068	0.024	0.11	18%	0.025	0.12	<.04	0.1	9%
Tl	NA	<DL	<DL	<DL	NA	NA	NA	NA	NA	NA
U	NA	<DL	<DL	<DL	NA	NA	NA	NA	NA	NA
V	<2.E-03	<DL	<DL	<DL	NA	NA	NA	<0.2	NA	NA
Y	0.0101	0.23	<DL	<DL	2%	0.013	0.046	<.14	0.05	0%
Zn	<4.E-03	0.00	<DL	<DL	NA	NA	NA	<4	0.15	NA
Zr	0.7	17	1.3	5	NA	0.85	3.2	(0.32)	3.5	NA

	Washed Solids		Formated Slurry (Run 3)		Melter Feed Slurry (Run 3)	
	wt %	g/100g oxide	wt %	g/100g oxide	wt %	g/100g oxide
NO_2^-	0.39	5.93	< 0.045	< 0.31	< 0.038	< 0.11
NO_3^-	0.4	6.09	0.53	3.68	0.37	1.08
F^-	0.009	0.13	0.028	0.19	0.014	0.04
Cl^-	< 0.019	< 0.29	< 0.018	< 0.12	< 0.015	< 0.04
TIC	0.138	2.1	0.61	4.26	0.29	0.86
TOC	0.011	0.2	0.16	1.12	0.11	0.31
TC	0.149	2.3	0.77	5.38	0.4	1.17
density	1.11		1.11		1.28	
TOx (meas)	112		161		438	
TOx (calc)	131		-		-	
pH	10		6.9		8.6	

Notes:
1. "wt%" = g analyte/100 g sample; "wt% oxide" = g analyte oxide/100 g total oxide; "g/100 g oxide" = g analyte/100 g sample.
2. "wt% oxides" are calculated using total oxides as measured by elemental analysis except for in glass where sample weight is used for total oxides.
3. "reproducibility" = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2)) / 2 * 100%.
4. TIC = Total inorganic carbon; TOC = total organic carbon; TC = total carbon; NM = not measured.
5. "g TO/L (meas)" = g total oxides/liter measured by calcined weight; "g TO/L (calc)" = g total oxide/liter calculated from elemental analysis data.

Table A.5. Chemical Composition of 102-AZ Core #1 Simulant Process Solutions

	Washed Solids		Formated Slurry				FS Supernate	
	Target	Measured	Analysis 1		Analysis 2			
	(wt%oxide)	(wt%oxide)	(wt%)	(wt%oxide)	(wt%)	(wt%oxide)	(µg/g)	eproducibility)
Ag	0.14	0.13	0.02	0	0.01	0.07	<.05	NA
Al	19.54	18.72	1.4	20.7	1.4	18.3	<.3	NA
B		0.34	NA				5.8	2%
Ba	0.12	0.11	0.016	0.1	0.02	0.14	0.37	25%
Be			NA				<.03	NA
Bi							<.60	NA
Ca	0.90	0.84	0.088	1	0.1	0.99	22	79%
Cd	3.35	3.25	0.49	4.4	0.43	3.46	0.18	NA
Ce	0.25				0.02	0.21	<.4	NA
Co			0.0002	0			<.10	NA
Cr	0.43	0.41	0.037	0.4	0.04	0.42	<.20	NA
Cs	0.57							
Cu	0.14	0.13	0.015	0.1	0.01	0.14	0.31	NA
Dy			NA				<.06	NA
Eu							<.04	NA
Fe	36.16	35.01	3.39	37.9	3.5	34.48	4.9	2%
K			0.11	1			85	81%
La	1.04				0.11	0.91	<.10	NA
Li			NA				<.05	NA
Mg	0.33	0.17	0.028	0.4	0.03	0.35	86	8%
Mn	0.89	0.83	0.098	1.2	0.085	0.91	0.12	NA
Mo			NA				<.1	NA
Na	25.78		2.24	23.6	2	19.7	26000	3%
Nd	2.45				0.28	2.25	1.5	NA
Ni	2.15	2.01	0.236	2.3	0.22	1.97	3.7	17%
P	1.90	1.53	0.032	0.6	0.24	3.87	12	0.5%
Pb	0.32	0.29	0.041	0.3	0.06	0.28	<.8	NA
Pd	0.13							
Re	0.03		NA					
Rh	0.11							
Ru	0.43							
S							250	3%
Si	2.03	0.38	0.012	0.2	0.42	6.19	11	18%
Sn							1.3	NA
Sr	0.07	0.07			0.01	0.07	1.4	4%
Te	0.17				0.02	0.14	<.6	NA
Ti	0.03		0.001	0			<.03	NA
V			NA				<.1	NA
Y			NA				<.03	NA
Zn	0.07	0.06	0.0085	0.1	0.06	0.56	10	21%
Zr	5.79	1.04	0.14	1.5	0.44	4.15	10	0.7%

	Washed Solids		Formated Slurry		
	Target	Measured			
	(g/100g oxide)	(g/100g oxide)	(wt%)	(g/100g oxide)	(reproducibility)
NO_2^-	9.6	8.75	TBD	TBD	TBD
NO_3^-	7.75	NM	TBD	TBD	TBD
F^-	0.065	NM	TBD	TBD	TBD
Cl^-	0.032	0.046	TBD	TBD	TBD
SO_4^{-2}	0.56	0.57	TBD	TBD	TBD
PO_4^{-3}	0.17	2.05	TBD	TBD	TBD
TIC	0.72	NM	TBD	TBD	TBD
TOC	0.86	0.86	TBD	TBD	TBD
TC	1.58	NM	TBD	TBD	TBD
pH		10.8	TBD	TBD	TBD

	Washed Solids	Formated Slurry	
		Analysis 1	Analysis 2
density (g/	1.14	1.14	1.14
TOx (meas)		147	147
TOx (by T	125.00	146	162

Notes:
1. "wt%" = g analyte/100 g sample; "wt% oxide" = g analyte oxide/100 g total oxide; "g/100 g oxide" = g analyte/100 g sample.
2. "wt% oxides" are calculated using total oxides as measured by elemental analysis.
3. "reproducibility" = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
4. TIC = Total inorganic carbon; TOC = total organic carbon; TC = total carbon; NM = not measured.
5. "g TO/L (meas)" = g total oxides/liter measured by calcined weight; "g TO/L (calc)" = g total oxide/liter calculated from elemental analysis data.

Table A.6. Chemical Composition of 101-AZ Core #1 Glass

	(Set 1) Na_2O_2 (wt% oxide)	(Set 2) Na_2O_2 (wt% oxide)	(Set 1) KOH (wt% oxide)	(Set 2) KOH (wt% oxide)	(Set 1&2) Na/K Ave. (wt% oxide)	(Set 2) HF (wt% oxide)	Adjusted (wt% oxide)	Standard Deviation[g]	%RSD
Ag_2O	0.11	0.11	0.09	0.10	0.10	0.11	0.10[a]	0.010	9.34
Al_2O_3	5.13	5.03	5.25	4.93	5.08	5.22	5.22[b]	0.137	2.70
As_2O_3									
B_2O_3	9.26	9.35	9.34	9.20	9.29	N/A	9.68[e]	0.069	0.75
BaO	0.05		0.05		0.05	0.05	0.05[a]	0.00	0.00
BeO	0.0024	0.0019	0.0013	0.0017	0.0018	0.0023	0.002[a]	0.0005	25.06
CaO	0.90	0.74	0.98	0.65	0.82	0.97	0.97[b]	0.150	18.32
CdO	0.30	0.31	0.30	0.31	0.30	0.33	0.30[a]	0.006	1.92
CeO_2							0.14[c]		
Co_2O_3							0.10[c]		
Cr_2O_3	0.20	0.20	0.19	0.19	0.20	0.20	0.20[a]	0.006	2.96
CuO	0.04	0.03		0.03	0.02	0.03	0.02[a]	0.006	28.87
Dy_2O_3							0.06[c]		
Fe_2O_3	9.64	9.79	9.61	9.64	9.67	10.18	10.18[b]	0.081	0.84
K_2O	2.08	2.15	N/A	N/A	2.12	2.03	2.12[a]	0.049	2.34
La_2O_3	0.15	0.14	0.14	0.14	0.14	0.15	0.14[a]	0.005	3.51
Li_2O	3.42	3.45	3.49	3.39	3.44	3.56	3.56[b]	0.043	1.24
MgO	0.73	0.73	0.73	0.72	0.73	0.77	0.77[b]	0.005	0.69
MnO_2	0.46	0.47	0.46	0.46	0.46	0.48	0.48[b]	0.005	1.08
MoO_3	0.01				0.01	0.01	0.01[a]		
Na_2O	N/A	N/A	9.39	10.18	9.79	10.68	9.79[a]	0.558	5.70
Nd_2O_3	0.16	0.16	0.11	0.11	0.13	0.20	0.16[a,b]	0.029	22.21
NiO	0.26	0.31	N/A	N/A	0.29	0.29	0.29[a]	0.035	12.41
P_2O_5						0.55	0.55[b]		
PbO	0.25	0.21	0.17	0.17	0.20	0.21	0.20[a]	0.038	19.15
ReO_2									
Rh_2O_3									
RuO_2									
Sb_2O_3						0.01			
SeO_2						0.09			
SiO_2	48.14	49.56	47.86	48.75	48.58	46.43	51.97[e]	0.754	1.55
SrO	0.03	0.04	0.04	0.03	0.04	0.04	0.04[a]	0.006	16.50
TeO_2						0.07			
ThO_2									
TiO_2	0.12	0.13	0.11	0.11	0.12	0.12	0.12[a]	0.010	8.15
Tl_2O_3									
U_3O_8						0.69	0.41[d]		
V_2O_3									
ZnO	0.05	0.01		0.01	0.03	0.05	0.05[b]	0.023	76.98
ZrO_2	N/A	N/A	2.11	1.88	1.99	1.97	1.99[a]	0.163	8.17
SO_3							0.40[c]		
Total[f]	93.01	94.96	92.74	93.46	93.58	94.76	100.07		

Notes:

a. Analysis by ICP-ES (Na_2O_2 and KOH fusion/dissolution) preparation methods.

b. Analysis by ICP-ES (HF digestion) preparation method.

c. Value calculated from washed solids analysis (same method as "a"). Below detection limits in glass.

d. Analysis by fluoresence (typically fluoresence would be used for U; however, the number appended would be a flier when compared to leached uranium in Core 1 and Core 2 and ICP-ES analyses in glass and washed solids.

e. Adjusted value by dividing ICP-ES (Na_2O_2. KOH) result by % yield of constituent determined by ATM-10 and NBS 688 analysis.

f. The totals includes values obtained from alternate preparation technique for elements lost during preparation.

g. The standard deviations are based on the number of Na_2O_2 and KOH samples present.

Table A.7. Chemical Composition of 101-AZ Core #2 Glass

	(Set 1) Na_2O_2 (wt% oxide)	(Set 2) Na_2O_2 (wt% oxide)	(Set 1) KOH (wt% oxide)	(Set 2) KOH (wt% oxide)	(Set 1&2) Na/K Ave. (wt% oxide)	(Set 2) HF (wt% oxide)	Adjusted (wt% oxide)	Standard Deviation[g]	%RSD
Ag_2O	0.03	0.04		0.03	0.03	0.03	0.03[a]	0.006	17.32
Al_2O_3	2.89	2.80	2.90	2.79	2.85	3.16	2.85[a]	0.058	2.04
As_2O_3									
B_2O_3	7.12	7.05	7.14	7.00	7.08		7.38[e]	0.065	0.91
BaO	0.07	0.07	0.07	0.07	0.07	0.07	0.07[a]	0.000	0.00
BeO	0.003	0.0046	0.0021	0.0032	0.0032	0.0036	0.003[a]	0.001	32.06
CaO	0.27	0.33	0.34	0.29	0.31	0.35	0.35[b]	0.033	10.74
CdO	0.79	0.80	0.80	0.80	0.80	0.85	0.80[a]	0.005	0.63
CeO_2						0.13			
Co_2O_3									
Cr_2O_3	0.06	0.07	0.05	0.05	0.06	0.06	0.06[a]	0.010	16.65
CuO	0.04	0.04	0.01	0.04	0.03	0.04	0.03[a]	0.015	46.15
Dy_2O_3									
Fe_2O_3	11.45	11.73	11.41	11.55	11.53	12.25	12.25[b]	0.143	1.24
K_2O	1.65				1.65	1.55	1.65[a]		
La_2O_3	0.46	0.46	0.45	0.45	0.46	0.47	0.46[a]	0.006	1.27
Li_2O	5.68	5.72	5.84	5.59	5.71	5.94	5.94[b]	0.104	1.83
MgO	0.08	0.10	0.07	0.08	0.08	0.12	0.12[b]	0.013	15.25
MnO_2	0.20	0.20	0.19	0.21	0.20	0.21	0.21[b]	0.008	4.08
MoO_3	0.01				0.01				
Na_2O			5.97	6.50	6.24	6.83	6.24[a]	0.378	6.06
Nd_2O_3	0.46	0.45	0.35	0.41	0.42	0.43	0.42[a]	0.050	11.96
NiO	0.66	0.72			0.69	0.71	0.69[a]	0.042	6.15
P_2O_5						0.88	0.88[b]		
PbO		0.13		0.09	0.11	0.09	0.11[a]	0.028	25.71
ReO_2									
Rh_2O_3									
RuO_2							0.09[c]		
Sb_2O_3									
SeO_2									
SiO_2	47.90	51.30	48.09	51.11	49.60	48.39	53.09[e]	1.857	3.74
SrO	0.05	0.05	0.05	0.05	0.05	0.05	0.05[a]	0.000	0.00
TeO_2	0.05				0.05	0.05	0.05[a]		
ThO_2									
TiO_2	0.03	0.03		0.24	0.08	0.04	0.08[a]	0.121	151.55
Tl_2O_3									
U_3O_8	2.03	1.39			1.71	1.01	1.71[a]	0.453	
V_2O_3									
ZnO	0.02	0.02		0.03	0.02	0.02	0.02[a]	0.006	24.74
ZrO_2			4.58	4.26	4.42	4.41	4.42[a]	0.226	5.12
SO_3							0.32[c]		
Total[f]	92.70	94.28	90.61	92.38	94.29	95.17	100.31		

Notes:

a. Analysis by ICP-ES (Na_2O_2 and KOH fusion/dissolution) preparation methods.

b. Analysis by ICP-ES (HF digestion) preparation method.

c. Value calculated from washed solids analysis (same method as "a"). Below detection limits in glass.

d. Analysis by fluoresence (typically fluoresence would be used for U; however, the number appended would be a flier when compared to leached uranium in Core 1 and Core 2 and ICP-ES analyses in glass and washed solids.

e. Adjusted value by dividing ICP-ES (Na_2O_2, KOH) result by % yield of constituent determined by ATM-10 and NBS 688 analysis.

f. The totals includes values obtained from alternate preparation technique for elements lost during preparation.

g. The standard deviations are based on the number of Na_2O_2 and KOH samples present.

Table A.8. Evaluation of Sample Preparation Methods Using ATM-10 Glass

	(Set 2) Na_2O_2 (wt% oxide)	(Set 2) KOH (wt% oxide)	(Set 2) Na/K Ave. (wt% oxide)	(Set 2) HF (wt% oxide)	MCC Measured (wt% oxide)	% Yield[b] Na/K Ave.	% Yield[b] HF
Ag_2O	0.01		0.01				
Al_2O_3	6.33	6.34	6.34	6.77	6.65	95%	102%
As_2O_3							
B_2O_3	8.82	8.79	8.81		9.17	96%	
BaO				0.03	0.05		58%
BeO							
CaO	0.28	0.26	0.27	0.60	0.60	45%	100%
CdO							
CeO_2					0.07		
Co_2O_3							
Cr_2O_3	0.27	0.26	0.26	0.13	0.24	111%	54%
CuO	0.01	0.01	0.01	0.01			
Dy_2O_3							
Fe_2O_3	10.89	10.88	10.89	11.35	11.53	94%	98%
K_2O	3.37		3.37	3.22	3.34	101%	96%
La_2O_3	0.03	0.02	0.03	0.02	0.03	102%	86%
Li_2O	2.72	2.70	2.71	2.85	2.88	94%	99%
MgO	1.09	1.09	1.09	1.16	1.15	95%	101%
MnO_2	1.21	1.21	1.21	1.24	1.29	93%	96%
MoO_3				0.01			
Na_2O		10.88	10.88	11.45	10.53	103%	109%
Nd_2O_3	0.17	0.14	0.16	0.18	0.17	92%	107%
NiO	0.34		0.34	0.30	0.30	116%	101%
P_2O_5	1.81	2.22	2.02	2.61	2.34	86%	111%
PbO	0.02		0.02	0.02			
ReO_2							
Rh_2O_3					0.01		
RuO_2					0.06		
Sb_2O_3							
SeO_2	0.09		0.09	0.09			
SiO_2	42.94	42.75	42.85	41.44	45.84	94%	91%
SrO	0.03	0.03	0.03	0.03	0.03	107%	112%
TeO_2				0.05			
ThO_2	3.14	3.13	3.14	1.62	3.29	95%	49%
TiO_2	0.90	0.88	0.89	0.93	0.86	103%	108%
Tl_2O_3							
U_3O_8					0.53		
V_2O_3	0.01		0.01	0.01			
ZnO							
ZrO_2		0.40	0.40	0.31	0.25	162%	124%
SO_3					0.307		
Total[a]	95.76	95.70	95.82	95.24	101.52		

Notes:
a. The totals includes values obtained from alternate preparation technique for elements lost during preparation.
b. % Yield = (Analytical Measured/MCC Measured) x 100%.

Table A.9. Evaluation of Sample Preparation Methods Using NBS Basalt 688 Standard

	(Set 2) Na_2O_2 (wt%)	(Set 2) KOH (wt%)	(Set 2) Na/K Ave. (wt%)	(Set 2) HF (wt%)	NBS Comp. (wt%)	% Yield[b] Na/K Ave.	% Yield[b] HF
Ag_2O							
Al_2O_3	15.94	16.06	16.00	8.26	17.40	92%	47%
As_2O_3							
B_2O_3							
BaO				0.01			
BeO							
CaO	11.25	11.27	11.26	5.91	12.20	92%	48%
CdO							
CeO_2							
Co_2O_3		0.50	0.50				
Cr_2O_3	0.05	0.05	0.05	0.04			
CuO	0.01	0.01	0.01	0.01			
Dy_2O_3							
Fe_2O_3	9.59	9.69	9.64	7.22	10.40	93%	69%
K_2O					0.19		
La_2O_3							
Li_2O							
MgO	7.96	8.01	7.99	3.22	8.40	95%	38%
MnO_2	0.19	0.20	0.20	0.12	0.17	115%	71%
MoO_3							
Na_2O		2.21	2.21	1.17	2.15	103%	54%
Nd_2O_3							
NiO				0.02			
P_2O_5				0.34	0.14		243%
PbO	0.12		0.12	0.01			
ReO_2							
Rh_2O_3							
RuO_2							
Sb_2O_3				0.01			
SeO_2				0.07			
SiO_2	45.40	43.56	44.48	42.20	48.40	92%	87%
SrO	0.02	0.02	0.02	0.01			
TeO_2				0.01			
ThO_2							
TiO_2	1.12	1.11	1.11	1.17	1.20	93%	97%
Tl_2O_3							
U_3O_8							
V_2O_3	0.03	0.03	0.03	0.03			
ZnO							
ZrO_2							
SO_3							
Total[a]	93.89	92.72	93.31	69.83	100.65		

Notes:
a. The totals includes values obtained from alternate preparation technique for elements lost during preparation.
b. % Yield = (Analytical Measured/NBS Composition) x 100%.

Table A.10. Leach Test Information and Leachate Analysis for 101-AZ Core #1

	7-Day PCT (Hot Cell Test #4)				28 Day MCC-1 (Hot Cell Test #1)				
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Blank 1 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)
Al	3.00	2.70	2.60		1.95	1.63	0.93	0.063	
As					0.091				
B	8.70	7.50	7.20		2.64	2.25	1.46		
Ba					0.003	0.006	0.006	0.005	
Ca	0.600	0.200	0.200	0.070	0.694	0.786	0.383	0.167	0.308
Cd			0.020		0.024		0.004	0.007	
Cr		0.060	0.050		0.092	0.076	0.044		0.010
Cu					0.018	0.148	0.006	0.014	0.031
Fe	0.500	0.720	0.800		0.201	0.200	0.020	0.030	0.091
K	<10	4.00	3.00		1.98	3.20	1.16	0.314	2.30
Li	7.50	7.60	7.30		1.46	1.26	0.80		
Mg		0.300			0.318	0.355	0.219	0.024	0.058
Mn		0.020	0.040	0.020	0.094	0.226	0.010	0.006	0.425
Mo	0.400				0.013				
Na	21.0	21.0	21.0	0.100	6.96	7.43	3.77	0.389	1.60
Nd									
Ni					0.044	0.106	0.010	0.016	0.104
P		0.300	0.300		0.270				
Pb					0.045	0.125			
Re									
Ru					0.008		0.009	0.223	
Si	38.0	38.0	36.0		26.2	23.7	15.3	4.17	6.47
Sr		0.030			0.012	0.014	0.011	0.002	0.002
Ti					0.005	0.008		0.002	
U					0.318				
Zn					0.038	0.142	0.026	0.085	0.070
Zr					0.003	0.011			
NO_2^-	<0.5	<0.5	<0.5	<0.5	<4	NM	<4	<4	NM
NO_3^-	<0.5	<0.5	<0.5	<0.5	<4	NM	<4	<4	NM
F^-	2.90	1.50	3.10	<0.25	<2	NM	<2	<2	NM
Cl^-	3.00	3.10	2.90	<0.25	2.50	NM	2.40	<2	NM
SO_4^{2-}	2.20	1.90	2.10	<0.5	<4	NM	<4	6.60	NM
PO_4^{3-}	1.40	1.80	1.20	<0.5	<4	NM	<4	<4	NM
Br^-	<0.25	<0.25	<0.25	<0.25	<2	NM	<2	<2	NM
Initial pH	7.10	7.10	7.10	7.10	7.12	7.12	7.12	7.12	7.12
Final pH	7.58	7.49	7.41	6.57	8.70	7.92	7.56	5.70	6.51
Leach Vessel	Stainless	Stainless	Stainless	Stainless	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica
SA/V (m^{-1})	2000	2000	2000	NA	10.0	10.0	10.0	NA	NA
Sample Log # (ICP)	94-03205	94-03206	94-03207	94-03194	91-0199	91-2787	91-0200	91-0198	91-2785
Sample Log # (IC)	94-03200	94-03201	94-03202	94-03188	90-9537		90-9538	90-9536	

Notes:

1. Samples #1 & #2 (hot cell test #4) had matrix interference with fluorine, actual values could be as much as 10% higher.
2. Sample #3 (hot cell test #4) had matrix interference with fluorine, actual value could be half or less of reported value.
3. NM = not measured; NA = not applicable; PCT = Product Consistency Test; MCC-1 = Materials Characterization Center (MCC-1) Test.

Table A.11. Leach Test Information and Leachate Analysis for 101-AZ Core #2

	7-Day PCT (Hot Cell Test #3)					28 Day MCC-1 (Hot Cell Test #1)				
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)
Al	5.00	4.50	6.00			1.03	0.661	0.613	0.063	
As						0.071				
B	9.00	9.35	10.0	0.300		1.74	1.32	1.29		
Ba						0.001	0.002	0.004	0.005	
Ca	1.00	2.00	10.0	0.700	0.600	0.109	0.168	0.199	0.167	0.308
Cd		0.150	0.750			0.003			0.007	
Cr						0.026	0.019	0.019		0.010
Cu						0.011	0.006	0.013	0.014	0.031
Fe	0.500	6.00	21.0	0.300	0.450	0.038	0.026	0.085	0.030	0.091
K						1.46	1.06	1.41	0.314	2.30
Li	14.0	14.5	20.0			2.06	1.60	1.68		
Mg						0.023	0.038	0.047	0.024	0.058
Mn				0.060	0.070	0.002	0.002	0.005	0.006	0.425
Mo						0.008				
Na	9.40	9.90	10.0			3.75	2.93	3.04	0.389	1.60
Nd										
Ni						0.012	0.009	0.019	0.016	0.104
P						0.223	0.248			
Pb								0.133		
Re										
Ru						0.011	0.007		0.223	
Si	68.0	69.0	72.5			23.7	19.0	19.2	4.17	6.47
Sr						0.003	0.009	0.009	0.002	0.002
Ti						0.004	0.002	0.005	0.002	
U						0.379				
Zn						0.021	0.016	0.020	0.085	0.070
Zr	0.200	0.300				0.003				
NO_2^-	NM	NM	NM	NM	NM	<4	<4	NM	<4	NM
NO_3^-	NM	NM	NM	NM	NM	<4	<4	NM	<4	NM
F^-	NM	NM	NM	NM	NM	<2	<2	NM	<2	NM
Cl^-	NM	NM	NM	NM	NM	2.40	2.60	NM	<2	NM
SO_4^{2-}	NM	NM	NM	NM	NM	<4	<4	NM	6.60	NM
PO_4^{3-}	NM	NM	NM	NM	NM	<4	<4	NM	<4	NM
Br^-	NM	NM	NM	NM	NM	<2	<2	NM	<2	NM
Initial pH	7.03	7.03	7.03	7.03	7.03	7.12	7.12	7.12	7.12	7.12
Final pH	9.83	9.88	9.84	5.75	5.12	8.54	8.25	8.00	5.70	6.51
Leach Vessel	Stainless	Stainless	Stainless	Stainless	Stainless	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica
SA/V (m^{-1})	2000	2000	2000	NA	NA	10.0	10.0	10.0	NA	NA
Sample Log # (ICP)	93-9392	93-9393	93-9394	93-9390	93-9391	91-0201	91-020	91-2787	91-0198	91-2785
Sample Log # (IC)						90-9539	90-9540		90-9536	

Notes:

1. Sample #3 (hot cell test #3) was diluted to 7X because of limited sample, causing the analytes to be close to detection limits (i.e., 8xDL for B, 7xDL for Li, 2xDL for Na, 13xDL for Si) and decreased accuracy.
2. Anion samples (hot cell test #3) leaked during transport and were cross contaminated with HNO3. The results are not valid and therefore not reported.
3. Boron spike recoveries for samples 1-3 (hot cell test #3) were 104%, 104%, and 103%, respectively.
4. NM = not measured; NA = not applicable; PCT = Product Consistency Test; MCC-1 = Materials Characterization Center (MCC-1) Test.

Table A.12. Leach Test Information and Leachate Analysis for 102-AZ Core #1

	7-Day PCT (Hot Cell Test #5)					
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Sample Ave. (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)
Al	9.29	9.04	8.97			
As	0.109	0.107	0.113			
B	17.6	17.4	16.9	17.3	0.016	0.009
Ba						
Ca	0.093		0.057		0.052	
Cd	0.167	0.165	0.165			
Cr	0.061	0.059	0.075			
Cu	0.011	0.011	0.013		0.019	0.011
Fe	2.54	2.49	2.49			
K						
Li	13.2	13.0	12.7	13.0	0.003	0.003
Mg						
Mn	0.039	0.055	0.038		0.252	0.068
Mo						
Na	6.30	5.40	5.30	5.67	0.232	0.212
Nd						
Ni	0.144	0.153	0.156		0.052	0.042
P	1.02	0.946	0.885			
Pb	0.078	0.062	0.066			
Re						
Ru						
Si	76.5	75.0	73.6	75.0	0.405	0.124
Sr						
Ti	0.010	0.010	0.009			
U						
Zn	0.027		0.021		0.027	0.024
Zr	0.497	0.501	0.498			
NO_2^-	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
NO_3^-	0.60	6.60	0.600	2.60	<0.5	0.600
F^-	2.80	2.80	2.70	2.77	<0.25	<0.25
Cl^-	1.70	1.60	2.00	1.77	0.600	0.600
SO_4^{2-}	1.10	1.00	1.00	1.03	0.800	0.600
PO_4^{3-}	2.70	2.80	2.70	2.73	<0.5	<0.5
Br^-	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25
Initial pH	6.16	6.16	6.16		5.54	5.54
Final pH	9.33	9.24	9.21		6.36	6.70
Leach Vessel	Stainless	Stainless	Stainless		Stainless	Stainless
SA/V (m^{-1})	2000	2000	2000		NA	NA
Sample Log # (ICP)	95-002646	95-002647	95-002648		95-002639	95-002640
Sample Log # (IC)	95-02634	95-02635	95-02636		95-02627	95-02628

Notes:

1. NM = not measured; NA = not applicable; PCT = Product Consistency Test.
2. Boron spike recoveries for blanks 1 and 2 were 114% and 127% respectively

Table A.13. Leach Test Information and Leachate Analysis for 101-AZ Core #1 Simulant

	7-Day PCT (Hot Cell Test #4)						7-Day PCT (Lab Test #6)			28 Day MCC-1 (Lab Test #7)	
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Sample 4 (ug/ml)	Sample Ave (ug/ml)	Blank (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample Ave (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)
Al	5.70	5.70	5.30	4.40	5.28		5.80	5.80		2.70	2.60
As											
B	13.0	13.0	12.0	10.0	12.0		14.0	14.0	14.0	4.30	4.10
Ba											
Ca	0.080	0.070	0.080	0.090	0.080	0.070	0.070	0.080		0.080	0.090
Cd	0.010	0.010	0.007		0.009		0.020	0.010			
Cr	0.050	0.050	0.050	0.040	0.048						
Cu											
Fe	1.50	1.40	0.850	0.720	1.12		1.30	1.40		0.120	0.130
K	4.00	4.00	4.00	4.00	4.00		6.00	6.00	6.00	3.00	3.00
Li	8.60	8.80	7.90	7.10	8.10		8.10	8.20	8.15	2.00	1.90
Mg	0.200	0.200			0.200		0.200	0.200			
Mn	0.063	0.053	0.050	0.030	0.049	0.020	0.040	0.040		0.006	0.006
Mo							0.030	0.030			
Na	31.0	32.0	28.0	25.0	29.0	0.100	29.0	29.0	29.0	9.20	8.90
Nd							0.040	0.030			
Ni											
P	0.900	0.900	0.800	0.700	0.825		0.900	0.900		0.300	0.200
Pb											
Re											
Ru											
Si	68.0	68.0	62.0	53.0.	62.8		64.0	64.0	64.0	25.0	24.0
Sr											
Ti	0.008	0.007			0.008		0.007	0.008			
U											
Zn											
Zr							0.130	0.080			
NO_2	<0.5	<0.5	<0.5	<0.5	NA	<0.5	NM	NM	NM	NM	NM
NO_3	<0.5	<0.5	<0.5	<0.5	NA	<0.5	NM	NM	NM	NM	NM
F^-	<0.25	<0.25	<0.25	<0.25	NA	<0.25	NM	NM	NM	NM	NM
Cl^-	<0.25	<0.25	0.3	0.4	0.4	<0.25	NM	NM	NM	NM	NM
SO_4^{2-}	2.1	1.9	1.4	1.6	1.8	<0.5	NM	NM	NM	NM	NM
PO_4^{3-}	2.8	2.8	2.5	2.2	2.6	<0.5	NM	NM	NM	NM	NM
Br	<0.25	<0.25	<0.25	<0.25	NA	<0.25	NM	NM	NM	NM	NM
Initial pH	7.10	7.10	7.10	7.10		7.10	5.82	5.82		5.99	5.99
Final pH	9.96	9.96	9.80	9.86	9.90	6.57	10.07	10.12	10.10	9.50	9.47
Leach Vessel	Stainless	Stainless	Stainless	Stainless		Stainless	Teflon	Teflon		Teflon	Teflon
SA/V (m^{-1})	2000	2000	2000	2000		NA	2000	2000		10.0	10.0
Sample Log # (ICP)	94-03195	94-03196	94-03208	94-03209		94-03194	92-06994	92-06995		92-08087	92-08088
Sample Log # (IC)	94-03189	94-03190	94-03203	94-03204							

Notes

1 Samples #1-2 (hot cell test #4) were prepared and washed in the laboratory
2 Samples #3-4 (hot cell test #4) were washed in the hot cell.
3 Second blank sample (hot cell test #4) was spilled.
4 NM = not measured; NA = not applicable; PCT = Product Consistency Test, MCC-1 = Materials Characterization Center (MCC-1) Test.

Table A.14. Leach Test Information and Leachate Analysis for 101-AZ Core #2 Simulant

	7-Day PCT (Hot Cell Test #3)					7-Day PCT (Lab Test #8)		7-Day PCT (Lab Test #10)			28 Day MCC-1 (Lab Test #9)	
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Blank (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)
Al	3.00	3.00	3.00			2.80	2.80	2.00	2.50		1.60	1.60
As												
B	16.0	16.0	15.0	0.300		15.0	15.0	16.0	18.0		3.20	3.30
Ba												
Ca	0.700	1.00	1.00	0.700	0.600		0.050					
Cd	0.095					0.010	0.010				0.008	
Cr												
Cu												
Fe	4.00	1.50	1.15	0.300	0.450	0.610	0.570	0.800	1.10		0.060	0.050
K						4.00	5.00				2.00	2.00
Li	20.0	21.0	20.0			19.0	19.0	21.0	21.5		3.30	3.30
Mg												
Mn				0.060	0.070	0.006	0.005					
Mo												
Na	24.0	23.0	22.0			21.0	21.0	23.0	26.0		5.80	5.80
Nd							0.030					
Ni												
P	2.00	2.00	2.00			2.00	2.00	2.00	2.00		0.400	0.400
Pb												
Re												
Ru												
Si	92.5	92.0	89.0			85.0	85.0	90.0	94.0		25.0	25.0
Sr												
Ti												
U												
Zn												
Zr						0.050	0.050					
Initial pH	7.03	7.03	7.03	7.03	7.03	5.82	5.82	6.12	6.12	6.12	5.99	5.99
Final pH	10.02	10.07	10.09	5.75	5.12	10.34	10.34	10.03	9.98	7.88	9.63	9.61
Leach Vessel	Stainless	Stainless	Stainless	Stainless	Stainless	Teflon	Teflon	Stainless	Stainless	Stainless	Teflon	Teflon
SA/V (m^{-1})	2000	2000	2000	NA	NA	2000	2000	2000	2000	NA	10.0	10.0
Sample Log # (ICP)	93-9384	93-9385	93-9386	93-9390	93-9391	92-06996	92-06997	93-10234	93-10235	93-10231	92-08089	92-08090

Notes:

1. Samples #1-2 (hot cell test #3) were prepared and washed in the laboratory.
2. Samples #3-4 (hot cell test #3) were washed in the hot cell
3. Second PCT (hot cell test #3) blank sample was spilled.
4. NM = not measured; NA = not applicable; PCT Product Consistency Test; MCC-1 = Materials Characterization Center (MCC-1) Test.

Table A.15. Leach Test Information and Leachate Analysis for 102-AZ Core #1 Simulant

	7-Day PCT (Hot Cell Test #5)								7-Day PCT (Lab Test #11)			28 Day MCC-1 (Lab Test #12)	
	Sample 1 (uncleaned) (ug/ml)	Sample 2 (uncleaned) (ug/ml)	Sample 3 (ug/ml)	Sample 4 (ug/ml)	Sample Ave. (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Blank Ave (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample Ave. (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)
Al	9.57	9.64	9.22	8.94					9.31	9.37	9.34	3.87	4.18
As	0.115	0.110	0.102	0.101									
B	26.6	26.5	21.8	22.3	24.3	0.016	0.009	0.013	22.2	22.6	22.4	5.81	6.33
Ca						0.052							
Cd	0.188	0.156	0.043	0.039					0.070	0.068	0.069		
Cr	0.040	0.035	0.032	0.030									
Cu	0.013	0.011				0.019	0.011						
Fe	2.97	2.55	1.10	1.06					1.47	1.43	1.45	0.080	0.107
K													
Li	17.9	17.9	15.0	15.1	16.5	0.003	0.003	0.003	15.1	15.3	15.2	3.09	3.34
Mg													
Mn	0.072	0.062	0.026	0.038		0.252	0.068		0.036	0.036	0.036		
Mo													
Na	16.1	16.1	14.1	14.3	15.2	0.232	0.212	0.222	15.2	15.2	15.2	7.60	8.24
Nd	0.052												
Ni	0.233	0.205	0.079	0.069		0.052	0.042		0.093		0.093		
P	1.19	1.19	1.03	1.06					0.970	1.03	1.00		
Pb	0.084	0.068											
Re													
Ru													
Si	84.2	84.1	79.1	78.1	81.4	0.405	0.124	0.265	76.0	76.6	76.3	29.6	32.1
Sr													
Ti	0.010	0.007											
Tl													
U													
V													
Zn	0.021					0.027	0.024						
Zr	0.272	0.243	0.180	0.179					0.227	0.220	0.224	0.020	0.031
NO_2^-	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	N/A	NM	NM	NM	NM	NM
NO_3^-	0.5	0.5	0.5	0.5	0.5	<0.5	0.6	0.6	NM	NM	NM	NM	NM
F^-	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	N/A	NM	NM	NM	NM	NM
Cl^-	0.8	0.9	1.3	0.8	1.0	0.6	0.6	0.6	NM	NM	NM	NM	NM
SO_4^{2-}	1.8	1.7	1.4	1.4	1.6	0.8	0.6	0.7	NM	NM	NM	NM	NM
PO_4^{3-}	3.4	3.3	3.3	3.1	3.3	<0.5	<0.5	N/A	NM	NM	NM	NM	NM
Br^-	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	<0.25	N/A	NM	NM	NM	NM	NM
Initial pH	5.54	5.54	6.16	6.16		5.54	5.54		5.69	5.82		5.95	5.95
Final pH	9.60	9.61	9.62	9.59		6.36	6.70		9.73	9.65	9.69	9.37	9.37
Leach Vessel	Stainless	Stainless	Stainless	Stainless		Stainless	Stainless		Teflon	Teflon		Teflon	Teflon
SA/V (m^{-1})	>2000	>2000	2000	2000		NA	NA		2000	2000		10.0	10.0
Sample Log # (ICP)	95-002641	95-002642	95-002649	95-002650		95-002639	95-002640		94-7716	94-7725		94-7736	94-7737
Sample Log # (IC)	95-02629	95-02630	95-02637	95-02638		95-02627	95-02628						

Notes:

1. Samples #1-2 (hot cell test #5) were prepared in the laboratory. Following the hot cell test, untested glass was evaluated and found to be uncleaned.
2. Samples #3-4 (hot cell test #5) were crushed, sieved and washed in the hot cell.
3. NM = not measured. NA = not applicable; PCT = Product Consistency Test; MCC-1 = Materials Characterization Center (MCC-1) Test
4. Sample #1 (lab test #12) was crystalline and sample #2 (lab test #9) was amorphous.

Table A.16. Leach Test Information and Leachate Analysis for CVS-IS-HW39-4

	7-Day PCT (Hot Cell Test #4)					7-Day PCT (Hot Cell Test #3)					
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Sample Ave. (ug/ml)	Blank (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Blank Ave. (ug/ml)
B	105	100	99	101	<0.02	100	100	99.5	0.3	<0.2	0.3
K	2	<2	<2	2	<1	<10	<10	<10	<10	<10	NM
Li	45.0	42.0	42.0	43.0	<0.03	43.0	43.0	41.5	<0.3	<0.3	NA
Na	220	200	200	207	0.1	200	200	190	<0.8	<0.8	NA
Si	200	190	190	193	<0.08	200	200	190	<0.8	<0.8	NA
NO$_2^-$	<0.5	0.5	<0.5	0.5	<0.5	NM	NM	NM	NM	NM	NM
NO$_3^-$	<0.5	<0.5	<0.5	NM	<0.5	NM	NM	NM	NM	NM	NM
F$^-$	<2	<2	<2	NM	<0.25	NM	NM	NM	NM	NM	NM
Cl$^-$	1.1	1.3	1.0	1.1	<0.25	NM	NM	NM	NM	NM	NM
SO$_4^{2-}$	26.7	24.4	24.1	25.1	<0.5	NM	NM	NM	NM	NM	NM
PO$_4^{3-}$	7.9	7.6	7.8	7.8	<0.5	NM	NM	NM	NM	NM	NM
Br$^-$	<0.25	<0.25	0.25	0.25	<0.25	NM	NM	NM	NM	NM	NM
Initial pH	7.10	7.10	7.10	7.10	7.10	7.03	7.03	7.03	7.03	7.03	
Final pH	10.26	10.24	10.25	10.25	6.57	10.14	10.15	10.12	5.75	5.12	5.44
Leach Vessel	Stainless	Stainless	Stainless		Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	
SA/V (m^{-1})	2000	2000	2000		NA	2000	2000	2000	NA	NA	
Sample Log # (ICP)	94-03197	94-03198	94-03199		94-03194	93-9387	93-9388	93-9389	93-9390	93-9391	
Sample Log # (IC)	94-03191	94-03192	94-03193		94-03188						

	7-Day PCT (Hot Cell Test #5)						7-Day PCT (Lab Test #10)		
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Sample Ave. (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Blank (ug/ml)
B	118	114	106	113	0.016	0.009	130	110	<0.2
K	NM	NM	NM	NM	NM	NM	<10	<10	<10
Li	47.3	46.1	43.2	46	0.003	0.003	50.0	46.0	<0.3
Na	228	219	205	217	0.232	0.212	230	210	<0.8
Si	221	218	206	215	0.405	0.124	210	200	<0.8
NO$_2^-$	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	NM	NM	NM
NO$_3^-$	0.6	<0.5	<0.5	<0.53	<0.5	0.6	NM	NM	NM
F$^-$	<0.25	<0.25	<0.25	<0.25	<0.25	0.25	NM	NM	NM
Cl$^-$	1.4	1.2	1.3	1.3	0.6	0.6	NM	NM	NM
SO$_4^{2-}$	25.9	25.4	24.1	25.1	0.8	0.6	NM	NM	NM
PO$_4^{3-}$	7.7	7.7	7.3	7.6	<0.5	0.5	NM	NM	NM
Br$^-$	<0.25	<0.25	<0.25	<0.25	<0.25	0.25	NM	NM	NM
Initial pH	5.54	5.54	5.54	5.54	5.54	5.54	6.12	6.12	6.12
Final pH	10.22	10.26	10.22		6.36	9.70	10.20	10.10	<7.88
Leach Vessel	Stainless	Stainless	Stainless		Stainless	Stainless	Stainless	Stainless	Stainless
SA/V (m^{-1})	2000	2000	2000		NA	NA	2000	2000	NA
Sample Log # (ICP)	95-002643	95-002644	95-002645		95-002639	95-002640	93-10232	93-10233	93-10231
Sample Log # (IC)	95-02631	95-02632	95-02633		95-02627	95-02628			

Notes:

1. Severe matrix spike with fluorine on hot cell test #4.
2. PCT (hot cell test #3) anion samples leaked during transport and were cross contaminated with HNO3. The results are not valid and therefore not reported.
3. Boron spike recoveries for PCT (hot cell test #3) samples 1-3 were 102%, 105%, and 104%, respectively.
4. NM = not measured; NA = not applicable; PCT = Product Consistency Test.

Table A.17. Leach Test Information and Leachate Analysis for NCAW Simulant

	28 Day MCC-1 (Lab Test #1)		28 Day MCC-1 (Hot Cell Test #1)				28 Day MCC-1 (Lab Test #3)		
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Blank (ug/ml)
B	4.9	4.8	2.79	2.31	<0.01	<0.1	3.7	4.3	<0.1
K	NM	NM	0.39	1.70	0.31	2.30	<0.8	<0.8	<0.8
Li	2.7	2.7	1.51	1.30	<0.0006	<0.01	1.9	2.2	<0.01
Na	10.3	10.1	6.29	6.75	0.39	1.60	7.8	9.0	<0.07
Si	32.5	31.9	31.0	23.4	4.17	6.47	41.0	45.0	7.80
NO_2^-	NM	NM	<4	NM	<4	NM	NM	NM	NM
NO_3^-	NM	NM	<4	NM	<4	NM	NM	NM	NM
F^-	NM	NM	<2	NM	<2	NM	NM	NM	NM
Cl^-	NM	NM	<2	NM	<2	NM	NM	NM	NM
SO_4^{2-}	NM	NM	5.7	NM	6.6	NM	NM	NM	NM
PO_4^{3-}	NM	NM	<4	NM	<4	NM	NM	NM	NM
Br^-	NM	NM	<2	NM	<2	NM	NM	NM	NM
Initial pH	6.41	6.41	7.12	7.12	7.12	7.12	5.84	5.84	5.84
Final pH	9.29	9.42	8.73	7.55	5.70	6.51	9.25	9.35	
Leach Vessel	Teflon	Teflon	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica
SA/V (m^{-1})	10.0	10.0	10.0	10.0	NA	NA	10.0	10.0	NA
Sample Log # (ICP)	90-5441-782/1851	90-5441-782/1842	91-0204	91-2789	91-0198	91-2785	91-09707	91-09708	91-09709
Sample Log # (IC)			90-9542		90-9536				

	7-Day PCT (Lab Test #2)		7-Day PCT (Lab Test #5)					7-Day PCT (Lab Test #4)			7-Day PCT (Hot Cell Test #2)		
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Sample 3 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Blank (ug/ml)	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Blank (ug/ml)
B	130	130	110	120	120	0.06	0.05	120	150	0.16	91.8	98.8	0.02
K	2.2	1.6	0.9	1.0	<0.8	<0.8	<0.8	0.8	0.9	1.00	0.5	<0.5	<0.5
Li	54.4	57.2	47.0	49.0	49.0	<0.01	<0.01	44.0	48.0	0.02	41.9	44.9	<0.015
Na	230	230	170	170	170	0.20	0.20	170	180	0.10	145	155	<0.25
Si	250	240	210	210	210	0.20	0.30	210	250	2.00	215	226	0.14
NO_2^-	NM	NM	<2	<2	<2	<2	<2	NM	NM	NM	NM	NM	NM
NO_3^-	NM	NM	<2	<2	<2	<2	<2	NM	NM	NM	NM	NM	NM
F^-	NM	NM	2.5	2.4	2.6	<1	<1	NM	NM	NM	NM	NM	NM
Cl^-	NM	NM	3.5	3.1	2.2	<1	<1	NM	NM	NM	NM	NM	NM
SO_4^{2-}	NM	NM	6.3	5.1	5.1	<1	<1	NM	NM	NM	NM	NM	NM
PO_4^{3-}	NM	NM	5.5	5.1	5.3	<2	<2	NM	NM	NM	NM	NM	NM
Br^-	NM	NM	<1	<1	<1	<2	<2	NM	NM	NM	NM	NM	NM
Initial pH	6.50	6.50	5.80	5.80	5.80	5.80	5.80	5.84	5.84	5.84			
Final pH	10.09	10.11	10.09	10.07	10.09	5.90	5.57	10.05	10.08		9.83	9.86	7.35
Leach Vessel	Teflon	Teflon	Stainless	Stainless	Stainless	Stainless	Stainless	Fused-Silica	Fused-Silica	Fused-Silica	Stainless	Stainless	Stainless
SA/V (m^{-1})	2000	2000	2000	2000	2000	NA	NA	2000	2000	NA	2000	2000	NA
Sample Log # (ICP)	90-5441-782/1828	90-5441-782/1884	91-9710	91-9711	91-9712	91-9713	91-9714	91-8598	91-8599	91-8600	92-7431	92-7432	92-7430
Sample Log # (IC)			91-9206	91-9207	91-9208	91-9259	91-9260						

Notes: 1. NM = not measured. NA = not applicable; MCC-1 = Materials Characterization Center (MCC-1) Test.

Table A.18. Leach Test Information and Leachate Analysis for ATM-10 and Environmental Assessment Glass

	28-Day MCC-1 on ATM-10 (Hot Cell Test #1)				7-Day PCT on EA Glass (Lab Test #10)	
	Sample 1 (ug/ml)	Sample 2 (ug/ml)	Blank 1 (ug/ml)	Blank 2 (ug/ml)	Sample 1 (ug/ml)	Blank 1 (ug/ml)
B	2.97	2.61	<0.01	<0.1	404	<0.02
K	3.27	3.42	0.31	2.30	<0.2	0.4
Li	1.35	1.22	<0.0006	<0.01	126	0.008
Na	8.35	7.96	0.39	1.60	1400	0.62
Si	26.9	28.3	4.17	6.47	577	0.14
NO_2^-	<4	NM	<4	NM	NM	NM
NO_3^-	<4	NM	<4	NM	NM	NM
F^-	<2	NM	<2	NM	NM	NM
Cl^-	<2	NM	<2	NM	NM	NM
SO_4^{2-}	<4	NM	6.6	NM	NM	NM
PO_4^{3-}	<4	NM	<4	NM	NM	NM
Br^-	<2	NM	<2	NM	NM	NM
Initial pH	7.12	7.12	7.12	7.12	6.12	6.12
Final pH	8.91	8.75	5.70	6.51	11.63	<7.88
Leach Vessel	Fused-Silica	Fused-Silica	Fused-Silica	Fused-Silica	Stainless	Stainless
SA/V (m^{-1})	10.0	10.0	NA	NA	2000	NA
Sample Log # (ICP)	91-0203	91-2788	91-0198	91-0279	93-10312	93-10309
Sample Log # (IC)	90-9541		90-9536			

Notes 1. NM = not measured, NA = not applicable; MCC-1 = Materials Characterization Center (MCC-1) Test, PCT = Product Consistency Test.

Table A.19. Analytical Laboratory-to-Analytical Laboratory Comparison 7-Day PCT (Lab Test #10)

	101-AZ-C2 Simulant						CVS-IS-HW39-4					
	Sample 1 Lab 1 (ug/ml)	Sample 1 Lab 2 (ug/ml)	Sample 2 Lab 1 (ug/ml)	Sample 2 Lab 2 (ug/ml)	Sample Ave. Lab 1 (ug/ml)	Sample Ave Lab 2 (ug/ml)	Sample 1 Lab 1 (ug/ml)	Sample 1 Lab 2 (ug/ml)	Sample 2 Lab 1 (ug/ml)	Sample 2 Lab 2 (ug/ml)	Sample Ave Lab 1 (ug/ml)	Sample Ave Lab 2 (ug/ml)
B	16.0	20.0	18.0	24.0	17.0	22.0	130	116	110	104	120.0	110.0
Li	21.0	20.5	21.5	19.6	21.2	20.1	50.0	47.1	46.0	41.4	48.0	44.3
Na	23.0	26.0	26.0	26.2	24.5	26.1	230	223	210	194	220.0	208.5
Si	90.0	90.0	94.0	89.0	92.0	89.5	210	197	200	186	205.0	191.5
Sample Log # (ICP)	93-10234	93-10307	93-10235	93-10311			93-10232	93-10308	93-10233	93-10310		

Notes:

1. Lab 1 = Jerry Wagner 325 Building; Lab 2 = Karl Pool 314 Building
2. PCT = Product Consistency Test.

Table A.20. CVS Model Predictions and 95% Prediction Intervals for 101-AZ Core #1 Composition

	Model Prediction ln(g/m^2)	Standard Error ln(g/m^2)	Lower 95% Prediction ln(g/m^2)	Upper 95% Prediction ln(g/m^2)	Model Prediction (g/m^2)	Lower 95% Prediction (g/m^2)	Upper 95% Prediction (g/m^2)
PCT 1st Order							
B	-0.41261	0.69719	-1.79386	0.96864	0.66192	0.16632	2.63436
Li	-0.46701	0.61035	-1.67610	0.74209	0.62687	0.18710	2.10032
Na	-0.74496	0.60262	-1.93886	0.44894	0.47475	0.14387	1.56665
PCT 2nd Order #1							
B	-0.69248	0.59657	-1.87451	0.48954	0.50033	0.15343	1.63157
Li	-0.78643	0.47423	-1.72634	0.15348	0.45547	0.17793	1.16588
Na	-1.05625	0.49613	-2.03937	-0.07313	0.34776	0.13011	0.92948
PCT 2nd Order #2							
B	-0.75755	0.52064	-1.78966	0.27457	0.46881	0.16702	1.31596
Li	-0.66681	0.44084	-1.54082	0.20719	0.51334	0.21421	1.23022
Na	-0.99603	0.44897	-1.88606	-0.10600	0.36934	0.15167	0.89942
PCT 2nd Order #3							
B	-0.73378	0.49992	-1.72503	0.25747	0.48009	0.17817	1.29365
Li	-0.69157	0.43116	-1.54648	0.16334	0.50079	0.21300	1.17744
Na	-1.02482	0.43916	-1.89551	-0.15414	0.35886	0.15024	0.85715
MCC-1 1st Order							
B	2.76819	0.42338	1.92861	3.60778	15.92978	6.87994	36.88408
Li	2.70651	0.41443	1.88468	3.52833	14.97691	6.58425	34.06703
Na	2.74538	0.41042	1.93151	3.55925	15.57053	6.89992	35.13683
MCC-1 2nd Order #1							
B	2.61419	0.33695	1.94578	3.28260	13.65615	6.99909	26.64496
Li	2.56718	0.34015	1.89241	3.24194	13.02903	6.63534	25.58331
Na	2.58999	0.32297	1.94930	3.23067	13.32964	7.02377	25.29660
MCC-1 2nd Order #2							
B	2.61572	0.32381	1.97321	3.25822	13.67706	7.19373	26.00321
Li	2.56895	0.32656	1.92099	3.21691	13.05211	6.82771	24.95090
Na	2.58593	0.31074	1.96936	3.20251	13.27563	7.16609	24.59418
MCC-1 2nd Order #3							
B	2.51137	0.33862	1.83947	3.18327	12.32180	6.29320	24.12552
Li	2.57485	0.30512	1.96894	3.18076	13.12935	7.16308	24.06504
Na	2.54650	0.29573	1.95949	3.13352	12.76236	7.09571	22.95464

Table A.21. CVS Model Predictions and 95% Prediction Intervals for 101-AZ Core #2 Compostion

	Model Predictions ln(g/m²)	Standard Error ln(g/m²)	Lower 95% Prediction ln(g/m²)	Upper 95% Prediction ln(g/m²)	Model Prediction (g/m²)	Lower 95% Prediction (g/m²)	Upper 95% Prediction (g/m²)
PCT 1st Order							
B	-0.56044	0.70701	-1.96115	0.84028	0.57096	0.14070	2.31702
Li	-0.61607	0.61795	-1.84022	0.60807	0.54006	0.15878	1.83688
Na	-1.08036	0.61111	-2.29108	0.13036	0.33947	0.10116	1.13924
PCT 2nd Order #1							
B	-0.59743	0.60345	-1.79310	0.59824	0.55022	0.16644	1.81891
Li	-0.70971	0.48040	-1.66184	0.24243	0.49179	0.18979	1.27434
Na	-1.05648	0.50163	-2.05050	-0.06247	0.34768	0.12867	0.93944
PCT 2nd Order #2							
B	-0.75572	0.52884	-1.80408	0.29263	0.46967	0.16463	1.33995
Li	-0.85230	0.44605	-1.73663	0.03203	0.42643	0.17611	1.03255
Na	-1.20633	0.45534	-2.10898	-0.30367	0.29929	0.12136	0.73810
PCT 2nd Order #3							
B	-0.82913	0.50848	-1.83736	0.17910	0.43643	0.15924	1.19614
Li	-0.86476	0.43449	-1.72627	-0.00326	0.42115	0.17795	0.99675
Na	-1.22129	0.44585	-2.10523	-0.33735	0.29485	0.12182	0.71366
MCC-1 1st Order							
B	2.73420	0.43166	1.87822	3.59019	15.39742	6.54185	36.24096
Li	2.66098	0.42253	1.82309	3.49886	14.31031	6.19096	33.07772
Na	2.73324	0.41844	1.90347	3.56301	15.38265	6.70913	35.26920
MCC-1 2nd Order #1							
B	2.61199	0.34254	1.93248	3.29150	13.62614	6.90662	26.88316
Li	2.54912	0.34580	1.86315	3.23508	12.79584	6.44400	25.40840
Na	2.61122	0.32833	1.95990	3.26254	13.61565	7.09862	26.11579
MCC-1 2nd Order #2							
B	2.59770	0.32956	1.94378	3.25163	13.43281	6.98510	25.83241
Li	2.53334	0.33236	1.87386	3.19283	12.59550	6.51339	24.35726
Na	2.58201	0.31664	1.95373	3.21030	13.22369	7.05495	24.78652
MCC-1 2nd Order #3							
B	2.49661	0.34491	1.81223	3.18099	12.14127	6.12409	24.07057
Li	2.58652	0.31666	1.95769	3.21535	13.28346	7.08295	24.91201
Na	2.71595	0.30576	2.10902	3.32288	15.11897	8.24016	27.74013

Table A.22. CVS Model Predictions and 95% Prediction Intervals for 102-AZ Core #1 Composition

	Model Predictions ln(g/m^2)	Standard Error ln(g/m^2)	Lower 95% Prediction ln(g/m^2)	Upper 95% Prediction ln(g/m^2)	Model Prediction (g/m^2)	Lower 95% Prediction (g/m^2)	Upper 95% Prediction (g/m^2)
PCT 1st Order							
B	-0.38941	0.70143	-1.77906	1.00024	0.67746	0.16880	2.71893
Li	-0.45601	0.61481	-1.67393	0.76192	0.63381	0.18751	2.14239
Na	-0.92368	0.60628	-2.12484	0.27748	0.39706	0.11945	1.31980
PCT 2nd Order #1							
B	-0.78024	0.60161	-1.97226	0.41178	0.45830	0.13914	1.50950
Li	-0.77381	0.47848	-1.72214	0.17452	0.46125	0.17868	1.19067
Na	-1.32993	0.50035	-2.32139	-0.33846	0.26450	0.09814	0.71287
PCT 2nd Order #2							
B	-0.81406	0.52534	-1.85548	0.22736	0.44306	0.15638	1.25528
Li	-0.62368	0.44816	-1.51220	0.26484	0.53597	0.22042	1.30322
Na	-1.23220	0.45271	-2.12965	-0.33475	0.29165	0.11888	0.71552
PCT 2nd Order #3							
B	-0.76267	0.50466	-1.76332	0.23799	0.46642	0.17147	1.26870
Li	-0.64025	0.43739	-1.50751	0.22701	0.52716	0.22146	1.25484
Na	-1.22517	0.44311	-2.10368	-0.34665	0.29371	0.12201	0.70705
MCC-1 1st Order							
B	2.89737	0.42643	2.05173	3.74300	18.12641	7.78135	42.22447
Li	2.82077	0.41741	1.99302	3.64852	16.78977	7.33766	38.41777
Na	2.89913	0.41337	2.07940	3.71886	18.15834	7.99967	41.21738
MCC-1 2nd Order #1							
B	2.72743	0.34074	2.05149	3.40336	15.29353	7.77948	30.06495
Li	2.66855	0.34398	1.98618	3.35091	14.41905	7.28764	28.52868
Na	2.72615	0.32660	2.07826	3.37405	15.27397	7.99055	29.19653
MCC-1 2nd Order #2							
B	2.74271	0.32805	2.09178	3.39364	15.52901	8.09932	29.77413
Li	2.68259	0.33084	2.02613	3.33905	14.62292	7.58468	28.19233
Na	2.71184	0.31546	2.08590	3.33777	15.05695	8.05183	28.15627
MCC-1 2nd Order #3							
B	2.53951	0.34471	1.85552	3.22350	12.67346	6.39502	25.11587
Li	2.57043	0.30878	1.95727	3.18360	13.07144	7.07997	24.13348
Na	2.56268	0.30102	1.96515	3.16020	12.97053	7.13598	23.57531

Table A.23. CVS Model Predictions and 95% Prediction Intervals for CVS-IS-HW39-4 Composition

	Model Predictions ln(g/m²)	Standard Error ln(g/m²)	Lower 95% Prediction ln(g/m²)	Upper 95% Prediction ln(g/m²)	Model Prediction (g/m²)	Lower 95% Prediction (g/m²)	Upper 95% Prediction (g/m²)
PCT 1st Order							
B	0.56924	0.69382	-0.80536	1.94383	1.76692	0.44693	6.98545
Li	0.40771	0.60783	-0.79639	1.61181	1.50337	0.45095	5.01187
Na	0.23470	0.59971	-0.95345	1.42284	1.26453	0.38541	4.14889
PCT 2nd Order #1							
B	0.62553	0.59224	-0.54791	1.79898	1.86924	0.57816	6.04348
Li	0.19795	0.47310	-0.73972	1.13562	1.21890	0.47725	3.11310
Na	0.11210	0.49432	-0.86744	1.09163	1.11862	0.42003	2.97913
PCT 2nd Order #2							
B	0.41449	0.51895	-0.61428	1.44325	1.51360	0.54103	4.23444
Li	0.21121	0.43805	-0.65726	1.07969	1.23517	0.51827	2.94377
Na	0.06974	0.44720	-0.81679	0.95627	1.07223	0.44185	2.60197
PCT 2nd Order #3							
B	0.40374	0.49822	-0.58413	1.39161	1.49741	0.55759	4.02132
Li	0.20228	0.42810	-0.64656	1.05112	1.22419	0.52384	2.86085
Na	0.00318	0.43778	-0.86477	0.87113	1.00319	0.42115	2.38961
MCC-1 1st Order							
B	3.01662	0.42106	2.18164	3.85160	20.42215	8.86083	47.06831
Li	2.92352	0.41216	2.10620	3.74084	18.60667	8.21696	42.13337
Na	2.97474	0.40817	2.16533	3.78414	19.58453	8.71748	43.99782
MCC-1 2nd Order #1							
B	3.06113	0.33379	2.39898	3.72328	21.35167	11.01194	41.39996
Li	2.96345	0.33697	2.29500	3.63190	19.36466	9.92444	37.78454
Na	3.01998	0.31995	2.38529	3.65466	20.49088	10.86221	38.65438
MCC-1 2nd Order #2							
B	3.03962	0.32086	2.40297	3.67628	20.89730	11.05596	39.49918
Li	2.94187	0.32359	2.29981	3.58394	18.95125	9.97229	36.01516
Na	3.02058	0.30800	2.40944	3.63172	20.50318	11.12773	37.77774
MCC-1 2nd Order #3							
B	2.93261	0.33739	2.26315	3.60208	18.77657	9.61332	36.67444
Li	2.88724	0.30177	2.28798	3.48649	17.94372	9.85501	32.67107
Na	2.91581	0.29401	2.33220	3.49941	18.46376	10.30058	33.09592

Table A.24. Frit and Recycle Compositions

Frit Compositions (wt% oxides)

	101-AZ Core #1 and NCAW Simulant	101-AZ Core #2	102-AZ Core #1 and 102-AZ Core #1 Simulant
B_2O_3	14.0	10.6	20.45
CaO	1.0	0.0	0.0
Li_2O	5.0	8.6	7.29
MgO	1.0	0.0	0.0
Na_2O	9.0	5.2	0.0
SiO_2	70.0	75.6	72.26

Composition of Simulant Recycle Added to 102-AZ Core #1 Simulant

Oxide	g Recycle Oxide per L of Recycle Simulant
CdO	2.01
MnO_2	0.60
Na_2O	25.17
P_2O_5	2.01
NO_3^-	40.27
Cl^-	0.04
TOC	1.36
Diatomaceous Earth	20.13
Zeolite (IE-96)	10.07
Sum of Nonvolatiles	60

APPENDIX B

Radiochemical Data

Table B.1. Radiochemical Data for 101-AZ Core #1

Analyte	Washed Solids (uCi/g)	Melter Feed (uCi/g)	Melter Feed Sup. (uCi/g)	Glass (uCi/g)	Glass Uncertainty (+/-)
H-3	<4.5E-04	1.55E-03			
C-14	4.70E-04				
GEA					
Cr-51			3.82E+00		
Fe-59			1.78E-01		
Co-60	1.20E+01	3.19E+00	1.59E-01	6.16E+00	5.8%
Se-79	< 4.0E-03	< 3.57E-04			
Nb-95			1.43E-01		
Zr-95			2.92E-01		
Ru-103			5.65E-01		
Ru-106	3.50E+02	4.03E+02	2.52E+00	2.79E+02	1.5%
Sn-113			7.55E-01		
Sb-125	5.30E+01	7.29E+01	1.94E+00	9.93E+01	2.2%
Cs-134	3.90E+00	8.91E+00	5.86E+00	4.44E+00	9.1%
Cs-137	2.40E+02	4.41E+02	4.52E+02	6.00E+02	0.5%
Ce-144	5.30E+02	6.55E+02	2.08E+00	3.50E+02	1.5%
Eu-152			3.08E-01		
Eu-154	1.60E+01	2.82E+01	1.71E-01	5.05E+01	1.9%
Eu-155		6.72E+01	9.54E-01	1.06E+02	1.6%
Am-241				5.24E+01	4.8%
Sr-90	6.10E+03	6.86E+03		1.96E+04	6.5%
Tc-99	6.60E-02	<1.02E-01		3.15E-03	70%
I-129	1.80E-04	<6.14E-05		< 5.00E-04	
Np-237	6.70E-03			4.50E-02	35%
Pu-238	1.20E-01			4.49E-02	12%
Pu-239+240	7.90E-01			2.40E-01	9%
Total Pu				2.85E-01	9%
Am-241	1.20E+01	2.92E+01		3.62E+01	7%
Cm-242	1.70E-03			6.41E-02	10%
Cm-243+244	8.50E-03			4.35E-01	27%
Total U (ug/g)				3.48E+03	5%
Total Beta		2.03E+04		4.31E+04	3.2%
Date of Analysis		6/20/90 - 4/6/91	6/19/90	7/2/91	

Note: Error estimates are one-sigma total analytical errors, not including contributions from sampling or experimental error. Calibration errors are estimated at an additional 2%.

Table B.2. Radiochemical Data for 101-AZ Core #2

Analyte	Washed Solids (uCi/g)	Melter Feed (uCi/g)	Melter Feed Sup. (uCi/g)	Glass (uCi/g)	Glass Uncertainty (+/-)
H-3	< 6.80E-04	1.43E-02			
C-14	3.34E-04				
GEA					
Cr-51					
Fe-59					
Co-60	1.38E+01	2.13E+01		1.62E+01	3.4%
Se-79	< 3.90E-04	< 3.38E-02			
Nb-95					
Zr-95					
Ru-103					
Ru-106	1.38E+03	1.99E+03	3.65E+00	6.67E+02	0.9%
Sn-113					
Sb-125	1.68E+02	3.23E+02		2.57E+02	1.0%
Cs-134			1.14E+00		
Cs-137	2.31E+02	5.14E+02	7.66E+01	2.72E+02	0.8%
Ce-144	2.81E+03	4.17E+03		1.09E+03	0.7%
Eu-152					
Eu-154	9.70E+01	1.45E+02		1.23E+02	1.1%
Eu-155	2.43E+02	3.65E+02		2.80E+02	0.8%
Am-241				1.37E+02	1.9%
Sr-90	1.96E+04	2.99E+04		2.92E+04	6.5%
Tc-99	<4.60E+00	<6.82E-01		2.06E-02	18%
I-129	1.03E-02	<3.45E-03		<4.41E-04	
Np-237	3.45E-02			5.73E-01	9%
Pu-238				8.09E-02	10%
Pu-239,240				4.36E-01	8%
Total Pu	3.86E+01			5.18E-01	8%
Am-241	6.71E+01	1.45E+02		1.06E+02	7%
Cm-242				2.55E-01	8%
Cm-243 + 244	4.61E-01			1.13E+00	15%
Total U (ug/g)	4.50E+03			3.14E+04	5.0%
Total Alpha	7.97E+01				
Total Beta	5.34E+04	6.77E+04		6.81E+04	3.2%
Date of Analysis		1/9/91 - 4/6/91	1/1/91	7/2/91	

Note: Error estimates are one-sigma total analytical errors, not including contributions from sampling or experimental error. Calibration errors are estimated at an additional 2%.

Table B.3. Radiochemical Data for 102-AZ Core #1

Analyte	Washed Solids (uCi/g)	Formated Feed (uCi/g)	Glass (uCi/g)	Glass Uncertainty (+/-)
H-3	6.80E-04			
C-14	2.10E-04			
GEA				
Cr-51				
Fe-59				
Co-60	4.60E+00		5.83E+00	3%
Se-79	<4.20e-03			
Nb-95				
Zr-95				
Ru-103				
Ru-106	9.89E+02		1.06E+02	5%
Sn-113				
Sb-125	6.18E+01		8.64E+01	3%
Cs-134				
Cs-137	1.46E+02		2.00E+02	4%
Ce-144	5.48E+03			
Eu-152				
Eu-154	2.49E+01		3.64E+01	2%
Eu-155	9.17E+01		1.03E+02	3%
Am-241			5.47E+01	3%
Sr-90	5.53E+03		1.27E+04	8%
Tc-99	1.25E-01		6.73E-03	31%
I-129	< 3.00E-05			
Np-237	5.16E-03			
Pu-238	8.66E-03		1.93E-01	13%
Pu-239+240	1.83E+01	TBD	1.73E+00	10%
Total Pu	1.84E+01			
Am-241	2.50E+01		5.56E+01	8%
Cm-242	1.40E-02			
Cm-243+244	9.92E-02		2.22E-01	28%
Total U	2.95E+03		1.49E+03	6%
Total Alpha			5.95E+01	4%
Total Beta			2.44E+04	4%
Date of Analysis			4/21/94	

Note: Error estimates are one-sigma total analytical errors, not including contributions from sampling or experimental error. Calibration errors are estimated at an additional 2%.

Table B.4. Radiochemical Leachate Analysis of 101-AZ Core #1

		MCC-1 (Hot Cell Test #1)		7-Day PCT (Hot Cell Test #4)									
		Sample 3	Blank 1	Sample 1	Sample 2	Sample 3	Blank 1	Blank 2	AS Sample 1	AS Sample 2	AS Sample 3	AS Blank 1	AS Blank 2
Uranium	(mg/ml)	4.20e-5	NM	2.83e-4	2.83e-4	2.54e-4	<2.0e-6	<2.0e-6	4.29e-5	3.99e-5	6.74e-5	2.91e-6	2.91e-6
Total Beta	(uCi/ml)	1.28	1.48e-3	3.34e-1	3.90e-1	4.13e-1	1.59e-4	2.32e-4	2.13e-1	2.04e-1	4.93e-1	2.47e-3	9.02e-4
Tc-99	(uCi/ml)	<5.86e-5	NM	4.28e-6	6.19e-6	6.08e-6	<3.0e-6	<3.0e-6	<4.0e-7	<4.0e-7	<4.0e-7	<4.0e-7	<4.0e-7
Sr-90	(uCi/ml)	6.60e-1	NM	1.30e-1	1.61e-1	1.63e-1	1.08e-5	2.33e-5	9.88e-2	8.57e-2	2.09e-1	1.28e-3	3.23e-4
Total Alpha	(uCi/ml)	2.74e-5	NM	2.82e-4	3.66e-4	3.94e-4	<8.0e-7	<1.0e-6	2.77e-4	3.20e-4	6.83e-4	3.68e-6	3.49e-6
Pu-238	(uCi/ml)	1.02e-5	NM	4.59e-6	5.81e-6	4.75e-6	ANM	ANM	7.61e-6	8.79e-6	1.22e-5	ANM	ANM
Pu-239/240	(uCi/ml)	1.53e-5	NM	2.61e-5	2.87e-5	2.81e-5	ANM	ANM	4.15e-5	4.07e-5	5.99e-5	ANM	ANM
Cm-243/244	(uCi/ml)	1.31e-6	NM	2.74e-6	5.57e-6	4.47e-6	ANM	ANM	7.37e-6	4.64e-6	1.13e-5	ANM	ANM
Am-241	(uCi/ml)	5.45e-6	NM	2.38e-4	3.26e-4	3.31e-4	ANM	ANM	2.29e-4	2.78e-4	5.97e-4	ANM	ANM
Np-237	(uCi/ml)	1.06e-6	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM
K-40	(uCi/ml)	<DL	<DL	<DL	1.30e-4	8.37e-5	<DL	<DL	<DL	<DL	<DL	<1.4E-5	<DL
Co-60	(uCi/ml)	2.99e-5	<DL	6.00e-5	7.25e-5	7.33e-5	<DL	<DL	6.57e-5	6.61e-5	9.78e-5	<DL	<DL
Ru-106	(uCi/ml)	<DL	<DL	<DL	<DL	<DL	<DL	<DL	1.20e-4	<DL	<DL	<DL	<DL
Sb-125	(uCi/ml)	5.36e-3	<DL	1.07e-2	9.85e-3	9.19e-3	<DL	<DL	9.81e-5	1.39e-4	3.55e-4	<DL	<DL
Cs-134	(uCi/ml)	3.66e-4	1.96e-6	4.38e-4	3.82e-4	3.27e-4	<DL	2.15e-5	1.48e-5	1.41e-5	2.83e-5	<DL	<DL
Cs-137	(uCi/ml)	2.96e-2	7.05e-5	5.06e-2	4.95e-2	4.54e-2	1.46e-4	3.57e-4	2.26e-3	2.44e-3	6.48e-3	9.54e-5	2.04e-4
Ce-144	(uCi/ml)	<DL	<DL	<DL	<DL	<DL	<DL	<DL	1.73e-4	2.01e-4	4.79e-4	<DL	<DL
Eu-154	(uCi/ml)	<DL	5.95e-7	2.53e-4	3.41e-4	3.73e-4	<DL	<DL	2.90e-4	3.33e-4	6.31e-4	<DL	<DL
Eu-155	(uCi/ml)	<DL	<DL	4.53e-4	6.12e-4	6.21e-4	<DL	<DL	5.34e-4	6.12e-4	1.22e-3	<DL	<DL
Am-241	(uCi/ml)	<DL	6.78e-7	2.16e-4	4.42e-4	3.32e-4	<DL	<DL	2.32e-4	2.88e-4	6.00e-4	<DL	<DL
	Leach Vessel	Fused Silica	Fused Silica	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless
	SA/V (m^{-1})	10.0	10.0	2000	2000	2000	NA	NA	2000	2000	2000	NA	NA
	Analysis/Date	1/91 (for GEA) 4/91 (for other)	1/91	4/94	4/94	4/94	4/94	4/94	4/94	4/94	4/94	4/94	4/94

Notes

1. ANM = analysis not made due to low alpha activity; NM = not measured. NA = not applicable; AS = Acid Strip; PCT = Product Consistency Test; MCC-1 = Materials Characterization Center (MCC-1) Test
2. Total Beta: (as SrY-90)
3. K-40 through Am-241: by gamma energy analysis (GEA)

Table B.5. Radiochemical Leachate Analysis of 101-AZ Core #2

		MCC-1 (Hot Cell Test #1)		7-Day PCT (Hot Cell Test #3)							
		Sample	Blank	Sample 1	Sample 2	Sample 3	Blank 1	Blank 2	AS Sample 1	AS Sample 2	AS Sample 3
Uranium	(mg/ml)	1.00e-4	NM	9.25e-4	2.34e-3	2.35e-4	9.93e-6	6.09e-5	6.80e-5	8.61e-3	1.15e-4
Total Beta	(uCi/ml)	0.52	1.48e-3	1.83e-1	1.88e-1	4.01e-2	5.63e-3	9.34e-3	3.85e-1	7.96e1	5.40e-1
Tc-99	(uCi/ml)	<1.58e-5	NM	<2e-6	6.20e-6	<4e-6	<1e-6	<1e-6	<3e-6	1.56e-4	<9e-7
Sr-90	(uCi/ml)	2.41e-1	NM	4.93e-2	5.37e-2	1.15e-2	1.07e-3	2.33e-3	1.88e-1	3.78e1	2.47e-1
Total Alpha	(uCi/ml)	4.34e-5	NM	4.52e-4	1.25e-3	5.75e-4	7.27e-5	3.63e-4	2.00e-4	1.57e-1	4.29e-4
Pu-238	(uCi/ml)	4.46e-6	NM	4.89e-6	1.83e-4	1.06e-4	1.12e-5	9.23e-5	2.16e-5	8.71e-4	5.86e-5
Pu-239/240	(uCi/ml)	3.40e-5	NM	3.26e-5	9.70e-5	3.93e-5	3.77e-6	2.80e-5	8.35e-5	4.23e-3	8.86e-5
Cm-243/244	(uCi/ml)	9.01e-8	NM	2.55e-4	5.97e-6	3.46e-4	3.11e-5	1.42e-4	1.54e-5	1.38e-3	6.70e-5
Am-241	(uCi/ml)	6.35e-6	NM	3.73e-4	5.81e-4	2.78e-4	1.29e-5	8.14e-5	6.70e-5	1.63e-1	1.67e-4
Np-237	(uCi/ml)	3.02e-6	NM	NM	NM	NM	NM	NM	NM	NM	NM
<u>By GEA Analysis:</u>											
K-40	(uCi/ml)	<DL	<DL	<DL	<5.40e-5	<DL	<DL	<DL	<DL	<DL	<DL
Co-60	(uCi/ml)	<DL	<DL	1.00e-4	4.73e-4	2.70e-4	5.68e-5	1.24e-4	1.25e-4	1.78e-2	4.18e-4
Ru-106	(uCi/ml)	<DL	<DL	<DL	<DL	<DL	<DL	<DL	3.44e-4	<DL	4.42e-4
Sb-125	(uCi/ml)	2.09e-2	<DL	5.43e-2	5.69e-2	7.99e-3	7.46e-5	<DL	<DL	2.11e-2	1.82e-4
Cs-134	(uCi/ml)	2.64e-4	1.96e-6	<DL	2.77e-4	1.21e-4	1.06e-4	6.67e-5	1.69e-5	3.11e-3	7.15e-5
Cs-137	(uCi/ml)	1.95e-2	7.05e-5	1.70e-2	2.67e-2	9.73e-3	3.30e-3	4.38e-3	1.20e-3	3.66e-1	4.40e-3
Ce-144	(uCi/ml)	<DL	<DL	<DL	4.40e-4	<DL	<DL	<DL	1.27e-4	2.43e-1	2.53e-4
Eu-154	(uCi/ml)	<DL	5.95e-7	3.83e-4	4.86e-4	1.46e-4	1.01e-5	7.21e-5	1.32e-4	1.45e-1	2.51e-4
Eu-155	(uCi/ml)	<DL	<DL	1.31e-3	7.71e-4	1.29e-5	<DL	2.50e-5	2.31e-4	2.97e-1	4.15e-4
Am-241	(uCi/ml)	<DL	6.78e-7	4.29e-4	5.58e-4	1.85e-4	1.07e-5	8.48e-5	8.75e-5	1.65e-1	2.34e-4
	Leach Vessel	Fused Silica	Fused Silica	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless
	SA/V (m^{-1})	10.0	10.0	2000	2000	2000	NA	NA	2000	2000	2000
	Analysis/Date	1/91 (for GEA) 4/91 (for other)	1/91	9/93	9/93	9/93	9/93	9/93	9/93	9/93	9/93

Notes:

1. NM = not measured; AS = Acid Strip; NA = not applicable; PCT = Product Consistency Test; MCC-1 = Materials Characterization Center (MCC-1) Test.
2. Total Beta: (as SrY-90)
3. K-40 through Am-241: by gamma energy analysis (GEA)

Table B.6. Radiochemical Leachate Analysis of 102-AZ Core #1

		7-Day PCT (Hot Cell Test #5)									
		Sample 1	Sample 2	Sample 3	Blank 1	Blank 2	AS Sample 1	AS Sample 2	AS Sample 3	AS Blank 1	AS Blank 2
Uranium	(mg/ml)	2.62e-4	2.62e-4	2.37e-4	<4.0e-6	6.70e-6	4.01e-5	2.59e-5	2.45e-5	<4.5e-6	<4.2e-6
Total Beta	(uCi/ml)	3.27e-1	2.81e-1	3.03e-1	1.94e-4	2.53e-4	1.53e-1	7.21e-2	7.07e-2	4.91e-4	3.88e-3
Tc-99	(uCi/ml)	3.50e-6	2.62e-6	2.92e-6	NM	NM	<1.5e-7	2.30e-7	<2.2e-7	NM	NM
Sr-90	(uCi/ml)	9.90e-2	8.69e-2	9.04e-2	<7.5e-5	4.45e-5	5.84e-2	2.80e-2	3.16e-2	7.50e-5	2.37e-3
Total Alpha	(uCi/ml)	1.96e-3	1.70e-3	1.77e-3	3.06e-6	1.25e-5	3.86e-4	1.23e-4	1.12e-4	7.60e-6	1.22e-5
Pu-238	(uCi/ml)	1.15e-5	1.13e-5	1.16e-5	ANM	ANM	9.60e-6	9.11e-6	5.25e-6	ANM	ANM
Pu-239/240	(uCi/ml)	9.30e-5	9.16e-5	1.01e-4	ANM	ANM	8.47e-5	4.00e-5	4.48e-5	ANM	ANM
Cm-243/244	(uCi/ml)	NM	NM	NM	ANM	ANM	NM	NM	NM	NM	NM
Am-241	(uCi/ml)	NM	NM	NM	ANM	ANM	NM	NM	NM	NM	NM
Np-237	(uCi/ml)	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM
<u>By GEA Analysis:</u>											
K-40	(uCi/ml)	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM
Co-60	(uCi/ml)	1.66e-4	1.78e-4	1.50e-4	8.11e-6	7.16e-6	7.12e-5	5.35e-5	6.34e-5	5.68e-5	2.10e-6
Ru-106	(uCi/ml)	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM
Sb-125	(uCi/ml)	2.43e-2	2.37e-2	2.48e-2	NM	NM	1.34e-4	NM	NM	NM	7.69e-6
Cs-134	(uCi/ml)	3.75e-5	4.49e-5	7.05e-5	NM	NM	2.32e-5	NM	NM	NM	5.52e-7
Cs-137	(uCi/ml)	1.04e-2	8.40e-3	8.91e-3	1.09e-4	9.36e-5	1.61e-3	3.06e-4	1.58e-4	1.78e-3	8.15e-5
Ce-144	(uCi/ml)	NM	NM	NM	NM	NM	NM	NM	NM	NM	NM
Eu-154	(uCi/ml)	8.82e-4	9.11e-4	9.78e-4	NM	NM	2.17e-4	9.84e-5	7.00e-5	NM	6.13e-6
Eu-155	(uCi/ml)	2.42e-3	2.41e-3	2.46e-3	NM	NM	5.13e-4	2.09e-4	2.08e-4	NM	1.65e-5
Am-241	(uCi/ml)	1.45e-3	1.41e-3	1.52e-3	NM	NM	2.38e-4	5.81e-5	5.65e-5	2.31e-5	9.67e-6
	Leach Vessel	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless	Stainless
	SA/V (m^{-1})	2000	2000	2000	NA	NA	2000	2000	2000	NA	NA
	Analysis/Date	1/31/95	1/31/95	1/31/95	1/31/95	1/31/95	1/31/95	1/31/95	1/31/95	1/31/95	1/31/95

Notes:

1. ANM = analysis not made due low alpha activity; NM = not measured; NA = not applicable; AS = Acid Strip; PCT = Product Consistency Test; MCC-1 = Materials Characterization Center (MCC-1) Test.
2. Total Beta: (as SrY-90)
3. K-40 through Am-241: by gamma energy analysis (GEA)

APPENDIX C

Physical Data

Table C.1. Physical Properties of 101-AZ Core #1 Process Slurries

	Washed Solids		Formated Slurry		Melter Feed	
	Average	Reproducibility	Average	Reproducibility	Average	Reproducibility
Intermediate Concentration						
Density	1.05					
Wt-Fraction Solid	0.104	2%				
Wt-Fraction Oxide	0.086	<0.5%				
g TO/L	93					
Intermediate Concentration	(concentration before formating)					
Density	1.09	1%				
Wt-Fraction Solid	0.140	1%			0.432	2%
Wt-Fraction Oxide	0.112	1%			0.380	1%
g TO/L	122					
Final Concentration						
Density	1.04		1.10	<0.5%	1.47	3%
Wt-Fraction Solid	0.094		0.159	1%	0.47	
Wt-Fraction Oxide	0.066		0.108	1%	0.41	
g TO/L	69		118		600	
Vol-Fraction Settled Solid	0.585		0.568	10%	0.84	2%
Density Centrifuged Solid	1.40		1.44	6%	1.74	1%
Density Centrifuged Supernate	0.98		1.04	1%	1.09	<0.5%
Vol-Fraction Centrifuged Solid	0.15		0.214	3%	0.635	5%
Wt-Fraction Centrifuged Solid	0.20		0.276	3%	0.739	4%
Wt-Fraction Dissolved Solid	NA		0.048	3%	0.068	2%

Settling Rate - Formated Slurry

Time (min)	Vol% Settled Solids	Reproducibility
0	100.0%	
65	91.5%	9%
135	86.2%	13%
260	78.8%	15%
375	74.2%	14%
435	71.8%	14%
1485	58.7%	2%
1605	58.5%	1%
1725	58.1%	1%
1870	57.2%	1%
2875	56.8%	3%
Total volume (cm):	11.8	<0.5%

Settling Rate - Melter Feed

Time (min)	Vol% Settled solids	Reproducibility
0	100.0%	<0.5%
60	98.4%	3%
120	95.5%	3%
180	94.6%	3%
300	92.2%	4%
360	90.1%	5%
1350	84.5%	6%
2875	84.0%	2%
Total Volume (cm):	12.15	2%

Settling Rate - Formated Slurry

Time (min)	Vol%	Reproducibility
0	100.0%	
60	87.3%	6%
125	80.1%	7%
180	76.7%	9%
340	69.3%	7%
420	68.0%	8%
1410	59.1%	2%
Total vol (cm):	11.8	<0.05%

Settling Rate - Formated Slurry

Time (min)	Vol%	Reproducibility
0	100.0%	
60	84.8%	2%
120	79.2%	1%
180	75.2%	1%
300	69.3%	1%
420	65.5%	2%
1610	55.9%	<0.05%
Total vol (cm):	11.8	<0.05%

Notes:
Density = g slurry/mL slurry; g TO/L = g total oxide/L sample
Density Centrifuged Solid = g centrifuged solids/mL centrifuged solids
Density Centrifuged Supernate = g centrifuged supernate/mL centrifuged supernate
Wt-Fraction Solid = g solids/g sample
Wt-Fraction Oxide = g oxide/g sample
Wt-Fraction Centrifuged Solid = g centrifuged solids/g sample
Wt-Fraction Dissolved Solid = g dissolved solids in centrifuged supernate/g sample
Vol-Fraction Settled Solid = g settled solids/g sample
Vol-Fraction Centrifuged Solid = mL centrifuged solids/mL sample
Reproducibility = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
NA = data not available.

Table C.2. Physical Properties of 101-AZ Core #2 Process Slurries

	Washed Solids		Formated Slurry		Melter Feed	
	Average	Reproducibility	Average	Reproducibility	Average	Reproducibility
Intermediate Concentration						
Density	1.19	1%				
Wt-Fraction Solid	0.215	1%				
Wt-Fraction Oxide	0.186	2%				
g TO/L	221					
Intermediate Concentration						
Density	1.10	2%				
Wt-Fraction Solid	0.128	2%				
Wt-Fraction Oxide	0.112	1%				
g TO/L	122					
Final Concentration						
Density	1.14		1.10	<0.05%	1.20[a]	1%
Wt-Fraction Solid	0.186		0.122	6%	0.429	2%
Wt-Fraction Oxide	0.166		0.092	15%	0.399	2%
g TO/L	189		101		479	
Vol-Fraction Settled Solid	0.66		0.374	4%	0.87	<0.05%
Density Centrifuged Solid	1.53		1.50	<0.05%	1.64	<0.05%
Density Centrifuged Supernate	0.98		1.01	1%	1.04	<0.05%
Vol-Fraction Centrifuged Solid	0.28		0.185	9%	0.553	1%
Wt-Fraction Centrifuged Solid	0.38		0.254	9%	0.667	<0.05%
Wt-Fraction Dissolved Solid	NA		0.0252	2%	0.0202	2%

Settling Rate - Formated Slurry

Time (min)	Vol-% Settled Solids	Reproducibility
0	100.0%	<0.5%
60	58.8%	4%
130	47.5%	5%
320	41.6%	6%
385	39.5%	6%
435	38.5%	5%
1430	37.6%	6%
1570	37.4%	4%
1690	37.4%	4%
Total Volume (cm):	11.9	<0.5%

Settling Rate - Formated Slurry

Time (min)	Vol%	Reproducibility
0	100.0%	<0.5%
10	95.6%	<0.5%
22	87.4%	<0.5%
35	80.0%	4%
45	72.5%	5%
70	58.4%	7%
Total Vol (cm):	11.9	<0.5%

Settling Rate - Melter Feed

Time (min)	Vol-% Settled Solids	Reproducibility
0	100.0%	<0.5%
30	97.5%	<0.5%
60	94.1%	1%
120	89.7%	<0.5%
270	87.4%	<0.5%
360	87.2%	<0.5%
440	87.2%	<0.5%
1435	87.2%	<0.5%
Total Volume (cm):	11.9	<0.5%

Notes:

[a] A second measurement of density before settling yielded 1.36 g/mL.
Density = g slurry/mL slurry; g TO/L = g total oxide/L sample
Density Centrifuged Solid = g centrifuged solids/mL centrifuged solids
Density Centrifuged Supernate = g centrifuged supernate/mL centrifuged supernate
Wt-Fraction Solid = g solids/g sample
Wt-Fraction Oxide = g oxide/g sample
Wt-Fraction Centrifuged Solid = g centrifuged solids/g sample
Wt-Fraction Dissolved Solid = g dissolved solids in centrifuged supernate/g sample
Vol-Fraction Settled Solid = g settled solids/g sample
Vol-Fraction Centrifuged Solid = mL centrifuged solids/mL sample
Reproducibility = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
NA = data not available.

Table C.3. Physical Properties of 102-AZ Core #1 Process Slurries

	Washed Solids		Formated Slurry		Melter Feed	
	Average	Reproducibility	Average	Reproducibility	Average	Reproducibility
Intermediate Concentration						
Density	1.10	(estimated value)	1.16	1%	1.30	1%
Wt-Fraction Solid	0.085	<0.5%	0.206	<0.5%	0.391	9%
Wt-Fraction Oxide	0.074	6%	0.155	2%	0.355	8%
g TO/L	82		180		461	
Final Concentration						
Density	1.11		1.12	<0.5%	1.34	<0.5%
Wt-Fraction Solid	0.14		0.186	1%	0.436	<0.5%
Wt-Fraction Oxide	0.11		0.131	2%	0.384	<0.5%
g TO/L	122		147		515	
Vol-Fraction Settled Solid	0.67		0.38	1%	0.635	1%
Density Centrifuged Solid	1.52		1.65	<0.5%	1.69	<0.5%
Density Centrifuged Supernate	0.99		1.04	4%	1.03	<0.5%
Vol-Fraction Centrifuged Solid	0.22		0.176	<0.5%	0.483	2%
Wt-Fraction Centrifuged Solid	0.31		0.255	<0.5%	0.603	1%
Wt-Fraction Dissolved Solid	NA		NA		NA	

Settling Rate - Formated Slurry

Time (min)	Vol-% Settled Solids	Reproducibility
0	100.0%	<0.5%
15	96.6%	<0.5%
30	93.7%	<0.5%
45	89.3%	<0.5%
60	86.1%	<0.5%
120	64.3%	4%
180	52.3%	1%
240	47.9%	<0.5%
300	46.2%	<0.5%
360	43.7%	<0.5%
420	41.6%	<0.5%
1430	38.0%	1%
1550	38.0%	1%
Total Volume (cm):	11.9	<0.5%

Settling Rate - Melter Feed

Time (min)	Vol-% Settled Solids	Reproducibility
0	100.0%	<0.5%
15	96.3%	1%
30	90.6%	1%
45	86.7%	2%
60	84.4%	3%
120	77.9%	3%
180	74.0%	2%
255	70.7%	2%
300	68.7%	1%
360	67.2%	<0.5%
1560	65.2%	<0.5%
1620	64.5%	<0.5%
2820	63.7%	1%
3000	63.5%	1%
Total Volume (cm):	12.2	<0.5%

Notes:

Density = g slurry/mL slurry; g TO/L = g total oxide/L sample
Density Centrifuged Solid = g centrifuged solids/mL centrifuged solids
Density Centrifuged Supernate = g centrifuged supernate/mL centrifuged supernate
Wt-Fraction Solid = g solids/g sample
Wt-Fraction Oxide = g oxide/g sample
Wt-Fraction Centrifuged Solid = g centrifuged solids/g sample
Wt-Fraction Dissolved Solid = g dissolved solids in centrifuged supernate/g sample
Vol-Fraction Settled Solid = g settled solids/g sample
Vol-Fraction Centrifuged Solid = mL centrifuged solids/mL sample
Reproducibility = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
NA = data not available.

Table C.4. Physical Properties of NCAW Simulant Process Slurries

	Washed Solids		Formated Slurry		Melter Feed	
	Average	Reproducibility	Average	Reproducibility	Average	Reproducibility
Run 1						
Density	1.13	2%			1.32	
Wt-Fraction Solid	NA				0.411	2%
Wt-Fraction Oxide	0.123	<0.5%			0.378	2%
g TO/L	139				499	
Run 2						
Density			1.09	<0.5%	1.32	
Wt-Fraction Solid			NA		0.453	4%
Wt-Fraction Oxide			0.102	1%	0.400	2%
g TO/L			111		528	
Hot Cell Run						
Density	1.14	1%	1.12	1%	1.39	<0.5%
Wt-Fraction Solid	NA		0.158	1%	0.456	1%
Wt-Fraction Oxide	0.153	<0.5%	0.097	50%	0.430	2%
g TO/L	175	2%	109	49%	597	2%
Run 3						
Density	1.11	<0.5%	1.11	<0.5%	1.28	<0.5%
Wt-Fraction Solid	0.153	26%	0.192	9%	0.370	2%
Wt-Fraction Oxide	0.101	48%	0.145	10%	0.341	5%
g TO/L	112		161		438	2%
Vol-Fraction Settled Solid			0.87	1%	0.870	1%
Density Centrifuged Solid			1.27	2%	1.50	<0.5%
Density Centrifuged Supernate			1.01	<0.5%	1.01	1%
Vol-Fraction Centrifuged Solid			0.400	1%	0.583	1%
Wt-Fraction Centrifuged Solid			0.462	1%	0.672	1%
Wt-Fraction Dissolved Solid			0.024	21%	0.014	11%

Settling Rate - Formated Slurry

Time (min)	Vol% Settled Solids	Reproducibility
0	100.0%	<0.5%
80	98.8%	<0.5%
120	98.2%	2%
180	98.3%	3%
270	97.3%	2%
300	97.1%	2%
1410	88.6%	2%
1580	88.2%	3%
1750	88.0%	3%
3165	86.7%	1%
Total Volume (cm):	11.85	3%

Settling Rate - Melter Feed

Time (min)	Vol% Settled Solids	Reproducibility
0	100.0%	<0.5%
65	99.2%	<0.5%
120	98.1%	<0.5%
250	96.0%	<0.5%
370	93.6%	1%
430	92.8%	<0.5%
1425	87.5%	<0.5%
1600	87.1%	<0.5%
1695	87.1%	<0.5%
1800	87.1%	<0.5%
7200	86.9%	1%
Total Volume (cm):	11.8	<0.5%

Notes: Density = g slurry/mL slurry; g TO/L = g total oxide/L sample
Density Centrifuged Solid = g centrifuged solids/mL centrifuged solids
Density Centrifuged Supernate = g centrifuged supernate/mL centrifuged supernate
Wt-Fraction Solid = g solids/g sample
Wt-Fraction Oxide = g oxide/g sample
Wt-Fraction Centrifuged Solid = g centrifuged solids/g sample
Wt-Fraction Dissolved Solid = g dissolved solids in centrifuged supernate/g sample
Vol-Fraction Settled Solid = g settled solids/g sample
Vol-Fraction Centrifuged Solid = mL centrifuged solids/mL sample
Reproducibility = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
NA = data not available.

Table C.5. Physical Properties of 102-AZ Core #1 Simulant Process Slurries

	Formatted Slurry		Formated Slurry		Melter Feed	
	Average	Reproducibility	Average	Reproducibility	Average	Reproducibility
Intermediate Concentration						
Density			1.16	1%		
Wt-Fraction Solid			0.239	<0.5%		
Wt-Fraction Oxide			0.148	<0.5%		
g TO/L			170.2			
Intermediate Concentration						
Density			1.14	<0.5%		
Wt-Fraction Solid			0.212	2%		
Wt-Fraction Oxide			0.120	1%		
g TO/L			137			
Intermediate Concentration						
Density			1.14	<0.5%		
Wt-Fraction Solid			0.212	1%		
Wt-Fraction Oxide			0.122	3%		
g TO/L			139			
Final Concentration						
Density	1.14		1.14	1%	1.42	2%
Wt-Fraction Solid			0.222	1%	0.478	<0.5%
Wt-Fraction Oxide			0.129	<0.5%	0.404	<0.5%
g TO/L	125		147		573	
Vol-Fraction Settled Solid			0.84	<0.5%	1.00	<0.5%
Density Centrifuged Solid			1.34	1%	NA	
Density Centrifuged Supernate			1.04	1%	NA	
Vol-Fraction Centrifuged Solid			0.421	2%	NA	
Wt-Fraction Centrifuged Solid			0.484	<0.5%	NA	
Wt-Fraction Dissolved Solid			NA		NA	

Settling Rate - Formated Slurry

Time (min)	Vol% Settled Solids	Reproducibility
0	100.0%	
17	100.0%	<0.5%
32	99.2%	<0.5%
47	99.2%	<0.5%
62	99.2%	<0.5%
127	98.3%	<0.5%
182	98.3%	<0.5%
242	99.2%	<0.5%
302	98.7%	1%
362	97.9%	1%
1277	89.9%	<0.5%
1397	89.0%	1%
1517	88.4%	1%
1637	88.2%	1%
1757	87.3%	1%
2822	85.7%	1%
2882	85.2%	<0.5%
7142	84.4%	<0.5%
7352	84.4%	<0.5%
Total Volume (cm):	11.9	4%

Settling Rate - Melter Feed (no settling observed)

Notes:
Density = g slurry/mL slurry; g TO/L = g total oxide/L sample
Density Centrifuged Solid = g centrifuged solids/mL centrifuged solids
Density Centrifuged Supernate = g centrifuged supernate/mL centrifuged supernate
Wt-Fraction Solid = g solids/g sample
Wt-Fraction Oxide = g oxide/g sample
Wt-Fraction Centrifuged Solid = g centrifuged solids/g sample
Wt-Fraction Dissolved Solid = g dissolved solids in centrifuged supernate/g sample
Vol-Fraction Settled Solid = g settled solids/g sample
Vol-Fraction Centrifuged Solid = mL centrifuged solids/mL sample
Reproducibility = (|analysis 1 - analysis 2| / (analysis 1 + analysis 2) / 2) x 100%.
NA = data not available.

```
G A L A I   —   C I S — 1

Computerized Inspection System

SAMPLE NAME : 101AZ-C1-FOR
FILE NAME   : 903616.001
--------------------------------------------------------------------------------------------
DATE        : 17/05/1990   | ACQ. RANGE   : 0.5-150    | COUNTS     : 12:120
TIME        : 15:30        | ACQ. MODE    : SAMPLE     | S.N.F.     : 1.73
CONFIG.     : 1 (0.7 S1)   | ACQ. TIME    : 256 SEC    | S.D.U.     : 299.
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE  : 4          | CONCENTR.  : 4.7E-0
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V) | SOLIDS     : 2.2E-0
--------------------------------------------------------------------------------------------

PROBABILITY VOLUME DENSITY GRAPH

Name:  101AZ-C1-FOR                                        Median   :  4.85µm
 2.2E-05 cc/ml(100.0%)             Mean(nv):  2.06µm      Mean(vm):  9.81µm
Mode at   37.04 µm                 S.D.(nv):  1.20µm      S.D.(vm):  9.99µm
   << SCALE RANGE (µm): ADJUSTED >>                        Conf(vm): 96.98 %
```

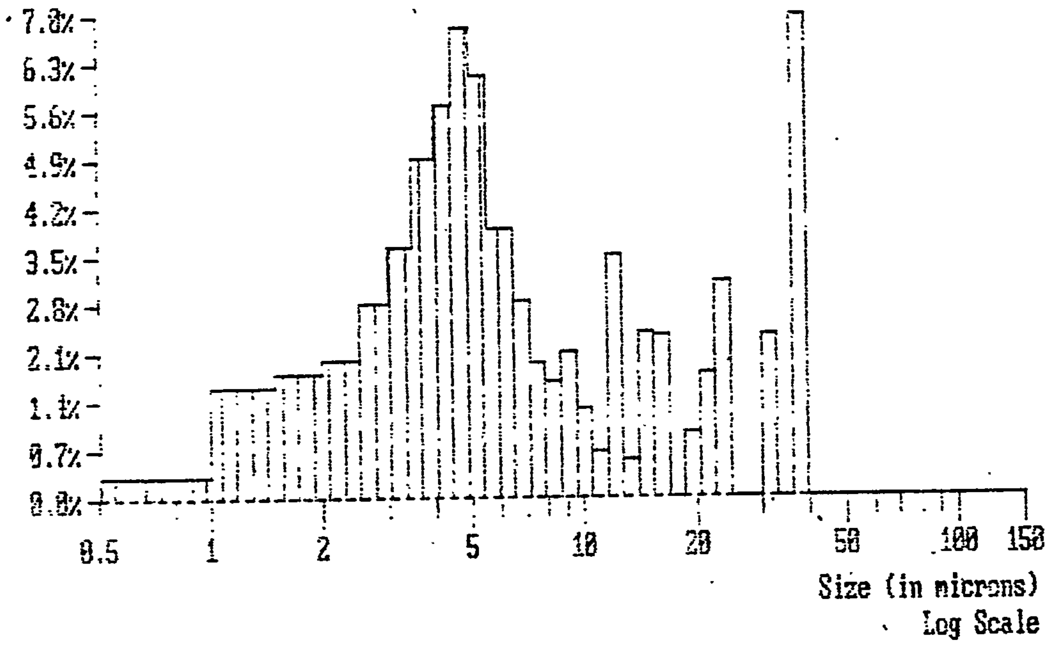


Table C.6. Particle Size Distributions - 101-AZ Core #1 Formated Slurry

```
SAMPLE NAME : 101AZ-C1-FOR
FILE NAME   : 903516.001
--------------------------------------------------------------------------------
DATE        : 17/05/1990   | ACQ. RANGE   : 0.5-150       | COUNTS    : 121020
TIME        : 15:30        | ACQ. MODE    : SAMPLE        | S.N.F.    : 0.73
CONFIG.     : 1 (0.7 S1)   | ACQ. TIME    : 256 SEC       | S.D.U.    . 2591
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE  : 4             | CONCENTR. : 4.9E+06 #/m
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V)    | SOLIDS    : 5.5E-02 %
--------------------------------------------------------------------------------
```

PROBABILITY NUMBER DENSITY GRAPH

```
Name:  101AZ-C1-FOR                                  Median  :  1.01μm
4.9E+06 #/ml(100.0%)                                 Mean(nl):  1.27μm
Mode at    1.25 μm                                   S.D.(nl):  0.90μm
   << SCALE RANGE (μm): ADJUSTED >>                  Conf(nl):100.00 %
```

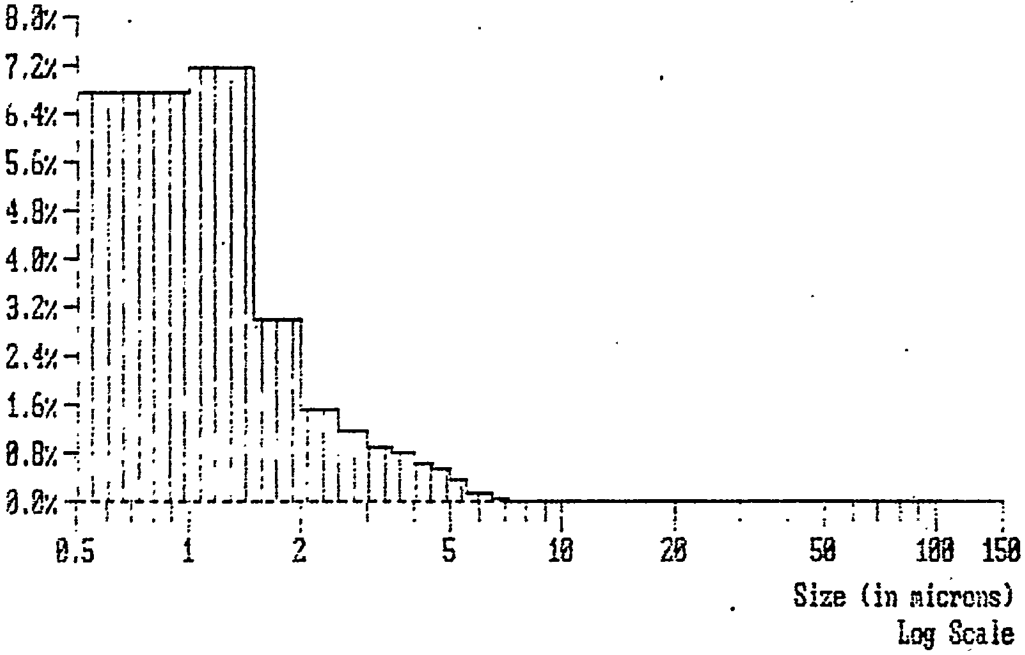


Table C.6. (continued) Particle Size Distributions - 101-AZ Core #1 Formated Slurry

```
VOLUME  DISTRIBUTION  TABLE  (RANGES)

SAMPLE NAME : 101AZ-C1-FOR
FILE NAME   : 903616.001
------------------------------------------------------------------------------------
DATE        : 17/05/1990  | ACQ. RANGE  : 0.5-150       | COUNTS     : 121123
TIME        : 15:30       | ACQ. MODE   : SAMPLE        | S.N.F.     : [illegible]
CONFIG.     : 1 (0.7 S1)  | ACQ. TIME   : 256 SEC       | S.D.U.     : 5991
CELL TYPE   : MAGNETIC (2)| SAMPLE SIZE : 4             | CONCENTR.  : 4.9E+04
SAMPLE TYPE : REGULAR     | REQ. CONF.  : 95.00 %(V)    | SOLIDS.    : 3.2E-03.
------------------------------------------------------------------------------------

      RANGE (microns)           LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%
       0.0 -    1.0               2.38                2.39                97.62
       1.0 -    2.0              12.36               14.74                85.26
       2.0 -    3.0              10.12               24.86                75.[illegible]
       3.0 -    4.0              12.82               37.68                [illegible]
       4.0 -    5.0              14.60               52.28
       5.0 -    6.0               9.77               62.05
       6.0 -    7.0               5.56               67.61                32.04
       7.0 -    8.0               2.72               70.40                29.60
       8.0 -    9.0               2.46               72.86                27.14
       9.0 -   10.0               1.33               74.19                25.81
      10.0 -   20.0              11.51               85.71                14.29
      20.0 -   30.0               4.95               90.66                 9.34
      30.0 -   40.0               9.34              100.00
      40.0 -   50.0               0.00              100.00                 [illegible]
      50.0 -   60.0               0.00              100.00                 0.00
      60.0 -   70.0               0.00              100.00                 0.00
      70.0 -   80.0               0.00              100.00                 0.00
      80.0 -   90.0               0.00              100.00                 0.0
      90.0 -  100.0               0.00              100.00                 0.00
     100.0 -  150.0               0.00              100.00                 0.0[illegible]
```

Table C.6. (continued) Particle Size Distributions - 101-AZ Core #1 Formated Slurry

NUMBER DISTRIBUTION TABLE (RANGES)

```
SAMPLE NAME : 101AZ-C1-FOR
FILE NAME   : 905615.001
--------------------------------------------------------------------------------
DATE        : 17/05/1990   | ACQ. RANGE   : 0.5-150      | COUNTS     : [illegible]
TIME        : 15:30        | ACQ. MODE    : SAMPLE       | S.N.F.     : [illegible]
CONFIG.     : 1 (0.7 81)   | ACQ. TIME    : 256 SEC      | S.D.U.     : 399.
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE  : 4            | CONCENTR.  : [illegible]
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V)   | SOLIDS     : [illegible]
--------------------------------------------------------------------------------
```

RANGE (microns)	LOCAL (%)	UNDER(%)-CUMULATIVE	-OVER(%)
0.0 - 1.0	49.46	49.46	50.54
1.0 - 2.0	39.69	89.15	10.85
2.0 - 3.0	5.89	95.04	[illegible]
3.0 - 4.0	2.66	97.70	2.30
4.0 - 5.0	1.42	99.13	[illegible]
5.0 - 6.0	0.54	99.66	[illegible]
6.0 - 7.0	0.18	99.84	[illegible]
7.0 - 8.0	0.06	99.90	[illegible]
8.0 - 9.0	0.03	99.93	[illegible]
9.0 - 10.0	0.01	99.95	[illegible]
10.0 - 20.0	0.05	99.99	[illegible]
20.0 - 30.0	0.00	100.00	[illegible]
30.0 - 40.0	0.00	100.00	[illegible]
40.0 - 50.0	0.00	100.00	[illegible]
50.0 - 60.0	0.00	100.00	[illegible]
60.0 - 70.0	0.00	100.00	[illegible]
70.0 - 80.0	0.00	100.00	[illegible]
80.0 - 90.0	0.00	100.00	[illegible]
90.0 - 100.0	0.00	100.00	[illegible]
100.0 - 150.0	0.00	100.00	[illegible]

Table C.6. (continued) Particle Size Distributions - 101-AZ Core #1 Formated Slurry

```
G A L A I   -   [illegible]

Computerized Inspection System

SAMPLE NAME : 101AZ-C1 MELT-AN
FILE NAME   : 904665.001
--------------------------------------------------------------------------------------------
DATE        : 07/0[illegible]/1990  | ACQ. RANGE   : 0.5-150      | COUNTS    : [illegible]
TIME        : 15:18          | ACQ. MODE    : SAMPLE       | S.D.F.    : [illegible]
CONFIG.     : 1 (0:7 S1)     | ACQ. TIME    : 21 SEC       | S.D.D.    : [illegible]
CELL TYPE   : MAGNETIC (2)   | SAMPLE SIZE  : 8            | CONCENTR. : [illegible]
SAMPLE TYPE : REGULAR        | REQ. CONF.   : 95.00 %(V)   | SOLIDS    : 1.9E-[illegible]
--------------------------------------------------------------------------------------------

PROBABILITY VOLUME DENSITY GRAPH

Name:  101AZ-C1 MELT-AN                                   Median   :  4.10µm
 1.9E-05 cc/ml(100.0%)              Mean(nv):  1.81µm     Mean(vm):  4.26µm
Mode at     4.25 µm                 S.D.(nv):  0.99µm     S.D.(vm):  2.46µm
    << SCALE RANGE (µm): ADJUSTED >>                      Conf(vm): 99.42 %
```

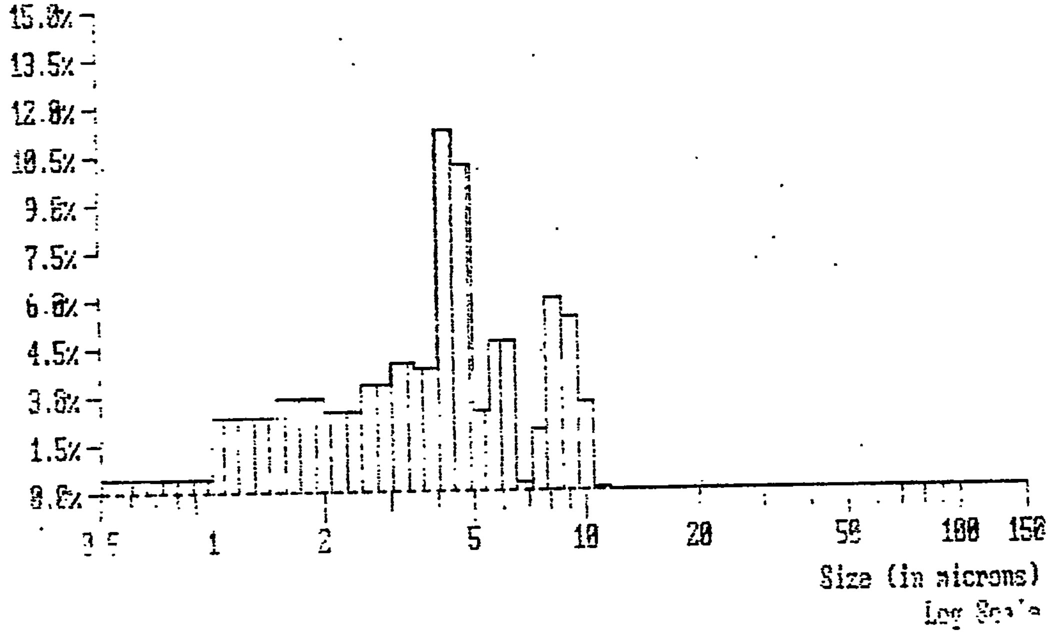


Table C.7. Particle Size Distributions - 101-AZ Core #1 Melter Feed

```
AMPLE NAME : 101AZ-C1 MELT-AN
ILE NAME   : 904665.001
------------------------------------------------------------------------------------------------
ATE         : 07/06/1990  | ACQ. RANGE  : 0.5-150       | COUNTS     : 9960
IME         : 15:18       | ACQ. MODE   : SAMPLE        | S.N.F.     : 0.87
ONFIG.      : 1 (0.7 S1)  | ACQ. TIME   : 21 SEC        | S.D.L.     : 3716
ELL TYPE    : MAGNETIC (2)| SAMPLE SIZE : 2             | CONCENTR.  : 3.6E+06 #.
AMPLE TYPE  : REGULAR       REG. CONF.  : 95.00 %(V)    | SOLIDS     : 1.6E-03 %
------------------------------------------------------------------------------------------------
```

PROBABILITY NUMBER DENSITY GRAPH

```
ame:  101AZ-C1 MELT-AN                      Median   :  0.99µm
5.0E+06 #/ml(100.0%)                        Mean(nl):  1.22µm
ode at     1.25 µm                          S.D.(nl):  0.79µm
   << SCALE RANGE (µm): ADJUSTED >>         Conf(nl):100.00 %
```

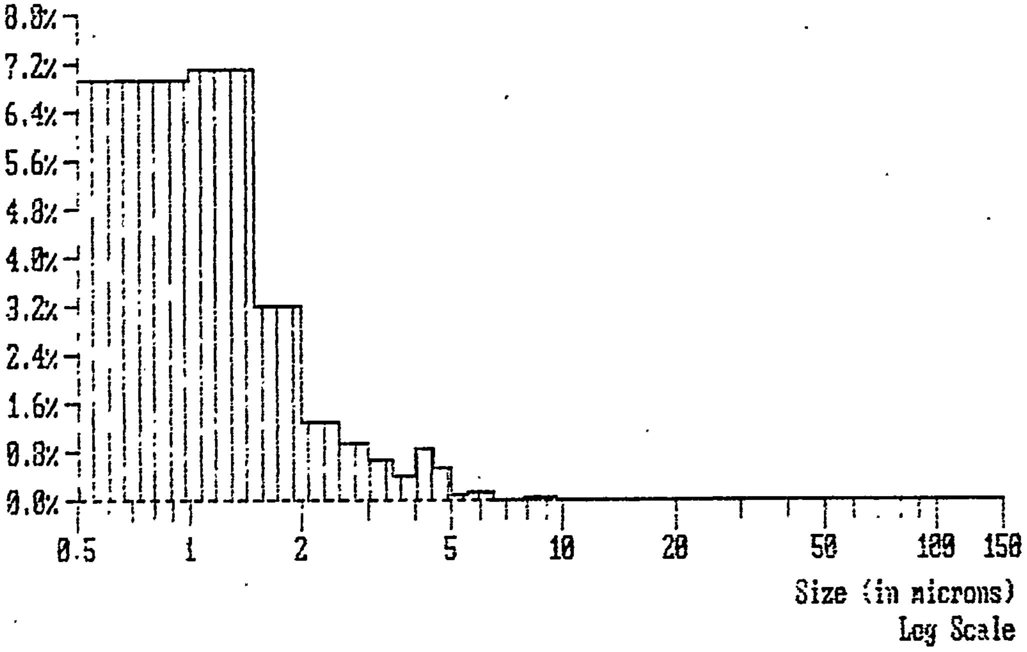


Table C.7. (continued) Particle Size Distributions - 101-AZ Core #1 Melter Feed

```
VOLUME  DISTRIBUTION  TABLE  (RANGES)

SAMPLE NAME  : 101AZ-C1 MELT-AN
FILE NAME    : 904665.001
-----------------------------------------------------------------------------------------
DATE         : 07/06/1990   | ACQ. RANGE   : 0.5-150      | COUNTS      : 8260
TIME         : 15:18        | ACQ. MODE    : SAMPLE       | S.N.F.      : 0.67
CONFIG.      : 1 (0.7 S1)   | ACQ. TIME    : 21 SEC       | S.D.U.      : 37.5
CELL TYPE    : MAGNETIC (2) | SAMPLE SIZE  : 2            | CONCENTR.   : 5.8E+06
SAMPLE TYPE  : REGULAR      | REQ. CONF.   : 95.00 %(V)   | SOLIDS      : 1.5E-0[illegible]
-----------------------------------------------------------------------------------------

      RANGE (microns)          LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%)
        0.0 -    1.0             3.64               3.64               96.36
        1.0 -    2.0            17.04              22.68               77.32
        2.0 -    3.0            12.44              35.13               64.87
        3.0 -    4.0            11.96              47.09               52.91
        4.0 -    5.0            25.29              72.38               27.62
        5.0 -    6.0             9.16              81.54               18.46
        6.0 -    7.0             1.79              83.33               16.67
        7.0 -    8.0             2.69              86.02               13.98
        8.0 -    9.0             8.87              94.39                5.61
        9.0 -   10.0             2.20              97.09                2.91
       10.0 -   20.0             2.91             100.00                0.00
       20.0 -   30.0             0.00             100.00                0.00
       30.0 -   40.0             0.00             100.00                0.00
       40.0 -   50.0             0.00             100.00                0.00
       50.0 -   60.0             0.00             100.00                0.00
       60.0 -   70.0             0.00             100.00                0.00
       70.0 -   80.0             0.00             100.00                0.00
       80.0 -   90.0             0.00             100.00                0.00
       90.0 -  100.0             0.00             100.00                0.00
      100.0 -  150.0             0.00             100.00                0.00
```

Table C.7. (continued) Particle Size Distributions - 101-AZ Core #1 Melter Feed

```
NUMBER DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : 101AZ-C1 MELT-AN
FILE NAME   : 904565.001
------------------------------------------------------------------------------------------
DATE        : 07/06/1990   | ACQ. RANGE   : 0.5-150      : COUNTS     : [illegible]
TIME        : 15:18        | ACQ. MODE    : SAMPLE       | S.N.F.     : [illegible]
CONFIG.     : 1 (0.7 S1)   | ACQ. TIME    : 21 SEC       | S.D.U.     : [illegible]
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE  : 2            | CONCENTR.  : [illegible]
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V)   | SOLIDS     : [illegible]
------------------------------------------------------------------------------------------

   RANGE (microns.          LOCAL (%)           UNDER(%)-CUMULATIVE-[illegible]
    0.0 -    1.0              50.85                 50.85              [illegible]
    1.0 -    2.0              40.30                 91.15              [illegible]
    2.0 -    3.0               4.92                 96.07              [illegible]
    3.0 -    4.0               1.73                 97.80              [illegible]
    4.0 -    5.0               1.69                 99.49              [illegible]
    5.0 -    6.0               0.31                 99.80              [illegible]
    6.0 -    7.0               0.04                 99.85              [illegible]
    7.0 -    8.0               0.04                 99.88              [illegible]
    8.0 -    9.0               0.08                 99.97              [illegible]
    9.0 -   10.0               0.02                 99.98              [illegible]
   10.0 -   20.0               0.02                100.00              [illegible]
   20.0 -   30.0               0.00                100.00              [illegible]
   30.0 -   40.0               0.00                100.00              [illegible]
   40.0 -   50.0               0.00                100.00              [illegible]
   50.0 -   60.0               0.00                100.00              [illegible]
   60.0 -   70.0               0.00                100.00              [illegible]
   70.0 -   80.0               0.00                100.00              [illegible]
   80.0 -   90.0               0.00                100.00              [illegible]
   90.0 -  100.0               0.00                100.00              [illegible]
  100.0 -  150.0               0.00                100.00              [illegible]
```

Table C.7. (continued) Particle Size Distributions - 101-AZ Core #1 Melter Feed

```
  G A L A I   —   C I S  —  1

  Computerized  Inspection  System

SAMPLE NAME : 101AZ-C2-FOR
FILE NAME   : 906009.001
------------------------------------------------------------------------------------------
DATE        : 07/09/1990   | ACQ. RANGE   : 0.5-150     | COUNTS    : 325[illegible]
TIME        : 11:24        | ACQ. MODE    : SAMPLE      | S.N.F.    : 0.7[illegible]
CONFIG.     : 1 (0.7 S1)   | ACQ. TIME    : 78 SEC      | S.O.U.    : [illegible]
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE  : 3           | CONCENTR. : [illegible]
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V)  | SOLIDS    : 2.3E-03
------------------------------------------------------------------------------------------
```

PROBABILITY VOLUME DENSITY GRAPH

```
Name:  101AZ-C2-FOR                                   Median   :  4.51µm
 2.3E-05 cc/ml(100.0%)        Mean(nv):  2.18µm        Mean(vm):  5.37µm
Mode at    4.75 µm            S.D.(nv):  1.26µm        S.D.(vm):  3.79µm
   << SCALE RANGE (µm): ADJUSTED >>                    Conf(vm): 99.83 %
```

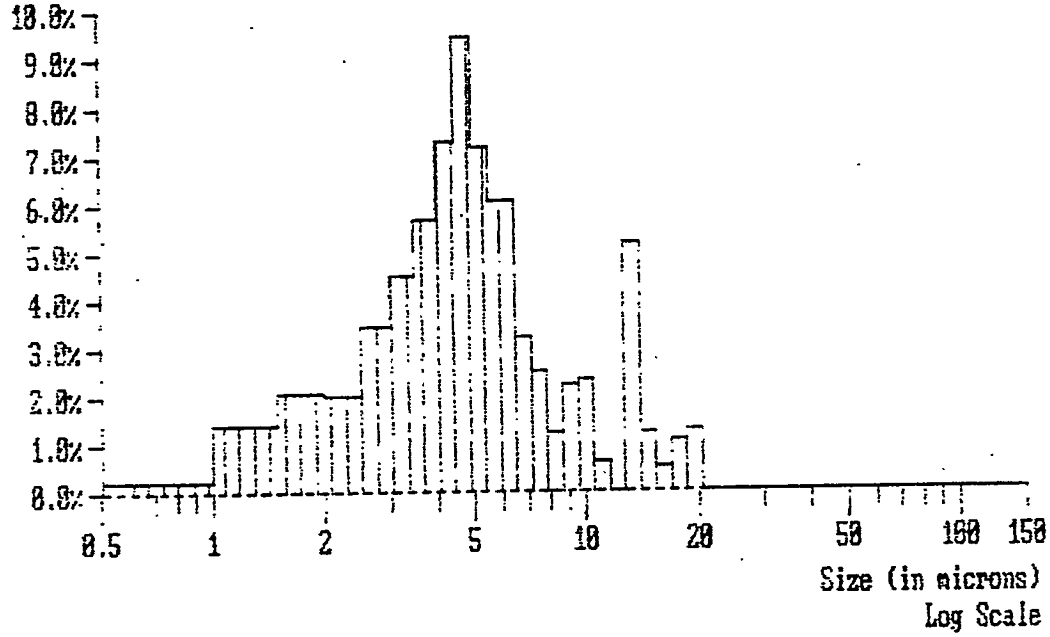


Table C.8. Particle Size Distributions - 101-AZ Core #2 Formated Slurry

```
SAMPLE NAME : 101AZ-C2-FOR
FILE NAME   : 903009.001
-------------------------------------------------------------------------------------------
DATE        : 07/09/1990   | ACQ. RANGE   : 0.5-150       | COUNTS    : 3250[illegible]
TIME        : 11:24.       | ACQ. MODE    : SAMPLE        | S.N.R.    : 0.9[illegible]
CONFIG.     : 1 (0.7 S1)   | ACQ. TIME    : 78 SEC        | S.D.U.    : 5395
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE  : 3             | CONCENTR. : 4.2E+06 #/ml
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V)    | SOLIDS    : 3.3E-03 %
-------------------------------------------------------------------------------------------
```

PROBABILITY NUMBER DENSITY GRAPH

```
Name:  101AZ-C2-FOR                              Median  :  1.14µm
 4.2E+06 #/ml(100.0%)                            Mean(nl):  1.42µm
Mode at     1.25 µm                              S.D.(nl):  1.01µm
   << SCALE RANGE (µm): ADJUSTED >>              Conf(nl):100.00 %
```

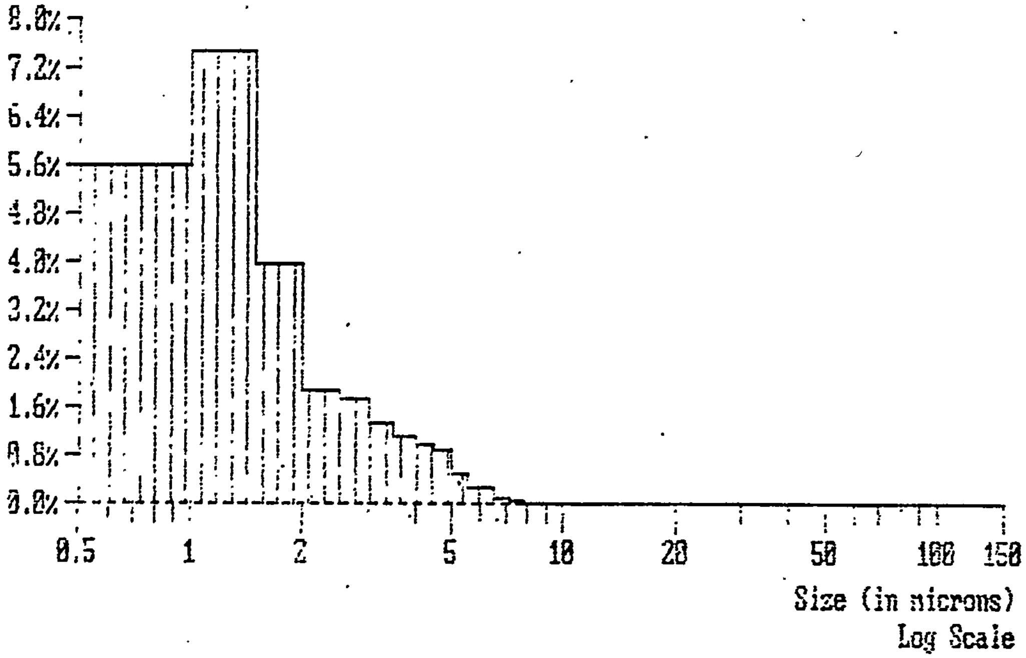


Table C.8. (continued) Particle Size Distributions - 101-AZ Core #2 Formated Slurry

```
VOLUME DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : 101AZ-C2-FOR
FILE NAME   : 906009.001
------------------------------------------------------------------------------------------
DATE        : 07/09/1990   | ACQ. RANGE   : 0.5-150          | COUNTS     : 38506
TIME        : 11:24        | ACQ. MODE    : SAMPLE           | S.N.F.     : [illegible]-4
CONFIG.     : [illegible] 7 81) | ACQ. TIME : 78 SEC         | S.D.U.     : [illegible]
CELL TYPE   : [illegible] (E) | SAMPLE SIZE : 3.             | CONCENTS.  : [illegible]
SAMPLE TYPE : REGULAR      | REQ. CONF.   : [illegible]      | SOLIDS     : [illegible]
------------------------------------------------------------------------------------------

       RANGE (microns)          LOCAL (%)          UNDER(%)-CUMULATIVE-[illegible]
        0.0 -    1.0              1.66               1.66                 98.34
        1.0 -    2.0             12.22              13.87                 86.13
        2.0 -    3.0             11.50              25.37                 74.63
        3.0 -    4.0             15.39              40.76                 59.24
        4.0 -    5.0             19.54              60.30                 [illegible]
        5.0 -    6.0             13.12              72.48                 [illegible]
        6.0 -    7.0              7.43              80.35                 [illegible]
        7.0 -    8.0              3.59              84.45                 [illegible]
        8.0 -    9.0              1.32              85.76                 [illegible]
        9.0 -   10.0              3.56              89.33                 [illegible]
       10.0 -   20.0             10.67             100.00                 [illegible]
       20.0 -   30.0              0.00             100.00                 [illegible]
       30.0 -   40.0              0.00             100.00                 [illegible]
       40.0 -   50.0              0.00             100.00                 [illegible]
       50.0 -   60.0              0.00             100.00                 [illegible]
       60.0 -   70.0              0.00             100.00                 [illegible]
       70.0 -   80.0              0.00             100.00                 [illegible]
       80.0 -   90.0              0.00             100.00                 [illegible]
       90.0 -  100.0              0.00             100.00                 [illegible]
      100.0 -  150.0              0.00             100.00                 [illegible]
```

Table C.8. (continued) Particle Size Distributions - 101-AZ Core #2 Formated Slurry

```
UMBER  DISTRIBUTION  TABLE  (RANGES)

AMPLE NAME : 101AZ-C2-FOR
ILE NAME   : 906009.001
------------------------------------------------------------------------------------------------
ATE          : 07/09/1990   | ACQ. RANGE    : 0.5-150        | COUNTS      : 32[illegible]6
IME          : 11:24        | ACQ. MODE     : SAMPLE         : S.N.F.      : [illegible]
ONFIG        : 1 (0.7 31)   | ACQ. TIME     : 73 SEC         | S.D.U.      : [illegible]
ELL TYPE     : MAGNETIC (2) | SAMPLE SIZE   : 3              : CONCENTR.   : [illegible]
AMPLE TYPE   : REGULAR      | REQ. CONF.    : 95.00 %(V)     | SOLIDS      : 2.2E-[illegible] %
------------------------------------------------------------------------------------------------

     RANGE (microns)          LOCAL (%)         UNDER(%)-CUMULATIVE-OVER(%)
       0.0 -     1.0            40.89              40.89                [illegible]
       1.0 -     2.0            43.93              84.57                [illegible]
       2.0 -     3.0             7.74              95.60                [illegible]
       3.0 -     4.0             3.81              96.42                [illegible]
       4.0 -     5.0             2.25              98.66                [illegible]
       5.0 -     6.0             0.34              99.51                [illegible]
       6.0 -     7.0             0.29              99.80                [illegible]
       7.0 -     8.0             0.09              99.89                [illegible]
       8.0 -     9.0             0.02              99.91                [illegible]
       9.0 -    10.0             0.04              99.96                [illegible]
      10.0 -    20.0             0.04             100.00                [illegible]
      20.0 -    30.0             0.00             100.00                [illegible]
      30.0 -    40.0             0.00             100.00                [illegible]
      40.0 -    50.0             0.00             100.00                [illegible]
      50.0 -    60.0             0.00             100.00                [illegible]
      60.0 -    70.0             0.00             100.00                [illegible]
      70.0 -    80.0             0.00             100.00                [illegible]
      80.0 -    90.0             0.00             100.00                [illegible]
      90.0 -   100.0             0.00             100.00                [illegible]
     100.0 -   150.0             0.00             100.00                [illegible]
```

Table C.8. (continued) Particle Size Distributions - 101-AZ Core #2 Formated Slurry

```
G A L A I  -  C I S - 1

Computerized Inspection System
```

```
SAMPLE NAME : 101AZ C2-FOR
FILE NAME   : 906009.002
---------------------------------------------------------------------------------------------
DATE        : 19/09/1990  ! ACQ. RANGE   : 0.5-150    ! COUNTS     : [illegible]
TIME        : 10:5?       ! ACQ. MODE    : SAMPLE     ! S.N.F.     : [illegible]
CONFIG.     : 1 (0.7 S1)  ! ACQ. TIME    : 236 SEC    ! S.D.U.     : 947,
CELL TYPE   : MAGNETIC (2)! SAMPLE SIZE  : 4          ! CONCENTR.  : 5.0E+??
SAMPLE TYPE : REGULAR     ! REQ. CONF.   : 95.00 %(V) ! SOLIDS     : 2.5E-05
---------------------------------------------------------------------------------------------
```

PROBABILITY VOLUME DENSITY GRAPH

```
Name:  101AZ C2-FOR                                        Median   :  4.78µm
 2.5E-05 cc/ml(100.0%)            Mean(nv):  2.11µm        Mean(vm):  6.50µm
Mode at    4.75 µm                S.D.(nv):  1.25µm        S.D.(vm):  5.57µm
    << SCALE RANGE (µm): ADJUSTED >>                       Conf(vm): 99.99 %
```

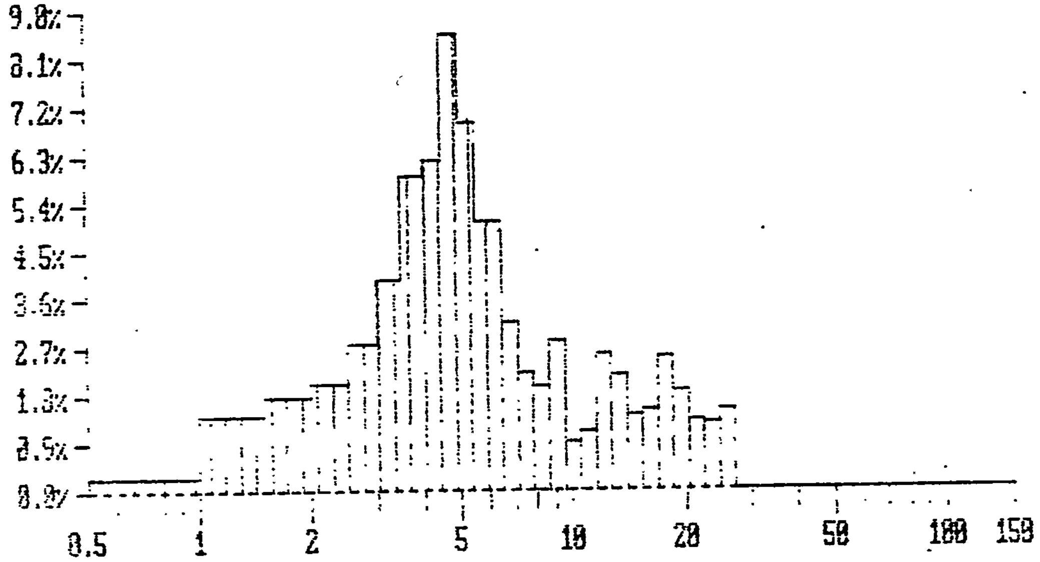


Table C.9. Particle Size Distributions - 101-AZ Core #2 Formated Slurry (Duplicate)

```
AMPLE NAME : 101AZ C2-FOR
ILE NAME   : 906009.008
----------------------------------------------------------------------------------------------
ATE         : 19/09/1990   | ACQ. RANGE   : 0.5-150        | COUNTS     : 94412
IME         : 10:55          ACQ. MODE    : SAMPLE         | S.N.F.     : 0.93
ONFIG.      : 1 (0.7 SI)   | ACQ. TIME    : 236 SEC        | S.D.U.     : 3470
ELL TYPE    : MAGNETIC (2) | SAMPLE SIZE  : 4              | CONCENTR.  : 5.0E+06 #/
AMPLE TYPE  : REGULAR      | REQ. CONF.   : 95.00 %(V)     | SOLIDS     : 3.8E-02 %
----------------------------------------------------------------------------------------------
```

PROBABILITY NUMBER DENSITY GRAPH

```
lame:  101AZ C2-FOR                              Median  :  1.03µm
5.0E+06 #/ml(100.0%)                             Mean(nl):  1.32µm
lode at     1.25 µm                              S.D.(nl):  0.96µm
  << SCALE RANGE (µm): ADJUSTED >>               Conf(nl):100.00 %
```

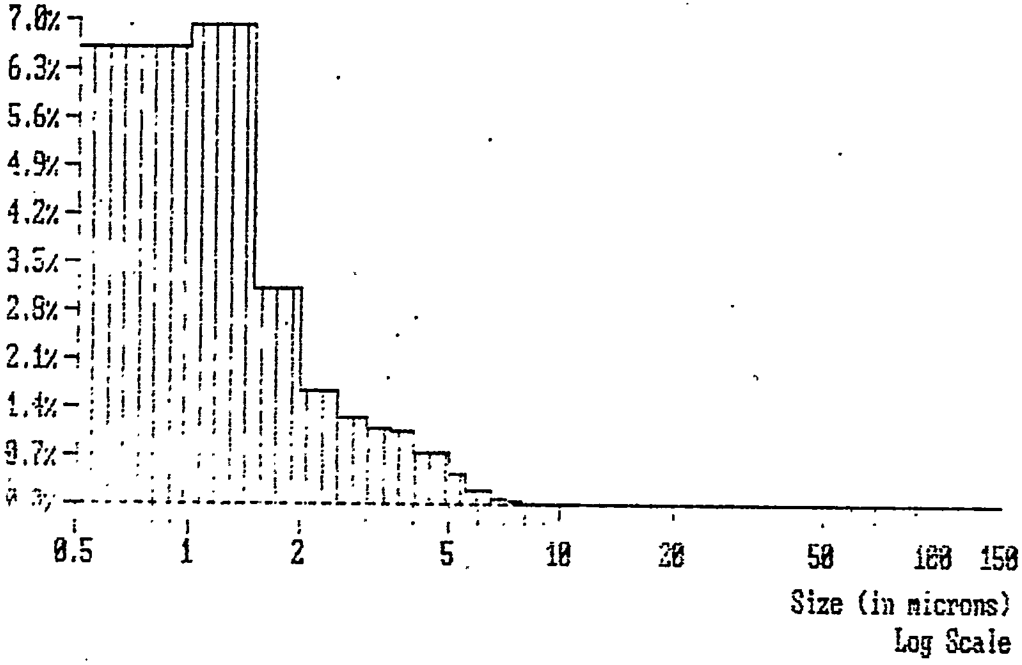


Table C.9. (continued) Particle Size Distributions - 101-AZ Core #2 Formated Slurry (Duplicate)

```
VOLUME DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : 101AZ C2-FOR
FILE NAME   : 906009.002
-----------------------------------------------------------------------------------------
DATE        : 19/09/1990  | ACQ. RANGE   : 0.5-150      | COUNTS     : 94412
TIME        : 10:55       | ACQ. MODE    : SAMPLE       | S.N.F.     : 0.93
CONFIG.     : 1 (0.7 31)  | ACQ. TIME    : 236 SEC      | S.D.U.     : 2470
CELL TYPE   : MAGNETIC (2)| SAMPLE SIZE  : 4            | CONCENTR.  : 5.3E+06
SAMPLE TYPE : REGULAR     | REQ. CONF.   : 95.00 %(V)   | SOLIDS     : 2.5E-03
-----------------------------------------------------------------------------------------

      RANGE (microns)            LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%)
        0.0  -    1.0               2.17                  2.17            97.83
        1.0  -    2.0              11.55                 13.72            86.28
        2.0  -    3.0               9.98                 23.70            76.30
        3.0  -    4.0              14.73                 38.42            61.58
        4.0  -    5.0              17.21                 55.63            44.37
        5.0  -    6.0              12.38                 68.01            31.99
        6.0  -    7.0               6.24                 74.25            25.75
        7.0  -    8.0               3.12                 77.36            22.64
        8.0  -    9.0               2.66                 80.02            19.98
        9.0  -   10.0               2.37                 82.40            17.60
       10.0  -   20.0              12.81                 95.21             4.79
       20.0  -   30.0               4.79                100.00             0.00
       30.0  -   40.0               0.00                100.00             0.00
       40.0  -   50.0               0.00                100.00             0.00
       50.0  -   60.0               0.00                100.00             0.00
       60.0  -   70.0               0.00                100.00             0.00
       70.0  -   80.0               0.00                100.00             0.00
       80.0  -   90.0               0.00                100.00             0.00
       90.0  -  100.0               0.00                100.00             0.00
      100.0  -  150.0               0.00                100.00             0.00
```

Table C.9. (continued) Particle Size Distributions - 101-AZ Core #2 Formated Slurry (Duplicate)

```
NUMBER DISTRIBUTION TABLE (RANGES)

AMPLE NAME : 101AZ C2-FOR
ILE NAME   : 906009.002
------------------------------------------------------------------------------------------
ATE          : 19/09/1990   | ACQ. RANGE   : 0.5-150        | COUNTS     : 94412
IME          : 10:55        | ACQ. MODE    : SAMPLE         | S.N.F.     : 0.95
ONFIG.       : 1 (0.7 S1)   | ACQ. TIME    : 236 SEC        | S.O.L.     : 3470
ELL TYPE     : MAGNETIC (2) | SAMPLE SIZE  : 4              | CONCENTR.  : 5.0E+05 #/
AMPLE TYPE   : REGULAR      | REQ. CONF.   : 95.00 %(V)     | SOLIDS     : 8.3E-03 %
------------------------------------------------------------------------------------------

  RANGE (microns)          LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%)
    0.0 -    1.0             48.38               48.38                51.58
    1.0 -    2.0             39.19               87.56                12.44
    2.0 -    3.0              6.28               93.84                 6.1[illegible]
    3.0 -    4.0              3.25               97.09                 [illegible]
    4.0 -    5.0              1.78               98.37                 [illegible]
    5.0 -    6.0              0.72               99.59                 [illegible]
    6.0 -    7.0              0.22               99.81                 [illegible]
    7.0 -    8.0              0.07               99.89                 0.13
    8.0 -    9.0              0.04               99.92                 [illegible]
    9.0 -   10.0              0.03               99.95                 [illegible]
   10.0 -   20.0              0.05              100.00                 [illegible]
   20.0 -   30.0              0.00              100.00                 [illegible]
   30.0 -   40.0              0.00              100.00                 [illegible]
   40.0 -   50.0              0.00              100.00                 [illegible]
   50.0 -   60.0              0.00              100.00                 [illegible]
   60.0 -   70.0              0.00              100.00                 0.00
   70.0 -   80.0              0.00              100.00                 0.00
   80.0 -   90.0              0.00              100.00                 0.00
   90.0 -  100.0              0.00              100.00                 0.00
  100.0 -  150.0              0.00              100.00                 0.00
```

Table C.9. (continued) Particle Size Distributions - 101-AZ Core #2 Formated Slurry (Duplicate)

```
      G  A  L  A  I     -     C  I  S  -  1
Computerized  Inspection  System

SAMPLE NAME : HWVP 101AZC2 MELT AN
FILE NAME   : 906202.001
-------------------------------------------------------------------------------
DATE        : 20/09/1990   ACQ. RANGE  : 0.5-150       : COUNTS       [illegible]
TIME        : 09:13      | ACQ. MODE   : SAMPLE        : S.N.F.     : 0.57
CONFIG.     : 1 (0.7 S1) | ACQ. TIME   : 337 SEC       : S.D.L.     : 32%
CELL TYPE   : MAGNETIC (2)| SAMPLE SIZE : 5 (ASORTED)  | CONCENTR.  : [illegible]
SAMPLE TYPE : REGULAR    | REQ. CONF.  : 95.00 %(V)    | SOLIDS     : 3.2E-02
-------------------------------------------------------------------------------
```

PROBABILITY VOLUME DENSITY GRAPH

```
Name:  HWVP 101AZC2 MELT AN                            Median  :  5.61µm
 3.2E-05 cc/ml(100.0%)           Mean(nv):  2.43µm     Mean(vm): 15.34µm
Mode at   50.33 µm               S.D.(nv):  1.48µm     S.D.(vm): 17.86µm
    << SCALE RANGE (µm): ADJUSTED >>                   Conf(vm): 96.42 %
```

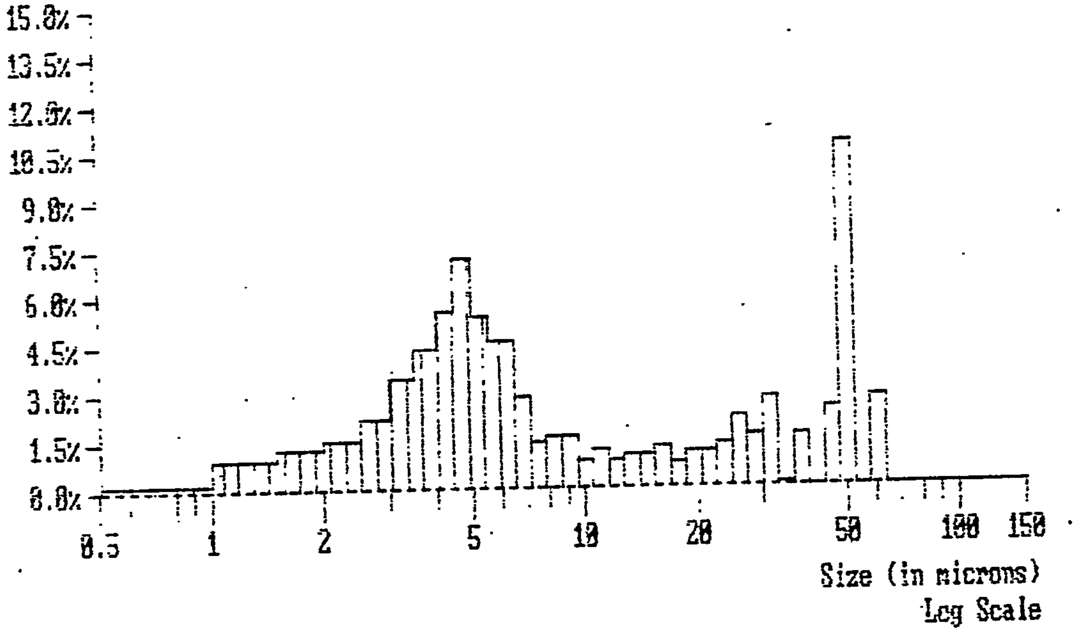


Table C.10. Particle Size Distributions - 101-AZ Core #2 Melter Feed

```
SAMPLE NAME : HWVP 101AZC2 MELT AN
FILE NAME   : 906202.001
------------------------------------------------------------------------------------------------
DATE        : 20/09/1990   | ACQ. RANGE  : 0.5-150        ! COUNTS     : 123922
TIME        : 09:13        | ACQ. MODE   : SAMPLE         ! S.N.F.     : 1.89
DILUTN.     : 1 (0.7 S1)   | ACQ. TIME   : 337 SEC        ! S.D.U.     : 38
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE : 5 (ABORTED)    ! CONCENTR.  : 4.2E+06 #/ml
SAMPLE TYPE : REGULAR      | REC. CONF.  : 95.00 %(V)     ! SOLIDS     : 3.2E-03 %
------------------------------------------------------------------------------------------------
```

PROBABILITY NUMBER DENSITY GRAPH

```
NAME: HWVP 101AZC2 MELT AN                        Median   :  1.09μm
 4.2E+06 #/ml(100.0%)                             Mean(nl):  1.41μm
Mode at    1.25 μm                                S.D.(nl):  1.07μm
   << SCALE RANGE (μm): ADJUSTED >>               Conf(nl):100.00 %
```

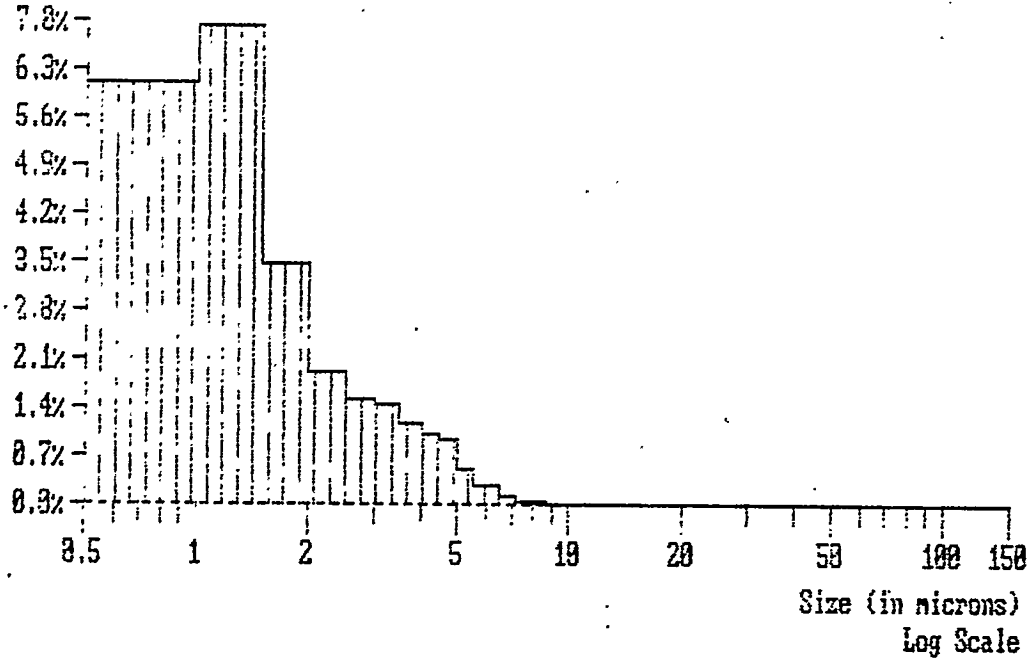
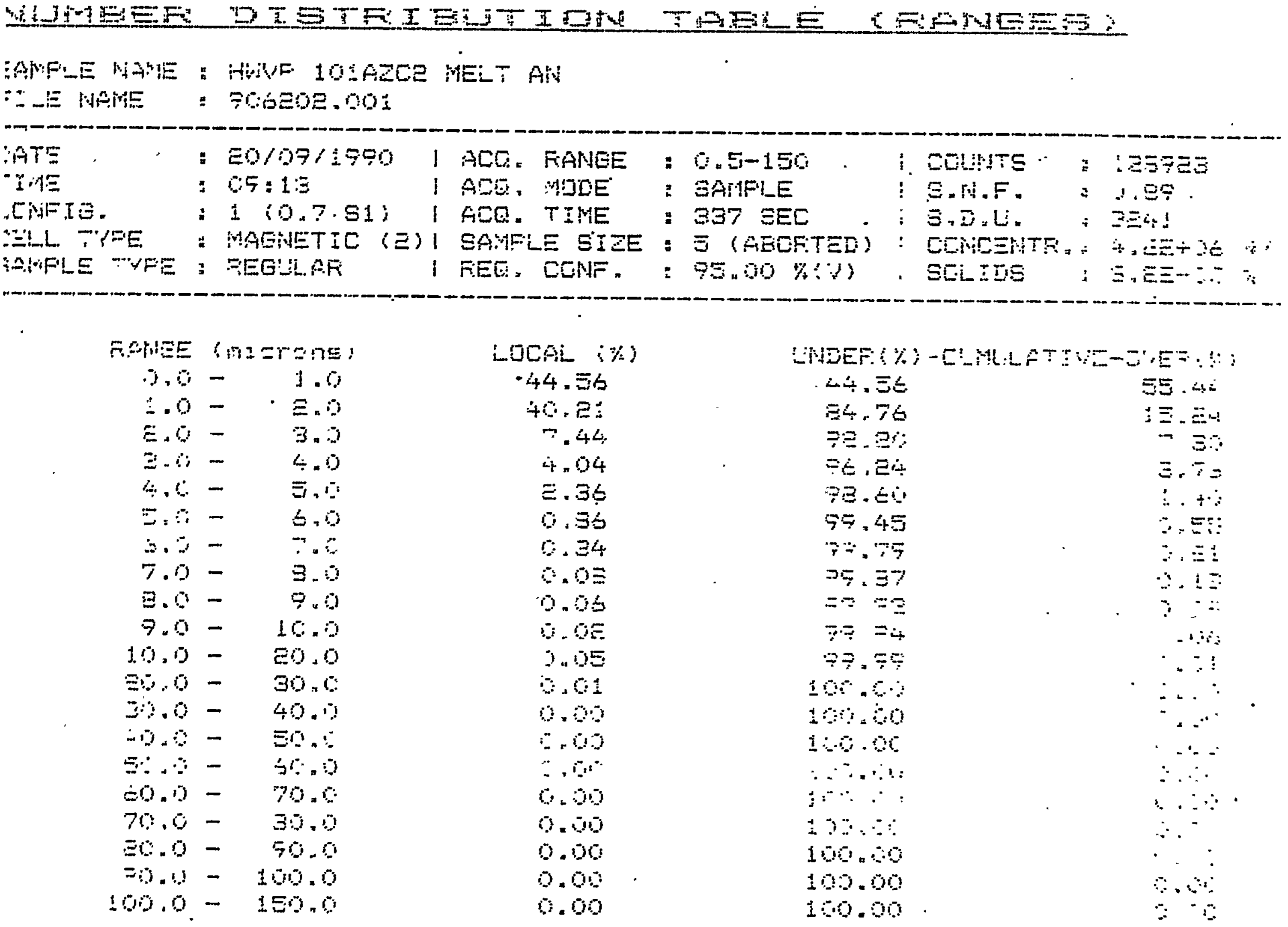


Table C.10. (continued) Particle Size Distributions - 101-AZ Core #2 Melter Feed

```
VOLUME  DISTRIBUTION  TABLE  (RANGES)

SAMPLE NAME : HWVP 101AZC2 MELT AN
FILE NAME   : 906202.001
-------------------------------------------------------------------------------
DATE        : 20/09/1990  | ACQ. RANGE  : 0.5-150      | COUNTS      : 1209[illegible]
TIME        : 09:13       | ACQ. MODE.  : SAMPLE       | S.N.F.      : 0.8[illegible]
CONFIG.     : 1 (0.7 S1)  | ACQ. TIME   : 337 SEC      | S.D.U.      : 224
CELL TYPE   : MAGNETIC (2)| SAMPLE SIZE : 5 (ABORTED)  | CONCENTR.   : [illegible].2E+05
SAMPLE TYPE : REGULAR     | REQ. CONF.  : 95.00 %(V)   | SOLIDS      : 3.2E-03
-------------------------------------------------------------------------------

     RANGE (microns)           LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%)
      0.0 -    1.0               1.31                1.31              98.69
      1.0 -    2.0               7.96                9.26              90.74
      2.0 -    3.0               7.80               17.06              82.94
      3.0 -    4.0              11.78               28.84              71.16
      4.0 -    5.0              14.83               43.67              56.33
      5.0 -    6.0               9.66               53.33              46.67
      6.0 -    7.0               6.25               59.58              40.42
      7.0 -    8.0               2.11               61.69              38.31
      8.0 -    9.0               2.38               64.05              35.95
      9.0 -   10.0               1.16               65.24              34.76
     10.0 -   20.0               7.49               72.73              27.27
     20.0 -   30.0               7.15               79.88              20.12
     30.0 -   40.0               4.20               84.08              15.92
     40.0 -   50.0               7.71               91.79               8.21
     50.0 -   60.0               5.41               97.21               2.79
     60.0 -   70.0               2.79              100.00               0.00
     70.0 -   80.0               0.00              100.00               0.00
     80.0 -   90.0               0.00              100.00               0.00
     90.0 -  100.0               0.00              100.00               0.00
    100.0 -  150.0               0.00              100.00               0.00
```

Table C.10. (continued) Particle Size Distributions - 101-AZ Core #2 Melter Feed



```
NUMBER  DISTRIBUTION  TABLE  (RANGES)

AMPLE NAME : HWVP 101AZC2 MELT AN
ILE NAME   : 906202.001
-------------------------------------------------------------------------------------------
ATE         : 20/09/1990   | ACQ. RANGE  : 0.5-150      | COUNTS    : 125923
IME         : 09:13        | ACQ. MODE   : SAMPLE       | S.N.F.    : .89
CNFIG.      : 1 (0.7-S1)   | ACQ. TIME   : 337 SEC      | S.D.U.    : 3241
ELL TYPE    : MAGNETIC (2) | SAMPLE SIZE : 5 (ABORTED)  | CONCENTR. : 4.22+06 #/
AMPLE TYPE  : REGULAR      | REQ. CONF.  : 95.00 %(V)   | SOLIDS    : 5.22-1  %
-------------------------------------------------------------------------------------------

     RANGE (microns)          LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%)
      0.0 -    1.0             44.56               44.56                 55.44
      1.0 -    2.0             40.21               84.76                 15.24
      2.0 -    3.0              7.44               92.20                  7.80
      3.0 -    4.0              4.04               96.24                  3.76
      4.0 -    5.0              2.36               98.60                  1.40
      5.0 -    6.0              0.36               99.45                  0.55
      6.0 -    7.0              0.34               99.79                  0.21
      7.0 -    8.0              0.08               99.87                  0.13
      8.0 -    9.0              0.06               99.93             [illegible]
      9.0 -   10.0              0.02               99.94                   .06
     10.0 -   20.0              0.05               99.99             [illegible]
     20.0 -   30.0              0.01              100.00             [illegible]
     30.0 -   40.0              0.00              100.00             [illegible]
     40.0 -   50.0              0.00              100.00             [illegible]
     50.0 -   60.0              0.00         [illegible]             [illegible]
     60.0 -   70.0              0.00         [illegible]             [illegible]
     70.0 -   80.0              0.00              100.00             [illegible]
     80.0 -   90.0              0.00              100.00             [illegible]
     90.0 -  100.0              0.00              100.00                  0.00
    100.0 -  150.0              0.00              100.00             [illegible]
```

Table C.10. (continued) Particle Size Distributions - 101-AZ Core #2 Melter Feed

```
G A L A I  -  C I S - 1
Computerized Inspection System

SAMPLE NAME : 93-03763/102AZ-C1-HWVP-FOR-ANL
FILE NAME   : 9303763.001
---------------------------------------------------------------------------------------
DATE        : 03/02/1993   | ACQ. RANGE   : 0.5-150       | COUNTS     : [illegible]
TIME        : 13:15        | ACQ. MODE    : SAMPLE        | S.N.F.     : [illegible]
CONFIG.     : 1 (0 7 81)   | ACQ. TIME    : 208 SEC       | S.D.U.     : [illegible]
CELL TYPE   : MAGNETIC-(2) | SAMPLE SIZE  : 4             | CONCENTR.  : [illegible]
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V)    | SOLIDS     : [illegible]
---------------------------------------------------------------------------------------

                 PROBABILITY VOLUME DENSITY GRAPH
Name: 93-03763/102AZ-C1-HWVP-FOR-ANL                       Median   : 54.74µm
 4.1E-05 cc/ml(100.0%)           Mean(nv):  2.37µm          Mean(vm): 47.36µm
Mode at   80.95 µm               S.D.(nv):  1.49µm          S.D.(vm): 40.24µm
   << SCALE RANGE (µm): ADJUSTED >>                         Conf(vm): 98.92 %
```

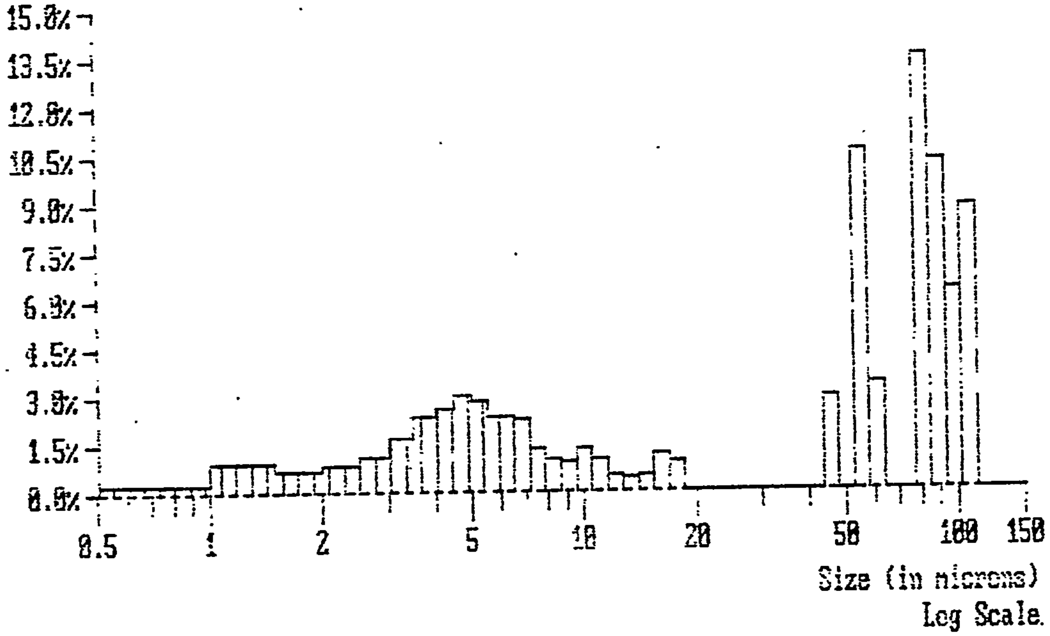


Table C.11. Particle Size Distributions - 102-AZ Core #1 Formated Slurry

```
AMPLE NAME  : 93-03763/102AZ-C1-HWVP-FOR-ANL
ILE NAME    : 9303763.001
---------------------------------------------------------------------------------------------
ATE         : 03/0[illegible]      | ACQ. RANGE   : 0.5-150      | COUNTS      : 6004[illegible]
IME         : 15:15               | ACQ. START   : SAMPLE       | S.N.F.      : [illegible]
ONFIG.      : 1 (0.7 S1)          | ACQ. TIME    : 208 SEC      | S.D.U.      : [illegible]
ELL TYPE    : MAGNETIC (2)        | SAMPLE SIZE  : 4            | CONCENTR.   : 6.0E+[illegible]
AMPLE TYPE  : REGULAR             | REG. CONF.   : 95.00 %(V)   | SOLIDS      : 4.[illegible]E-03 %
---------------------------------------------------------------------------------------------
```

PROBABILITY NUMBER DENSITY GRAPH

```
ame: 93-03763/102AZ-C1-HWVP-FOR-ANL              Median  :  0.92µm
6.0E+06 #/ml(100.0%)                              Mean(nl):  1.14µm
ode at    0.75 µm                                 S.D.(nl):  0.85µm
  (( SCALE RANGE (µm): ADJUSTED ))                Conf(nl):100.00 %
```

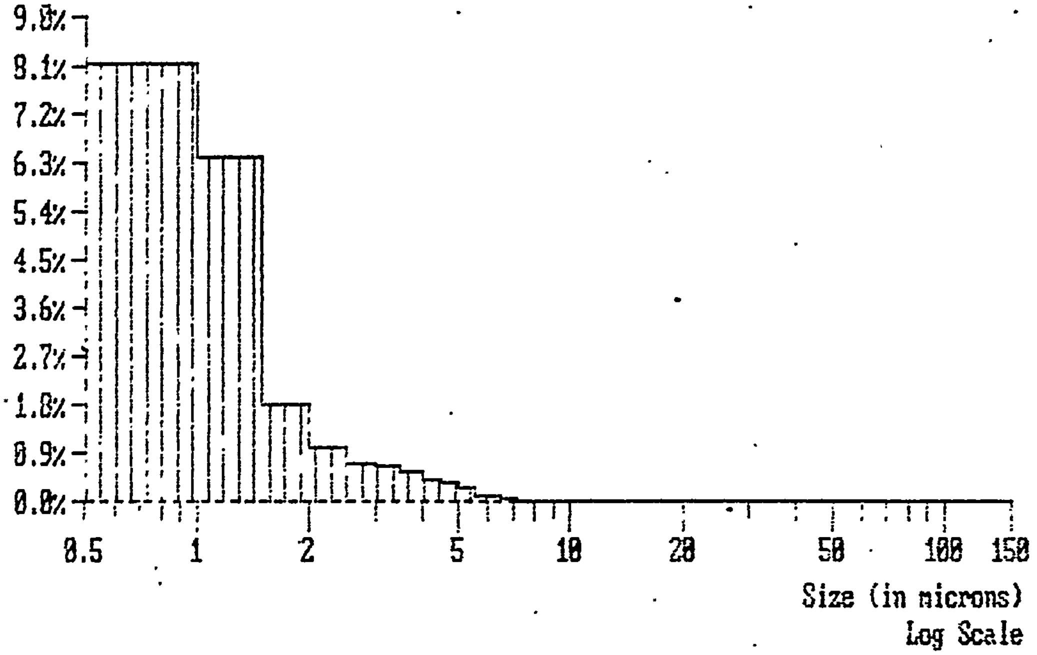


Table C.11. (continued) Particle Size Distributions - 102-AZ Core #1 Formated Slurry

```
VOLUME  DISTRIBUTION  TABLE  (RANGES)

SAMPLE NAME  : 93-03763/102AZ-C1-HWVP-FOR-ANL
FILE NAME    : 9303763.001
------------------------------------------------------------------------------------
DATE         : 03/02/1992  ! ACQ. RANGE   : 0.5-150        | COUNTS      : [illegible]
TIME         : 15:15       ! ACQ. MODE    : SAMPLE         | S.N.F.      : [illegible]
CONFIG.      : 1 (0.7 31)  ! ACQ. TIME    : 208 SEC        | S.D.U.      : [illegible]
CELL TYPE    : MAGNETIC (2)! SAMPLE SIZE  : 4              | CONCENTR.   : [illegible]
SAMPLE TYPE  : REGULAR     ! REQ. CONF.   : 95.00 %(V)       SOLIDS      : [illegible]
------------------------------------------------------------------------------------

   RANGE (microns)          LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(
     0.0 -    1.0              1.90                 1.90              98.10
     1.0 -    2.0              6.26                 8.16              91.84
     2.0 -    3.0              4.22                12.38              87.62
     3.0 -    4.0              6.12                18.50              81.50
     4.0 -    5.0              6.59                25.09              [illegible]
     5.0 -    6.0              5.16                30.25              69.75
     6.0 -    7.0              3.85                34.12              65.88
     7.0 -    8.0              1.95                36.07              63.93
     8.0 -    9.0              1.36                37.43              62.57
     9.0 -   10.0              1.21                38.65              61.35
    10.0 -   20.0              5.39                44.04              55.96
    20.0 -   30.0              0.00                44.04              55.96
    30.0 -   40.0              0.00                44.04              55.96
    40.0 -   50.0              2.98                47.02              52.98
    50.0 -   60.0             13.95                60.97              39.03
    60.0 -   70.0              0.00                60.97              39.03
    70.0 -   80.0              9.06                70.03              29.97
    80.0 -   90.0             14.80                84.83              15.17
    90.0 -  100.0              6.33                91.16               8.8
   100.0 -  150.0              8.84               100.00               0.00
```

Table C.11. (continued) Particle Size Distributions - 102-AZ Core #1 Formated Slurry

```
NUMBER DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : 93-03763/102AZ-C1-HWVP-FOR-ANL
FILE NAME   : 9303763.001
------------------------------------------------------------------------------------------
DATE        : 03/02/1993   | ACQ. RANGE  : 0.5-150       | COUNTS     : 60542
TIME        : 15:15        | ACQ. MODE   : SAMPLE        | S.N.F.     : 0.89
CONFIG.     : 1 (0.7 Si)   | ACQ. TIME   : 208 SEC       | S.D.U.     : 3159
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE : 4             | CONCENTR.  : 6.0E+05 #/
SAMPLE TYPE : REGULAR      | REQ. CONF.  : 95.00 %(V)    | SOLIDS     : 4.1E-03 %
------------------------------------------------------------------------------------------

    RANGE (microns)           LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%)
      0.0 -    1.0              59.72               59.72                40.28
      1.0 -    2.0              32.83               92.60                 7.40
      2.0 -    3.0               3.76               96.36                 3.64
      3.0 -    4.0               1.91               98.27                 1.73
      4.0 -    5.0               0.97               99.24                 0.76
      5.0 -    6.0               0.42               99.67                 0.33
      6.0 -    7.0               0.19               99.85           [illegible]
      7.0 -    8.0               0.06               99.92                 0.08
      8.0 -    9.0               0.03               99.95                 0.05
      9.0 -   10.0               0.02               99.96                 0.04
     10.0 -   20.0               0.03              100.00                 0.00
     20.0 -   30.0               0.00              100.00                 0.00
     30.0 -   40.0               0.00              100.00                 0.00
     40.0 -   50.0               0.00              100.00                 0.00
     50.0 -   60.0               0.00              100.00                 0.00
     60.0 -   70.0               0.00              100.00                 0.00
     70.0 -   80.0               0.00              100.00                 0.00
     80.0 -   90.0               0.00              100.00                 0.00
     90.0 -  100.0               0.00              100.00                 0.00
    100.0 -  150.0               0.00              100.00                 0.00
```

Table C.11. (continued) Particle Size Distributions - 102-AZ Core #1 Formated Slurry

```
G A L A I  —  C I S - 1
Computerized Inspection System

SAMPLE NAME : CR3-FOR
FILE NAME   : 905406.001
------------------------------------------------------------------------------------------
DATE        : 16/08/1990   | ACQ. RANGE   : 0.5-150       | COUNTS     : [illegible]
TIME        : 14:30        | ACQ. MODE    : SAMPLE        | S N.F.     : [illegible]
CONFIG.       1 (0.7 81)   | ACQ. TIME    : 137 SEC       | S.D.J.     : [illegible]
CELL TYPE   : MAGNETIC (2) | SAMPLE SIZE  : 3             | CONCENTR.  : [illegible]
SAMPLE TYPE : REGULAR      | REQ. CONF.   : 95.00 %(V)    | SOLIDS     : 4.2E-03
------------------------------------------------------------------------------------------
```

PROBABILITY VOLUME DENSITY GRAPH

```
Name: CR3-FOR                                              Median   : 28.91µm
 4.8E-05 cc/ml(100.0%)            Mean(nv):  3.42µm        Mean(vm): 25.57µm
Mode at   34.41 µm                S.D.(nv):  2.53µm        S.D.(vm): 13.27µm
   << SCALE RANGE (µm): ADJUSTED >>                        Conf(vm): 97.77 %
```

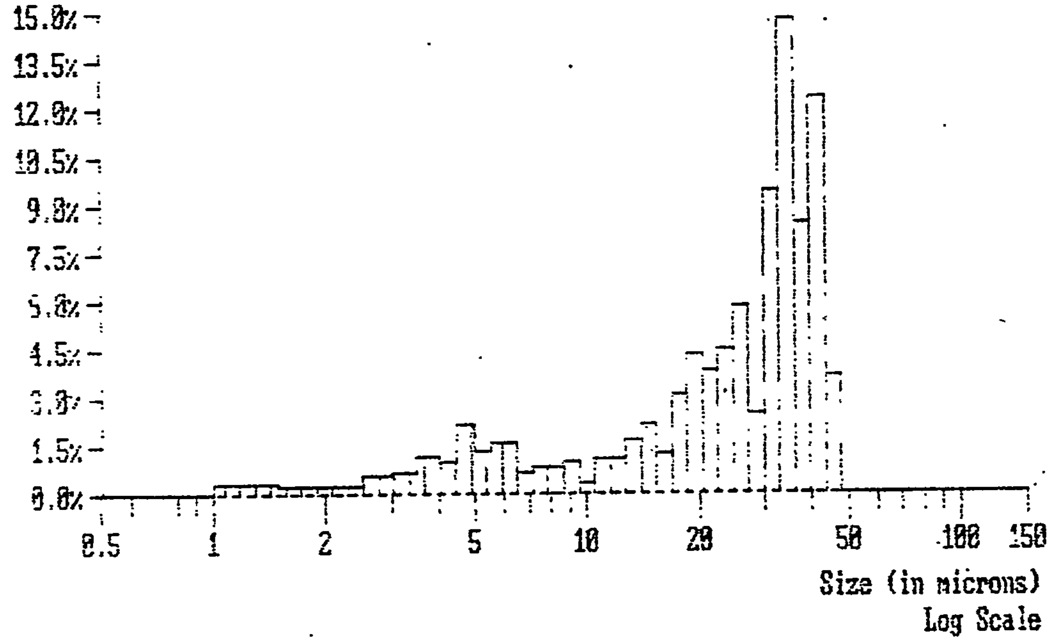


Table C.12. Particle Size Distributions - NCAW Simulant Formated Slurry

```
SAMPLE NAME : CR3-FOR
FILE NAME   : 905406.001
--------------------------------------------------------------------------------------------
DATE        : 16/08/1990    | ACQ. RANGE   : 0.5-150      | COUNTS    : 30168
TIME        : 14:30         | ACQ. MODE    : SAMPLE       | S.N.F.    : 0.97
CONFIG.     : 1 (0.7 S1)    | ACQ. TIME    : 137 SEC      | S.D.U.    : 1591
CELL TYPE   : MAGNETIC (2)  | SAMPLE SIZE  : 3            | CONCENTR. : 8.7E+06 #/
SAMPLE TYPE : REGULAR       | REQ. CONF.   : 95.00 %(V)   | SOLIDS    : 4.8E-03 %
--------------------------------------------------------------------------------------------
```

PROBABILITY NUMBER DENSITY GRAPH

```
Name:  CR3-FOR                                    Median   :  0.98µm
 2.3E+06 #/ml(100.0%)                             Mean(nl):  1.30µm
Mode at    0.75 µm                                S.D.(nl):  1.30µm
   << SCALE RANGE (µm): ADJUSTED >>               Conf(nl):100.00 %
```

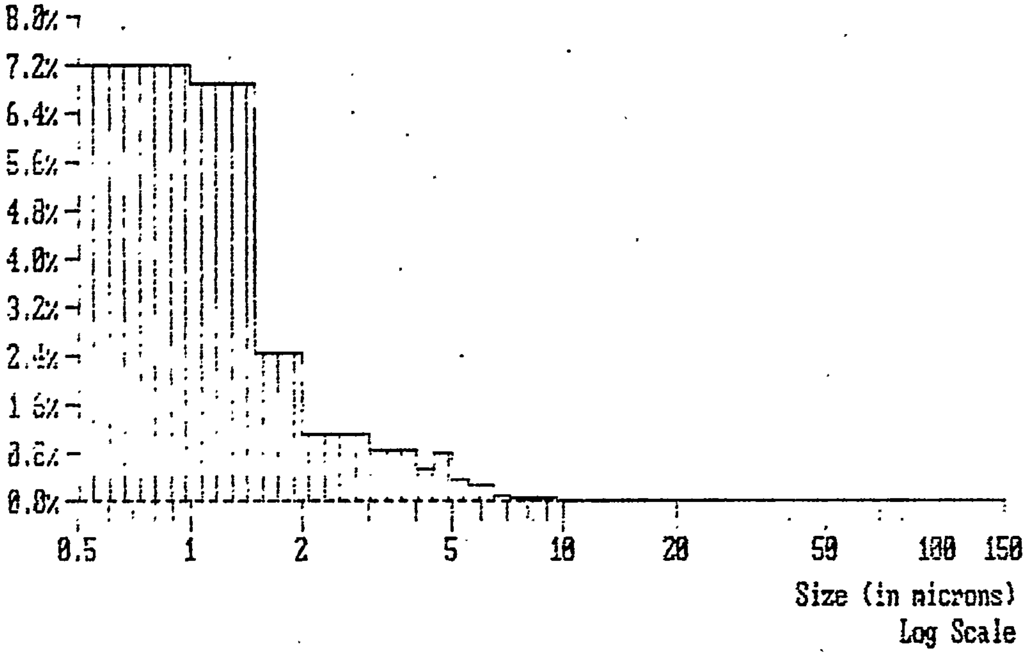


Table C.12. (continued) Particle Size Distributions - NCAW Simulant Formated Slurry

```
VOLUME DISTRIBUTION TABLE (RANGES)

SAMPLE NAME : CRS-FOR
FILE NAME   : 705406.001
------------------------------------------------------------------------------------------
DATE          : 16/08/1990   | ACQ. RANGE   : 0.5-150       : COUNTS      : 301e3
TIME          : 14:30        | ACQ. MODE    : SAMPLE        | S.N.F.      : 0.97
CONFIG.       : 1 (0.7 51).  | ACQ. TIME    : 137 SEC       | S.D.U.      : 1591
CELL TYPE     : MAGNETIC (2) | SAMPLE SIZE  : 3             | CONCENTR.   : 2.3E+[illegible]
SAMPLE TYPE   : REGULAR      | REQ. CONF.   : 95.00 %(V)    : SOLIDS      : [illegible]
------------------------------------------------------------------------------------------

      RANGE (microns)          LOCAL (%)          UNDER(%)-CUMULATIVE-OVER(%)
        0.0 -    1.0              0.55                0.55                  99.45
        1.0 -    2.0              2.43                2.98                  97.02
        2.0 -    3.0              1.87                4.83                  [illegible]
        3.0 -    4.0              2.82                7.67                  [illegible]
        4.0 -    5.0              3.79               11.46                  [illegible]
        5.0 -    6.0              3.14               14.60                  [illegible]
        6.0 -    7.0              1.61               16.22                  [illegible]
        7.0 -    8.0              1.19               17.41                  [illegible]
        8.0 -    9.0              1.38               18.79                  [illegible]
        9.0 -   10.0              0.60               19.39                  [illegible]
       10.0 -   20.0             12.78               32.17                  [illegible]
       20.0 -   30.0             19.75               51.92                  [illegible]
       30.0 -   40.0             34.23               86.15                  13.85
       40.0 -   50.0             13.85              100.00                  [illegible]
       50.0 -   60.0              0.00              100.00                  [illegible]
       60.0 -   70.0              0.00              100.00                  [illegible]
       70.0 -   80.0              0.00              100.00                  [illegible]
       80.0 -   90.0              0.00              100.00                  [illegible]
       90.0 -  100.0              0.00              100.00                  [illegible]
      100.0 -  150.0              0.00              100.00                  [illegible]
```

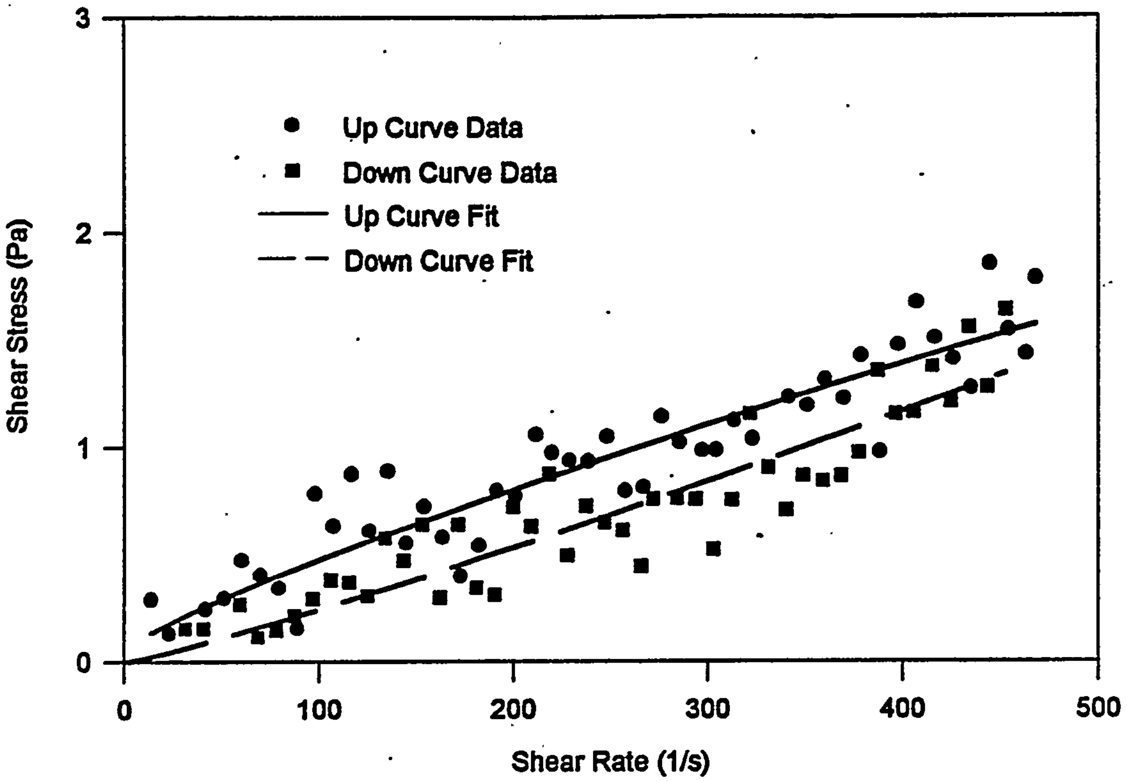
Table C.12. (continued) Particle Size Distributions - NCAW Simulant Formated Slurry

```
NUMBER  DISTRIBUTION  TABLE  (RANGES)

AMPLE NAME : CR3-FOR
ILE NAME   : 905406.001
----------------------------------------------------------------------------------------------
ATE         : 16/08/1990  ! ACQ. RANGE   : 0.5-150       ! COUNTS     : 30142
IME         : 14:30       ! ACQ. MODE    : SAMPLE        : S.N.F.     : ..97
ONFIG.      : 1 (0.7 S1)  ! ACQ. TIME    : 137 SEC       ! S.D.J.     : 1041
ELL TYPE    : MAGNETIC (2)! SAMPLE SIZE  : 3             ! CONCENT.   : 5.3E+06 #/
AMPLE TYPE  : REGULAR     ! REQ. CONF.   : 95.00 %(V)    : SOLIDS     : 4.6E-03 %
----------------------------------------------------------------------------------------------

       RANGE (microns)           LOCAL (%)            UNDER(%)-CUMULATIVE-OVER(%)
         0.0 -     1.0             52.61                 52.61                 47.39
         1.0 -     2.0             34.86                 87.46                 12.54
         2.0 -     3.0              4.79                 94.25                  5.75
         3.0 -     4.0              2.63                 96.88                  3.12
         4.0 -     5.0              1.61                 98.49                  1.51
         5.0 -     6.0              0.75                 99.25                  0.75
         6.0 -     7.0              0.25                 99.49                  0.51
         7.0 -     8.0              0.12                 99.61                  0.39
         8.0 -     9.0              0.09                 99.70                  0.30
         9.0 -    10.0              0.03                 99.73                  0.27
        10.0 -    20.0              0.17                 99.90                  0.10
        20.0 -    30.0              0.06                 99.96                  0.04
        30.0 -    40.0              0.03                 99.99                  0.01
        40.0 -    50.0              0.01                100.00                  0.00
        50.0 -    60.0              0.00                100.00                  0.00
        60.0 -    70.0              0.00                100.00                  0.00
        70.0 -    80.0              0.00                100.00                  0.00
        80.0 -    90.0              0.00                100.00                  0.00
        90.0 -   100.0              0.00                100.00                  0.00
       100.0 -   150.0              0.00                100.00                  0.00
```

Table C.12. (continued) Particle Size Distributions - NCAW Simulant Formated Slurry

APPENDIX D

Rheological Data



	Average	Reproducibility
Density [a]	1.10	<0.5%
Wt-Fraction Solid [a]	0.159	1%
Wt-Fraction Oxide [a]	0.108	1%
g TO/L [a]	118	
Vol-Fraction Settled Solid [a]	0.568	10%
Density Centrifuged Solid [a]	1.44	6%
Density Centrifuged Supernate [a]	1.04	1%
Vol-Fraction Centrifuged Solid [a]	0.214	3%
Wt-Fraction Centrifuged Solid [a]	0.276	3%
Wt-Fraction Dissolved Solid [a]	0.048	3%
Mean Particle Size Volume Density [b]	9.01	9.99
Mean Particle Size Number Density [b]	1.27	0.90

Notes: a. Refer to Table C.1. for more information
b. Complete particle size distribution information is provided in Appendix C

Figure D.1. Shear Stress versus Shear Rate of 101-AZ Core #1 Formated Slurry (Run 1) with Yield-Pseudoplastic Model Fits and Corresponding Physical Properties

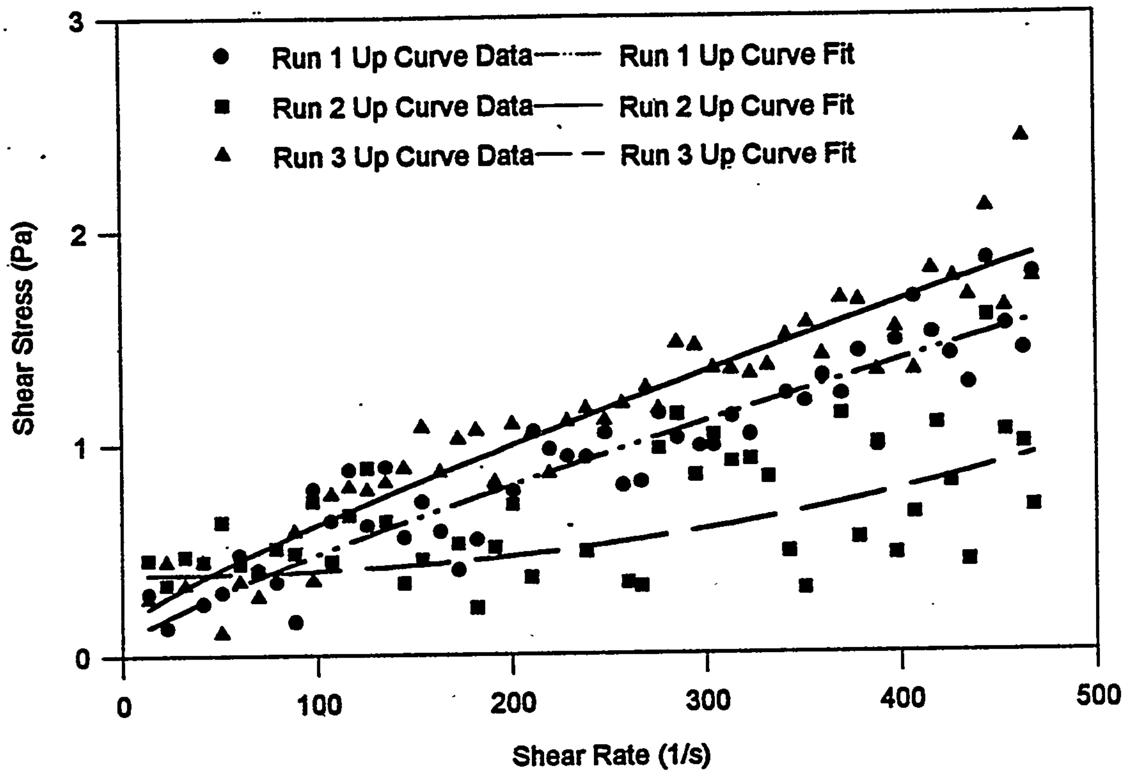


Figure D.2. Shear Stress versus Shear Rate of 101-AZ Core #1 Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits (Down Curve Data omitted for clarity)

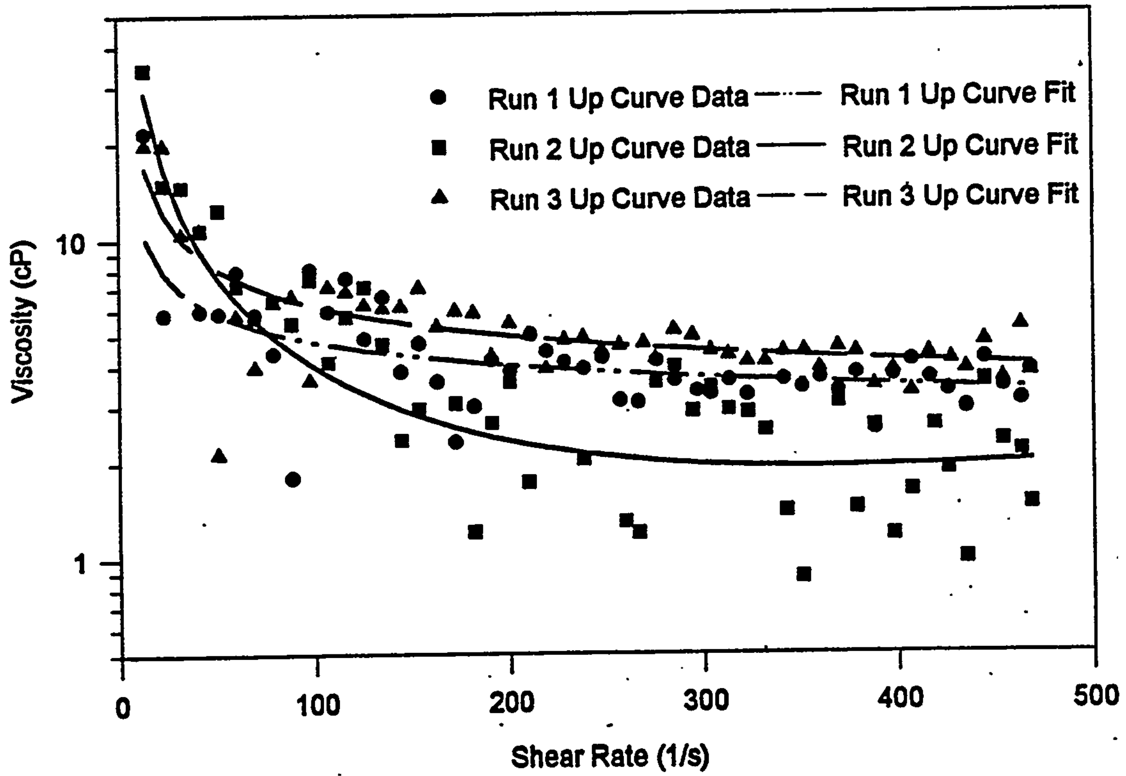


Figure D.3. Viscosity versus Shear Rate of 101-AZ Core #1 Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits (Down Curve Data omitted for clarity)

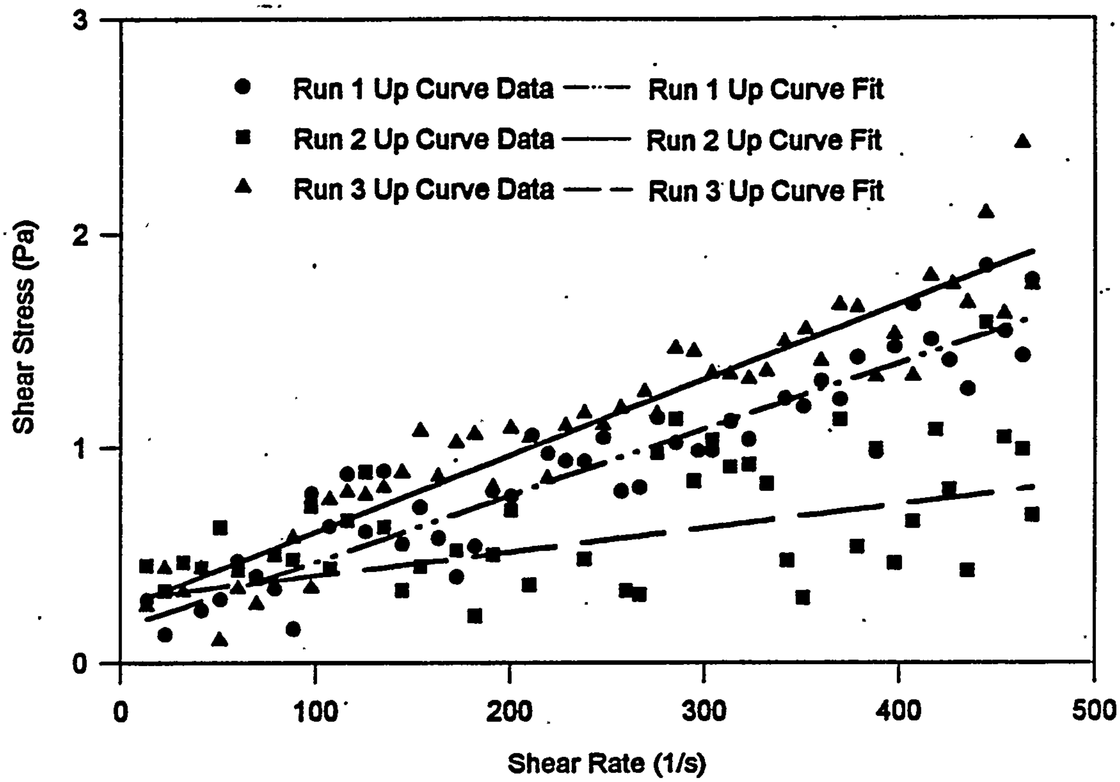


Figure D.4. Shear Stress versus Shear Rate of 101-AZ Core #1 Formated Slurry (All Runs) with Bingham Curve Fit (Down Curve Data Omitted for Clarity)

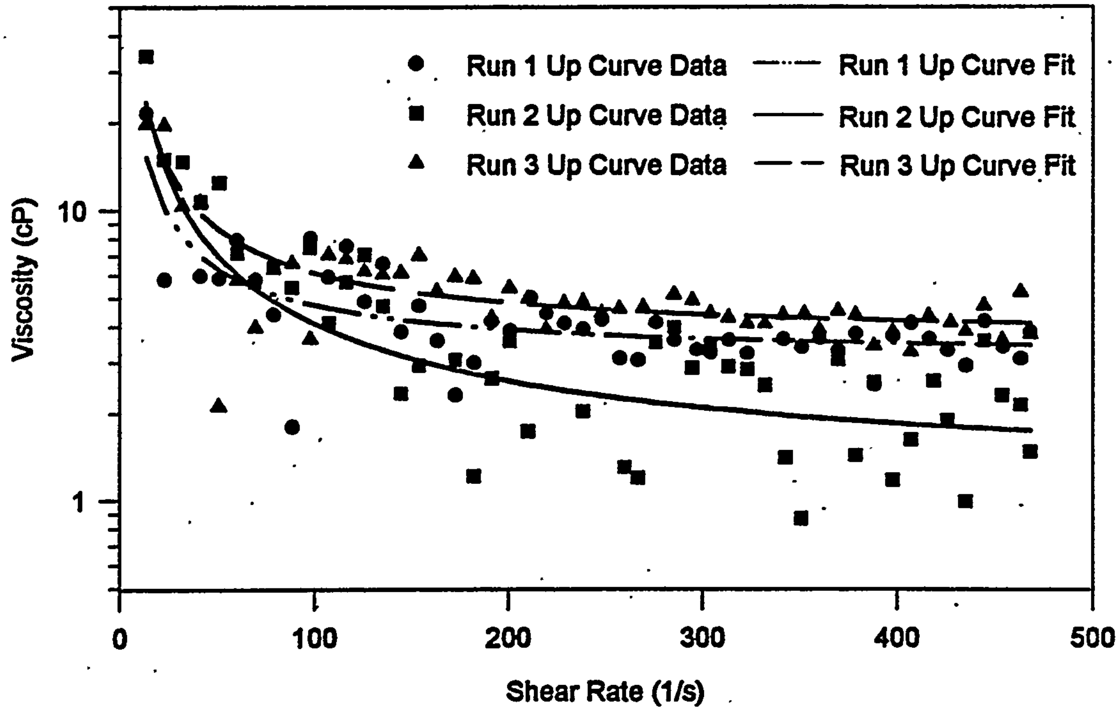
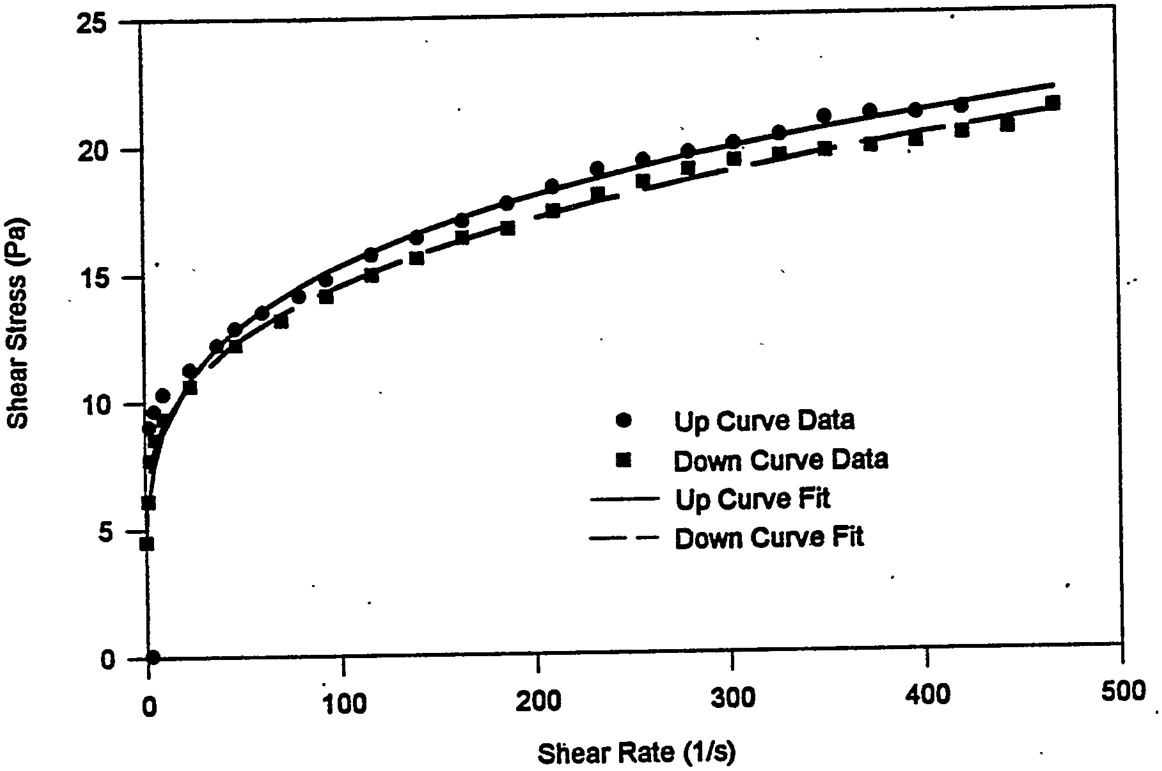


Figure D.5. Viscosity versus Shear Rate of 101-AZ Core #1 Formated Slurry (All Runs) with Bingham Curve Fit (Down Curve Data Omitted for Clarity)



	Average	Reproducibility
Density [a]	1.47	3%
Wt-Fraction Solid [a]	0.47	
Wt-Fraction Oxide [a]	0.41	
g TO/L [a]	600	
Vol-Fraction Settled Solid [a]	0.84	2%
Density Centrifuged Solid [a]	1.74	1%
Density Centrifuged Supernate [a]	1.09	<0.5%
Vol-Fraction Centrifuged Solid [a]	0.635	5%
Wt-Fraction Centrifuged Solid [a]	0.739	4%
Wt-Fraction Dissolved Solid [a]	0.068	2%
Mean Particle Size Volume Density [b]	4.26	2.48
Mean Particle Size Number Density [b]	1.22	0.79

Notes: a. Refer to Table C.1. for more information
b. Complete particle size distribution information is provided in Appendix C

Figure D.6. Shear Stress versus Shear Rate of 101-AZ Core #1 Melter Feed Slurry (Run 1) with Yield-Pseudoplastic Model Curve Fits and Corresponding Physical Properties

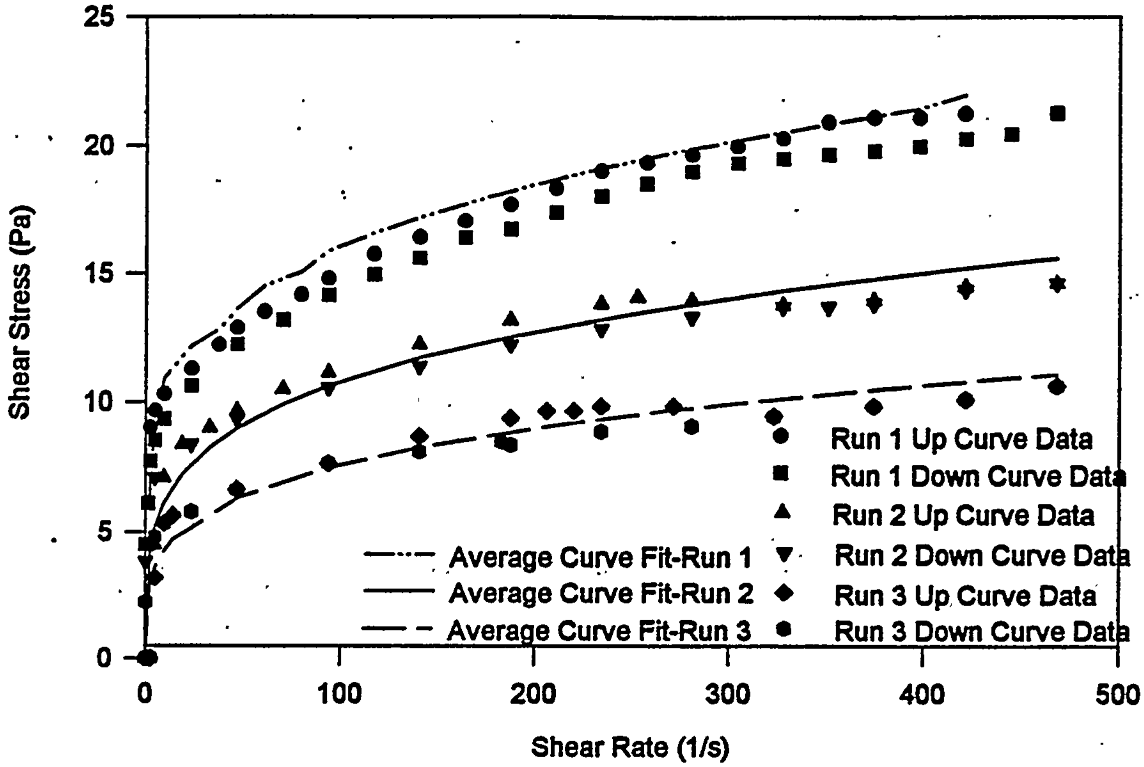


Figure D.7. Shear Stress versus Shear Rate of 101-AZ Core #1 Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

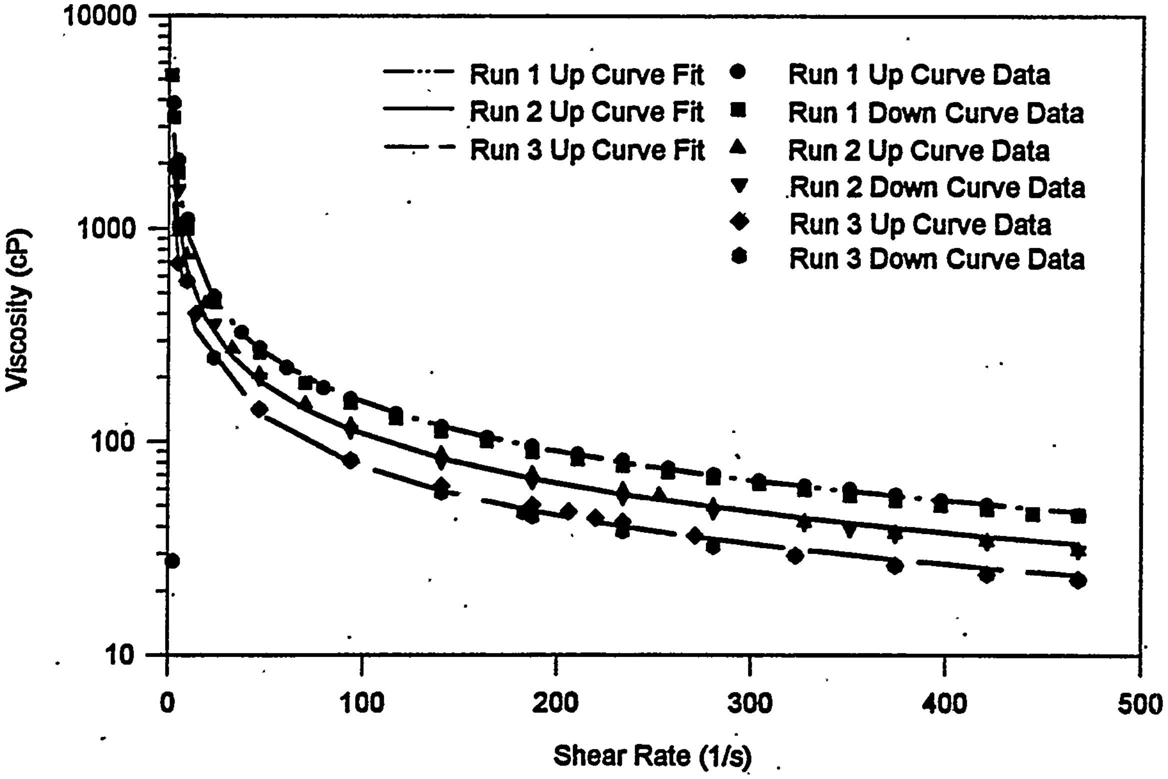


Figure D.8. Viscosity versus Shear Rate of 101-AZ Core #1 Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

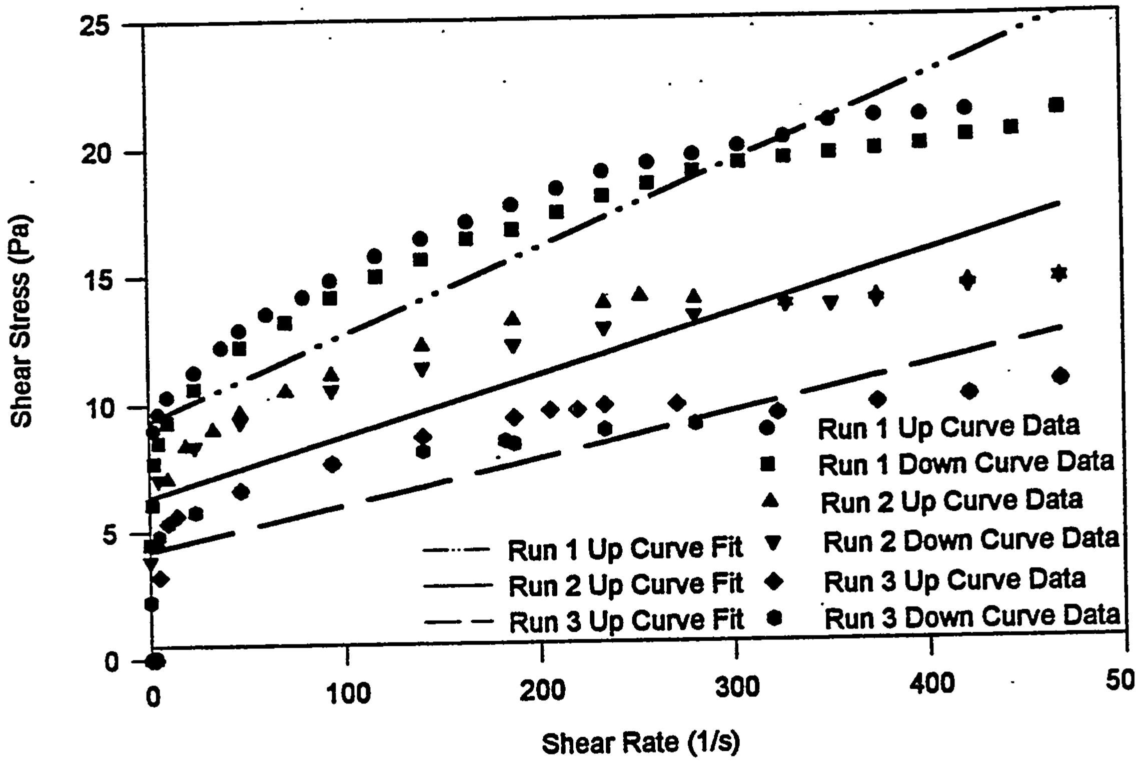


Figure D.9. Shear Stress versus Shear Rate of 101-AZ Core #1 Melter Feed Slurry (All Runs) with Bingham Curve Fits

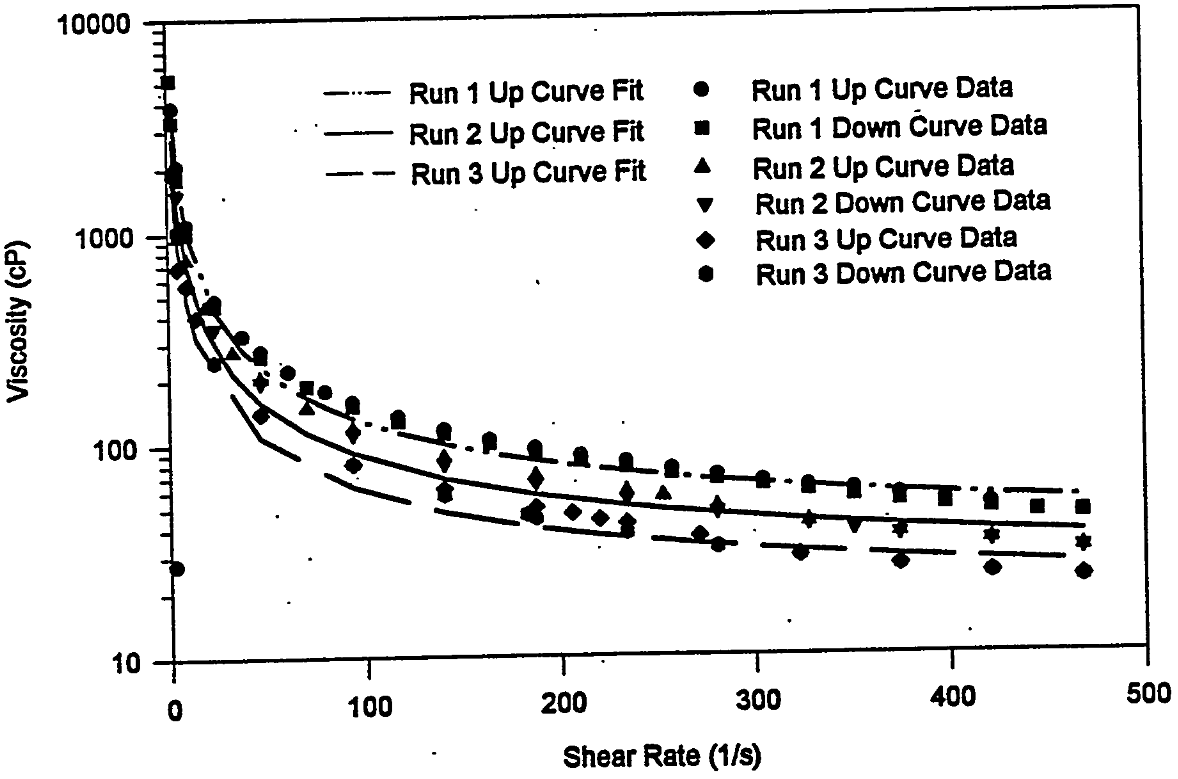
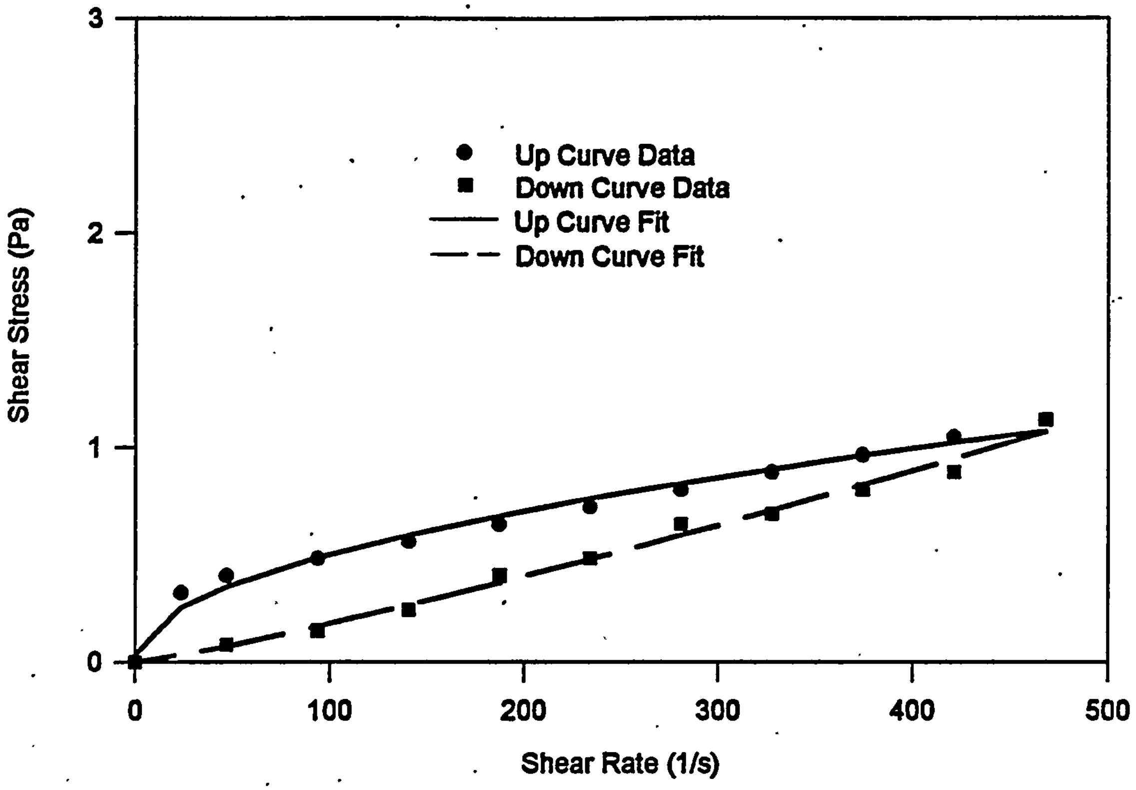


Figure D.10. Viscosity versus Shear Rate of 101-AZ Core #1 Melter Feed Slurry (All Runs) with Bingham Curve Fits



	Average	Reproducibility
Density [a]	1.10	<0.05%
Wt-Fraction Solid [a]	0.122	6%
Wt-Fraction Oxide [a]	0.092	15%
g TO/L [a]	101	
Vol-Fraction Settled Solid [a]	0.374	4%
Density Centrifuged Solid [a]	1.5	<0.05%
Density Centrifuged Supernate [a]	1.01	1%
Vol-Fraction Centrifuged Solid [a]	0.185	9%
Wt-Fraction Centrifuged Solid [a]	0.254	9%
Wt-Fraction Dissolved Solid [a]	0.0252	2%
Mean Particle Size Volume Density [b]	5.37	3.79
Mean Particle Size Number Density [b]	1.42	1.01

Notes: a. Please refer to Table C.2. for more information
b. Complete particle size distribution information is provided in Appendix C

Figure D.11. Shear Stress versus Shear Rate of 101-AZ Core #2 Formated Slurry (Run 1) with Yield-Pseudoplastic Model Curve Fits and Corresponding Physical Properties

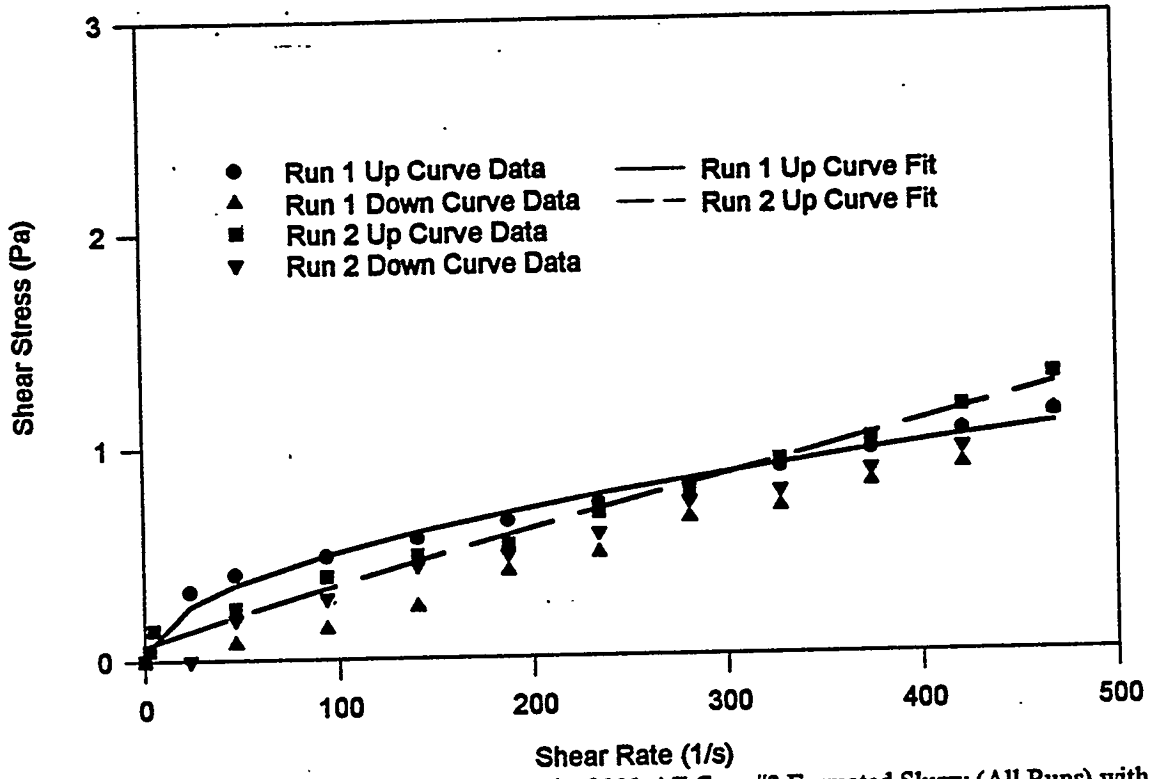


Figure D.12. Shear Stress versus Shear Rate of 101-AZ Core #2 Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

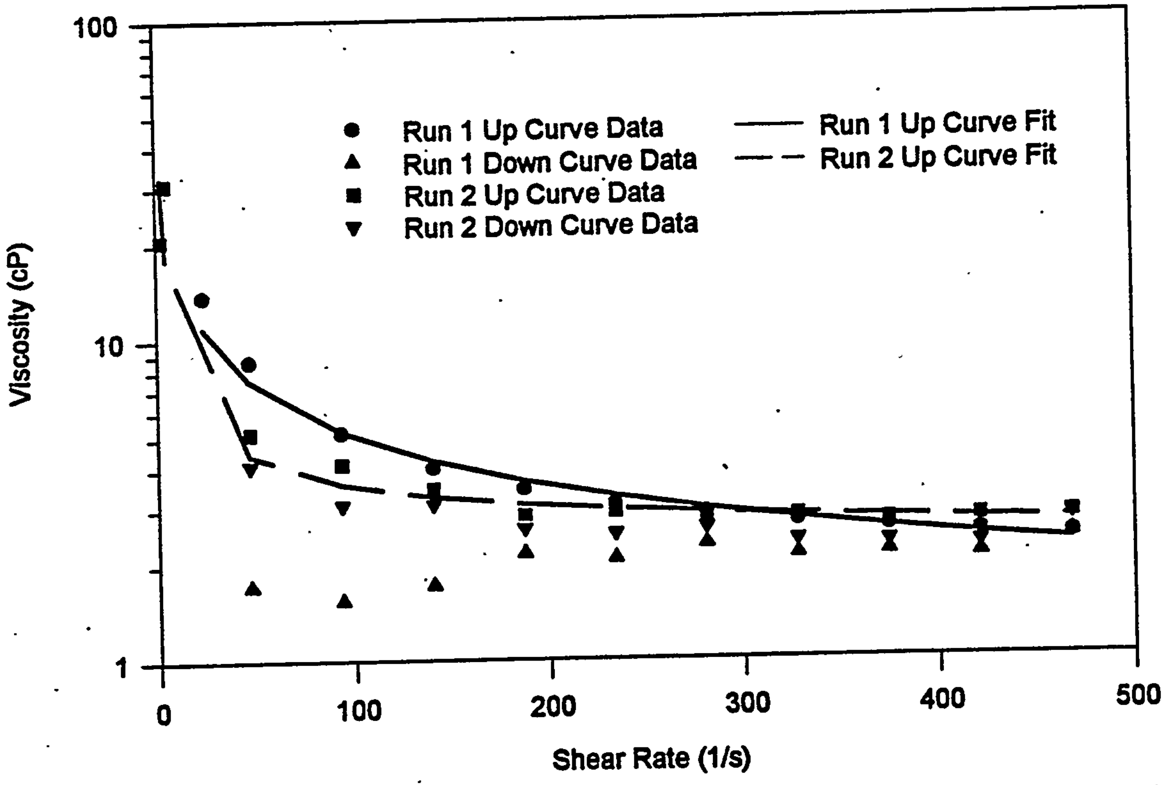


Figure D.13. Viscosity versus Shear Rate of 101-AZ Core #2 Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

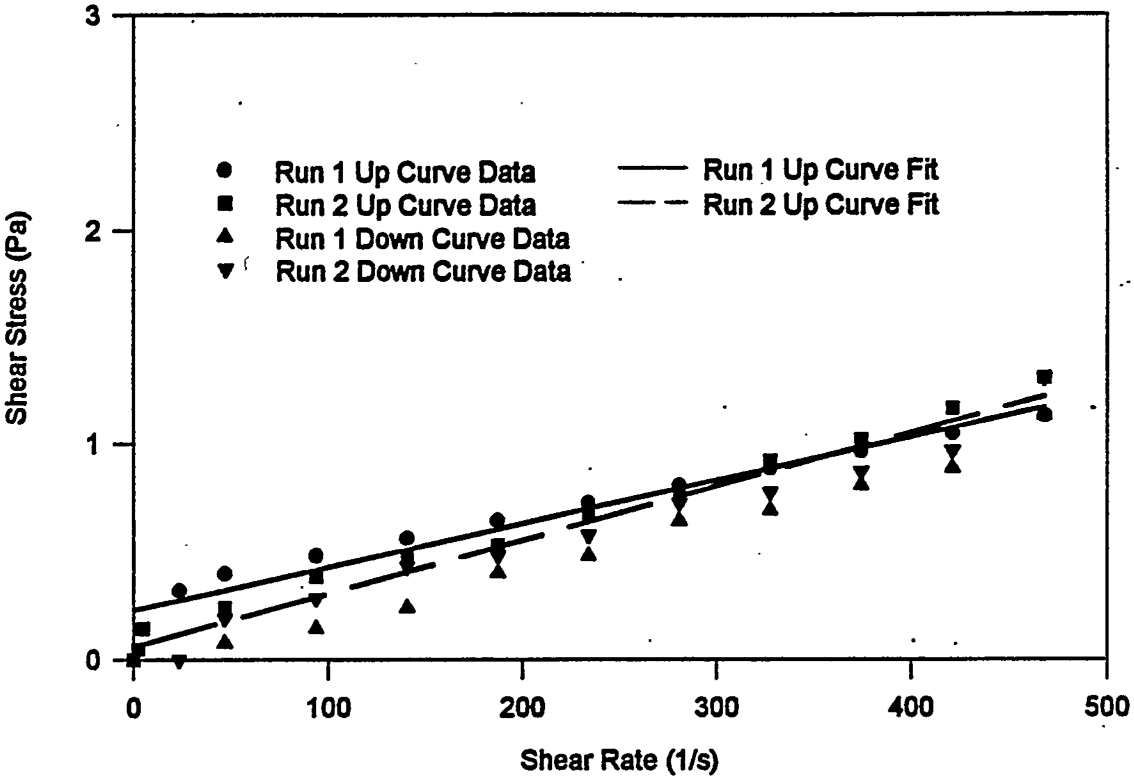


Figure D.14. Shear Stress versus Shear Rate of 101-AZ Core #2 Formated Slurry (All Runs) with Bingham Curve Fits

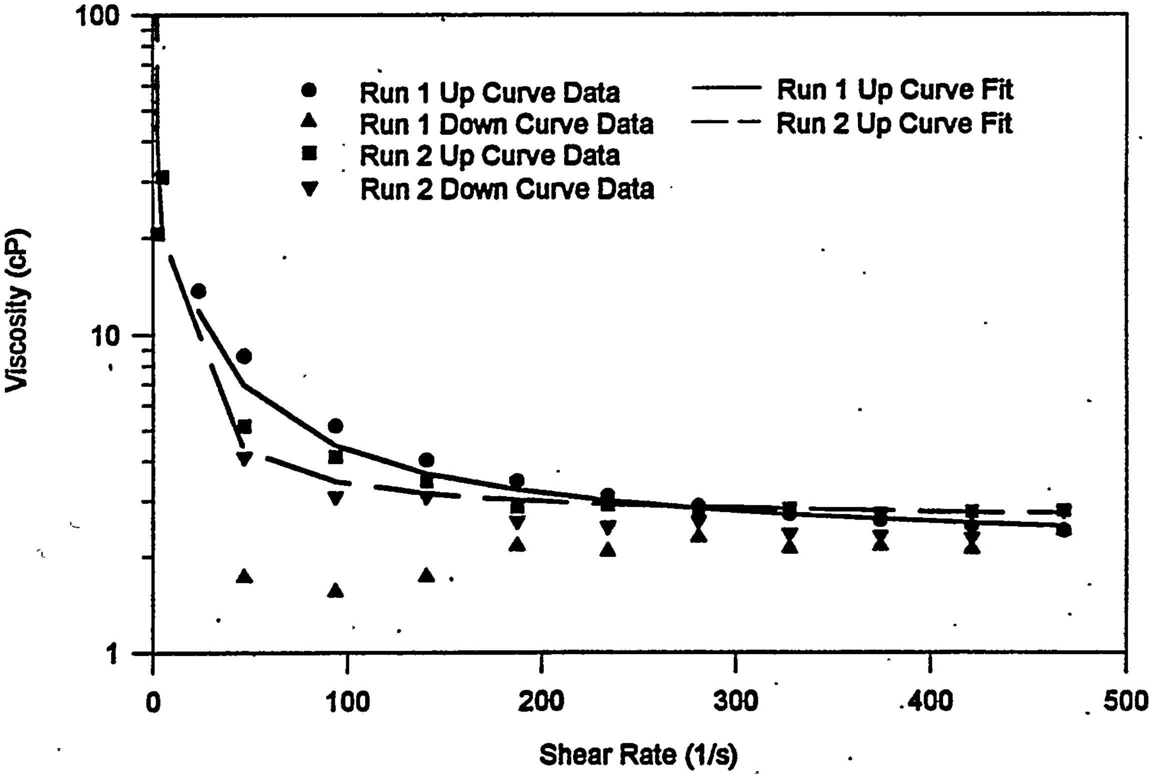
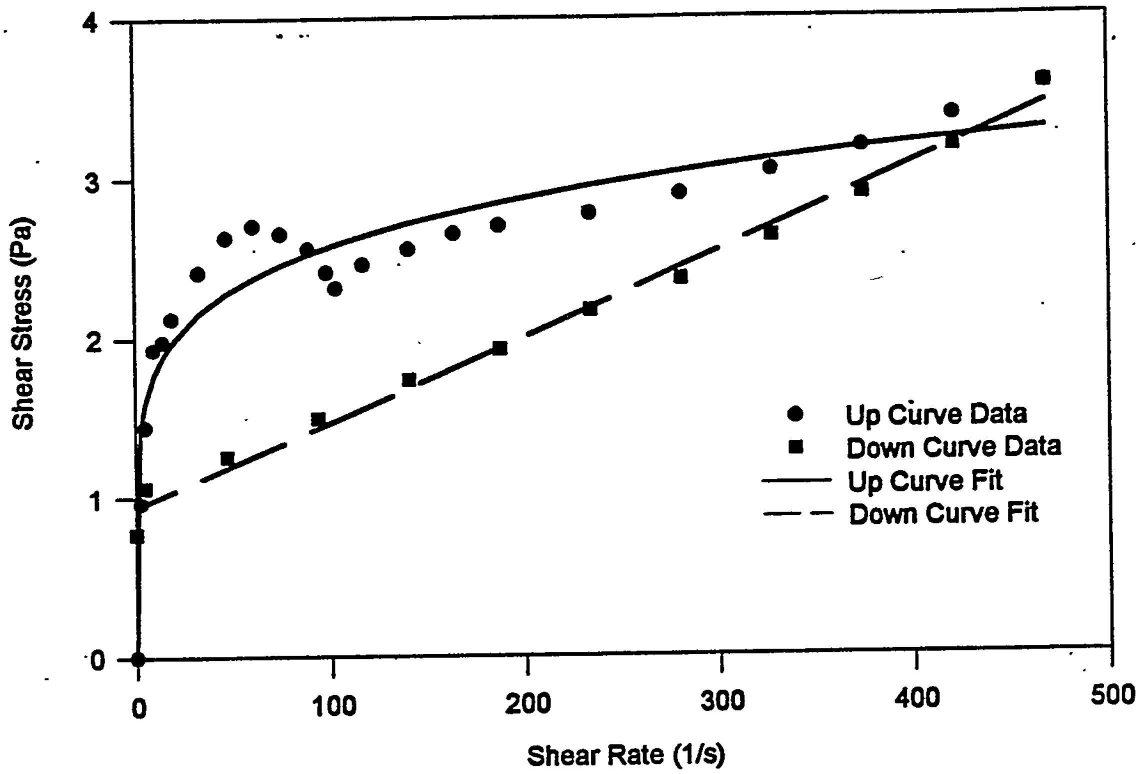


Figure D.15. Viscosity versus Shear Rate of 101-AZ Core #2 Formated Slurry (All Runs) with Bingham Curve Fits



	Average	Reproducibility
Density [a]	1.20	1%
Wt-Fraction Solid [a]	0.429	2%
Wt-Fraction Oxide [a]	0.399	2%
g TO/L [a]	479	
Vol-Fraction Settled Solid [a]	0.87	<0.05%
Density Centrifuged Solid [a]	1.64	<0.05%
Density Centrifuged Supernate [a]	1.04	<0.05%
Vol-Fraction Centrifuged Solid [a]	0.553	1%
Wt-Fraction Centrifuged Solid [a]	0.667	<0.05%
Wt-Fraction Dissolved Solid [a]	0.0202	2%
Mean Particle Size Volume Density [b]	15.34	17.86
Mean Particle Size Number Density [b]	1.41	1.07

Notes: a. Please refer to Table C.2. for more information

b. Complete particle size distribution information is provided in Appendix C

Figure D.16. Shear Stress versus Shear Rate of 101-AZ Core #2 Melter Feed Slurry (Run 1) with Yield-Pseudoplastic Model Curve Fits and Corresponding Physical Properties

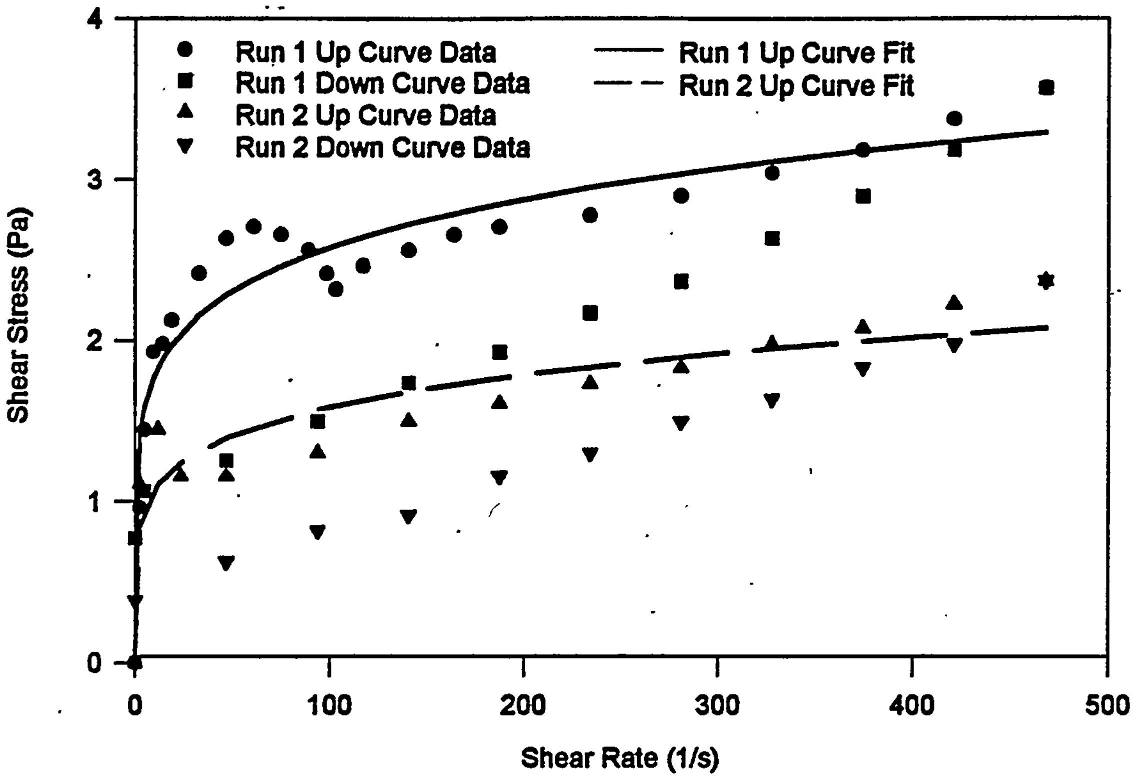


Figure D.17. Shear Stress versus Shear Rate of 101-AZ Core #2 Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

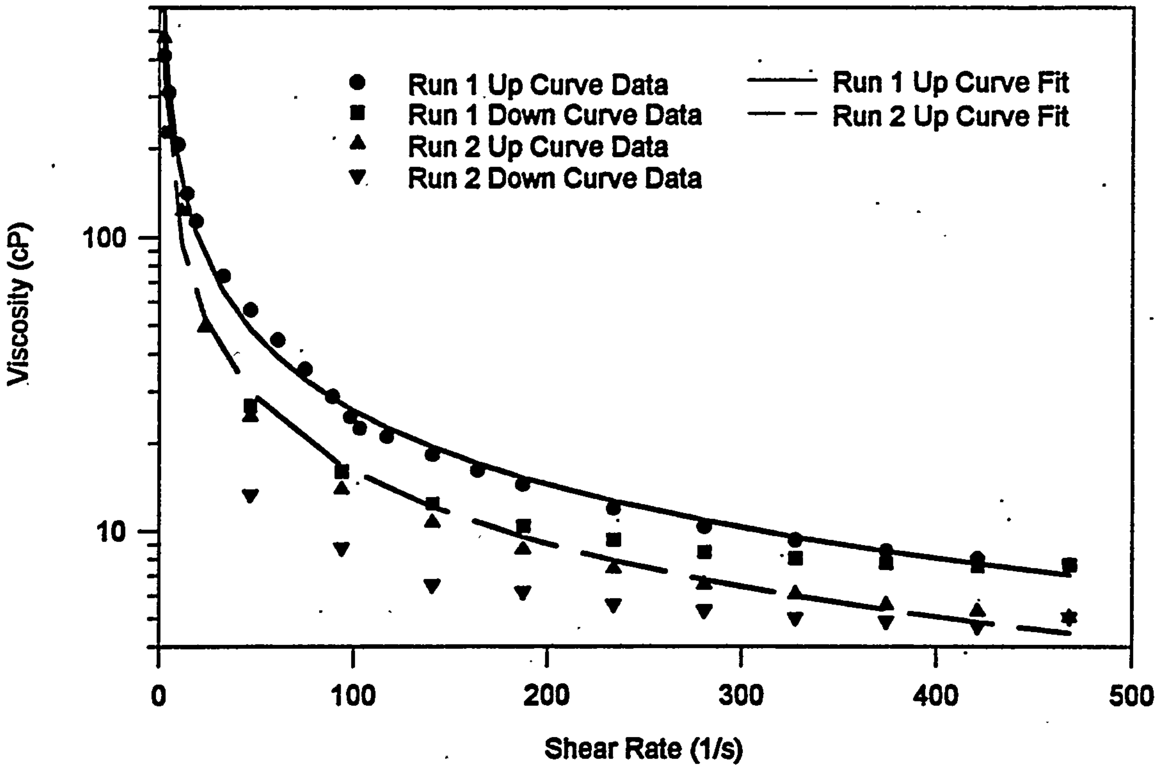


Figure D.18. Viscosity versus Shear Rate of 101-AZ Core #2 Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

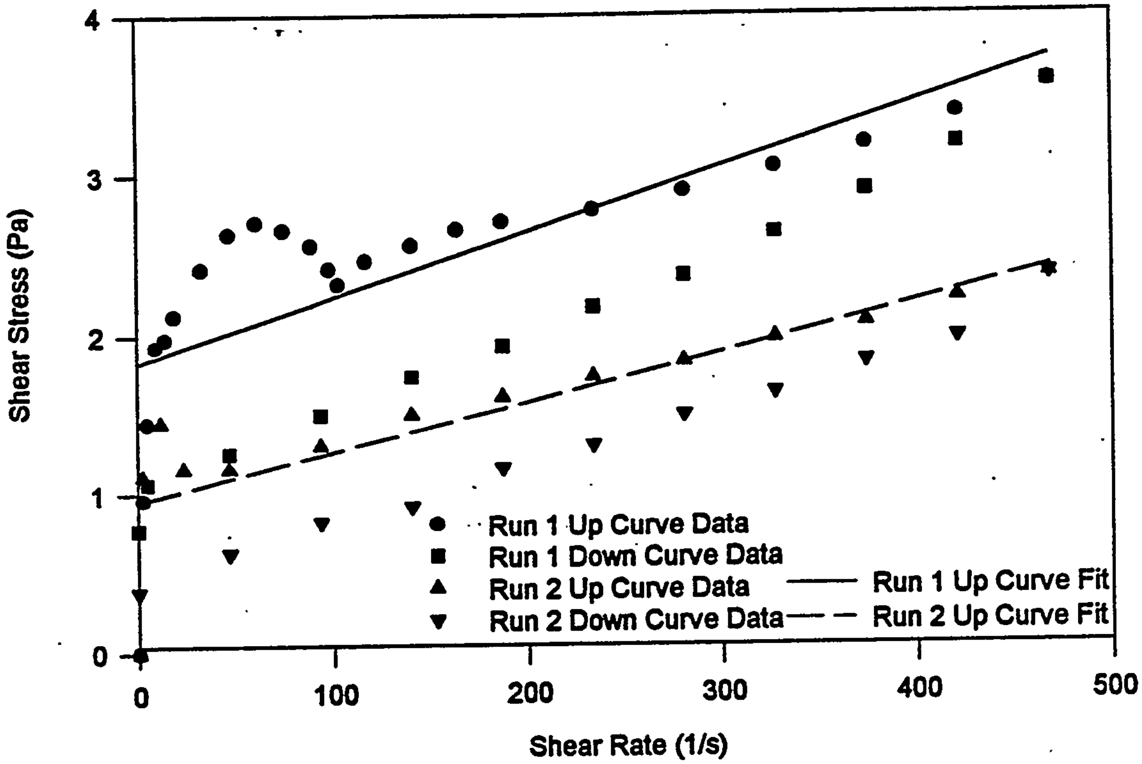


Figure D.19. Shear Stress versus Shear Rate of 101-AZ Core #2 Melter Feed Slurry (All Runs) with Bingham Curve Fits

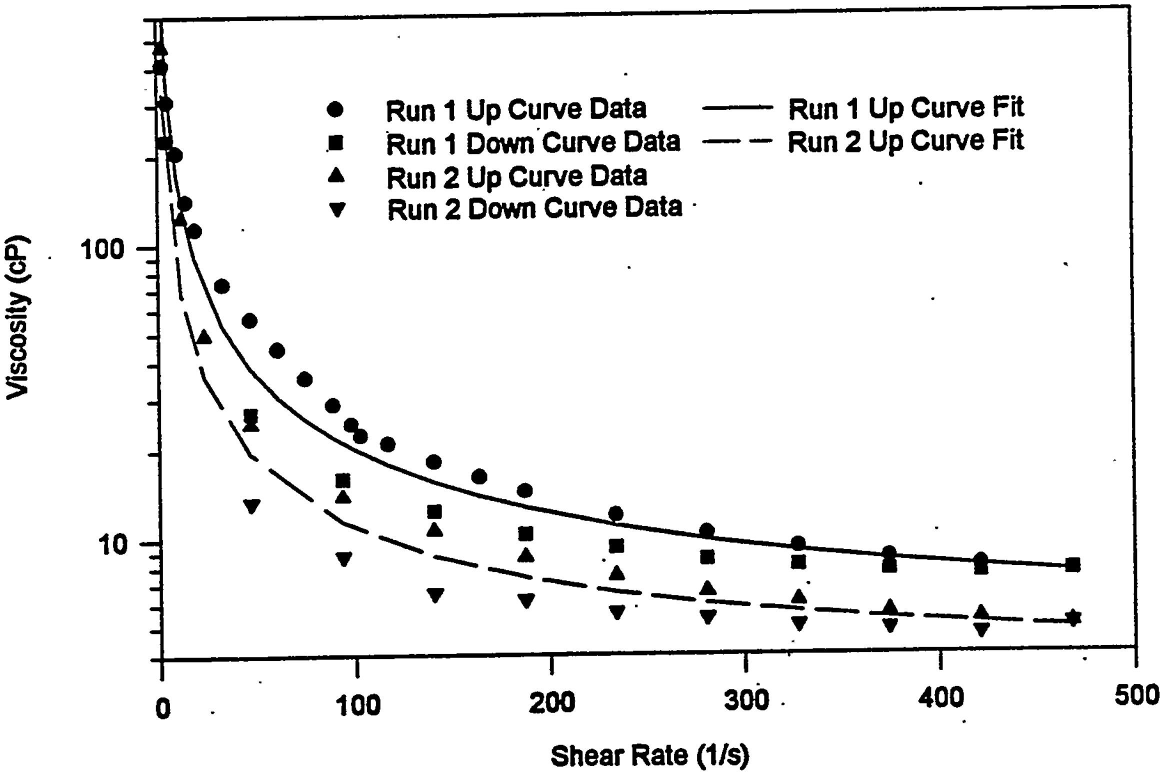
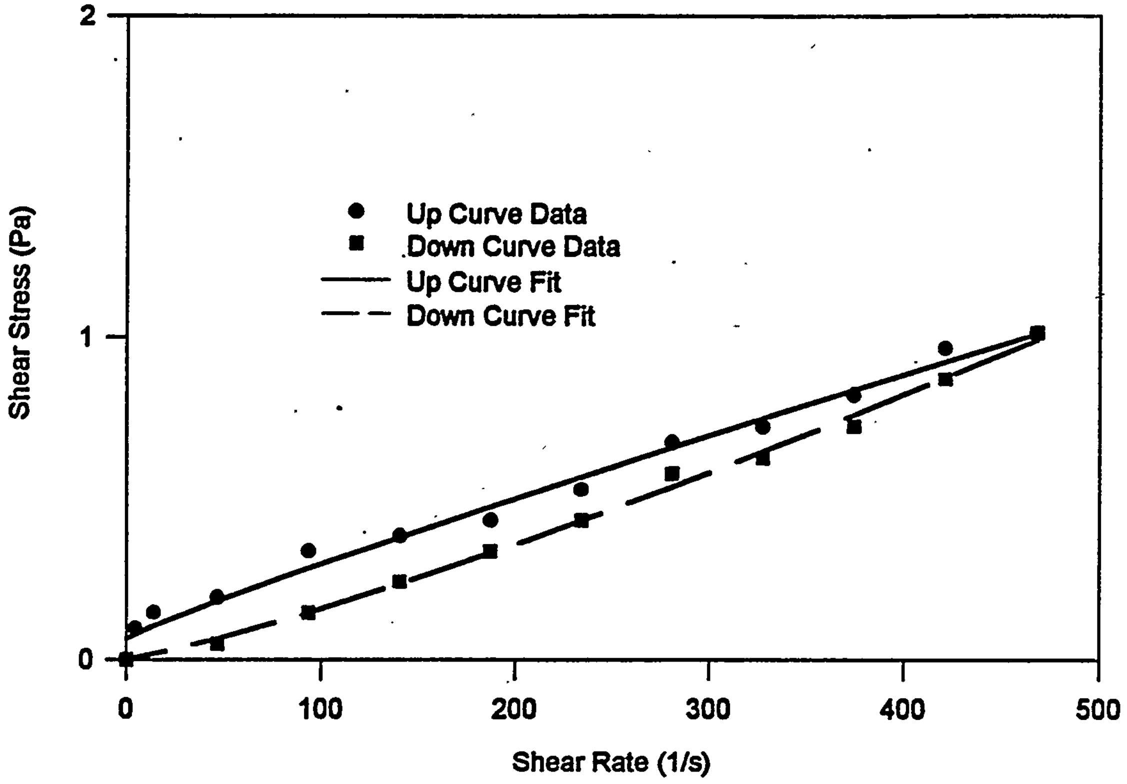


Figure D.20. Viscosity versus Shear Rate of 101-AZ Core #2 Melter Feed Slurry (All Runs) with Bingham Curve Fits



	Average	Reproducibility
Density [a]	1.12	<0.5%
Wt-Fraction Solid [a]	0.186	1%
Wt-Fraction Oxide [a]	0.131	2%
g TO/L [a]	147	
Vol-Fraction Settled Solid [a]	0.38	1%
Density Centrifuged Solid [a]	1.65	<0.5%
Density Centrifuged Supernate [a]	1.04	4%
Vol-Fraction Centrifuged Solid [a]	0.176	<0.5%
Wt-Fraction Centrifuged Solid [a]	0.255	<0.5%
Wt-Fraction Dissolved Solid [a]	NA	
Mean Particle Size Volume Density [b]	47.36	40.24
Mean Particle Size Number Density [b]	1.14	0.85

Notes: a. Please refer to Table C.3. for more information
b. Complete particle size distribution information is provided in Appendix C

Figure D.21. Shear Stress versus Shear Rate of 102-AZ Core #1 Formated Slurry (Run 1) With Yield-Pseudoplastic Model Curve Fits and Corresponding Physical Properties

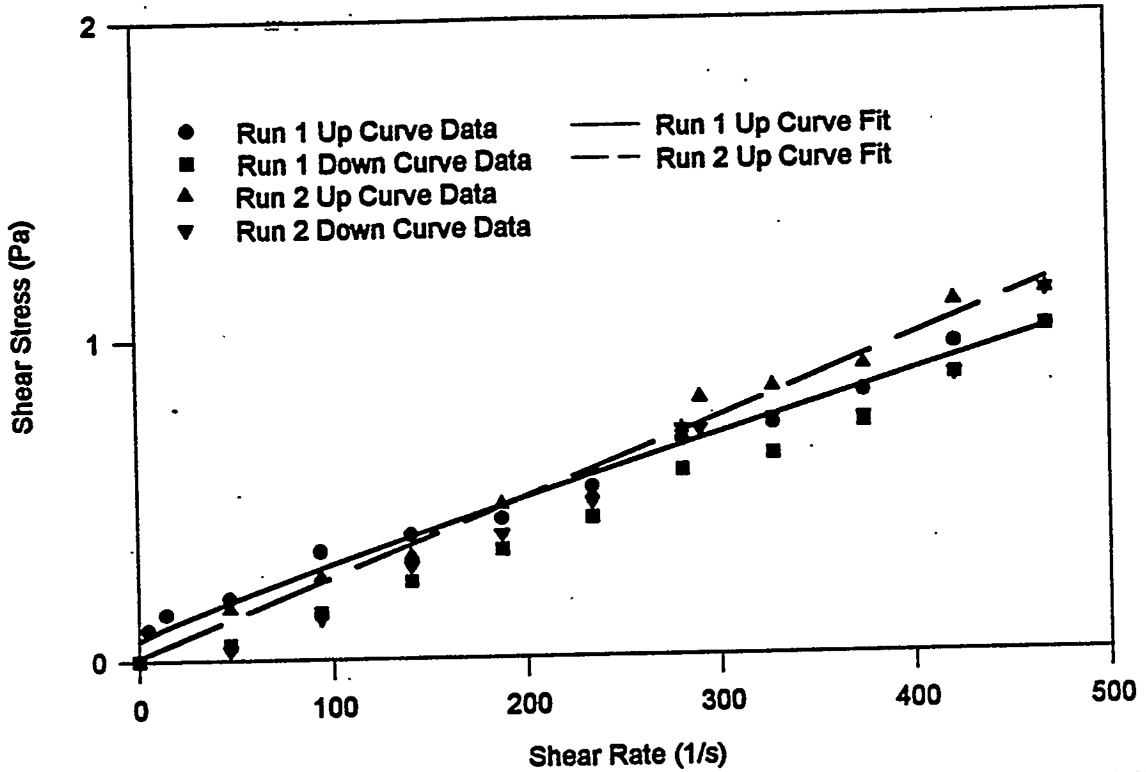


Figure D.22. Shear Stress versus Shear Rate of 102-AZ Core #1 Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

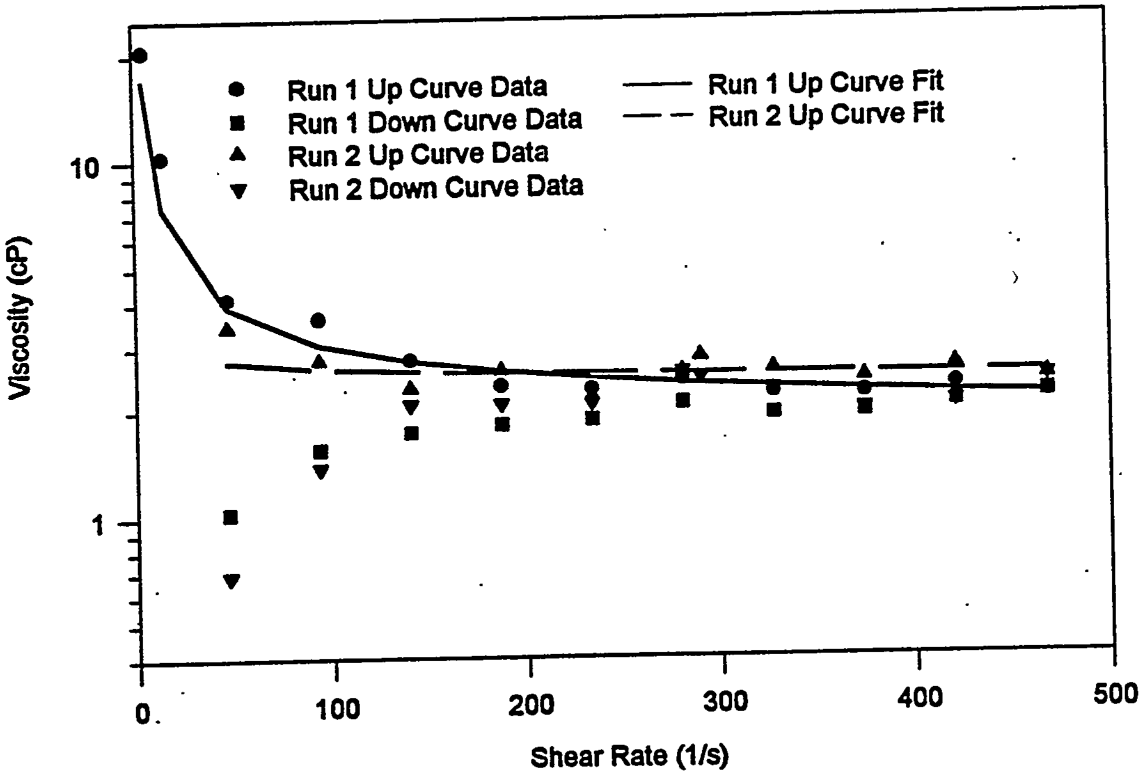


Figure D.23. Viscosity versus Shear Rate of 102-AZ Core #1 Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

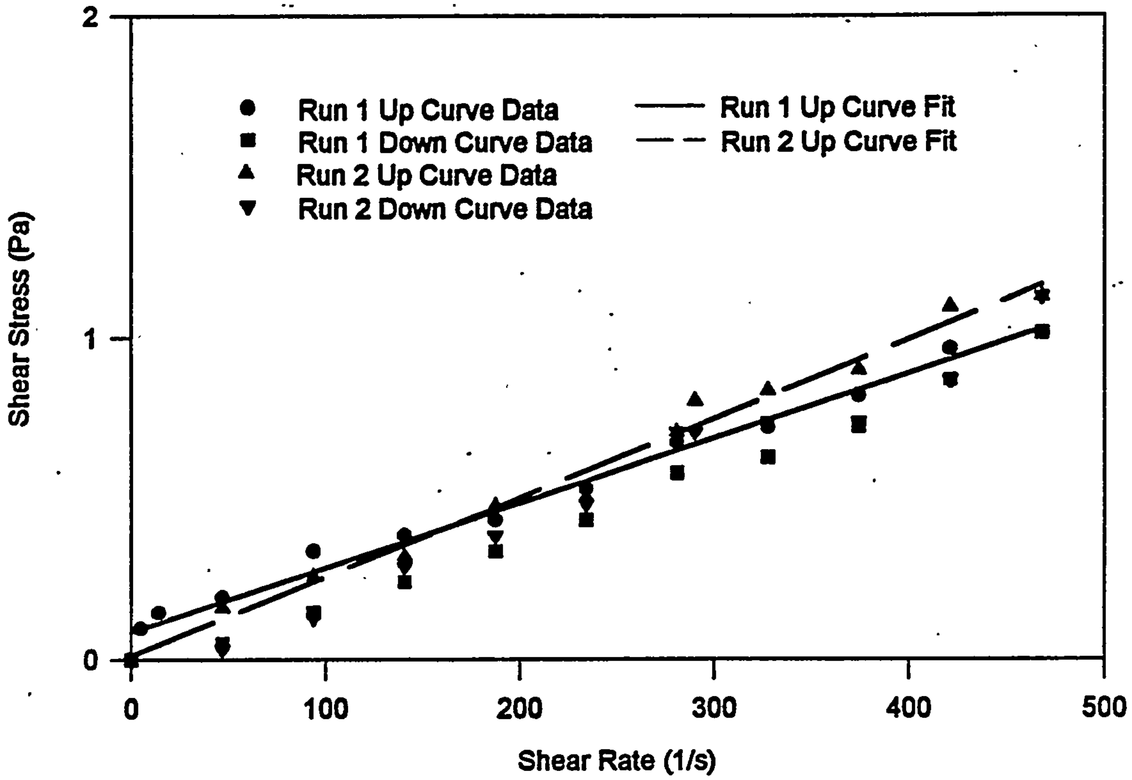


Figure D.24. Shear Stress versus Shear Rate of 102-AZ Core #1 Formated Slurry (All Runs) with Bingham Curve Fits

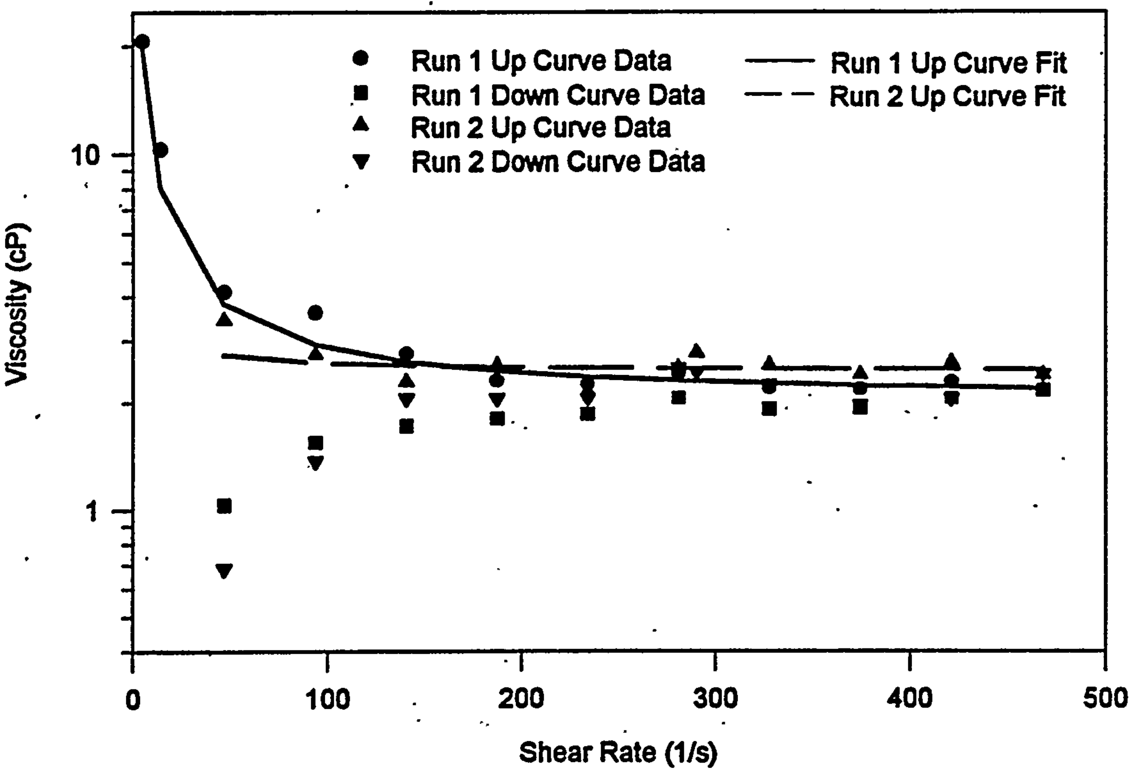
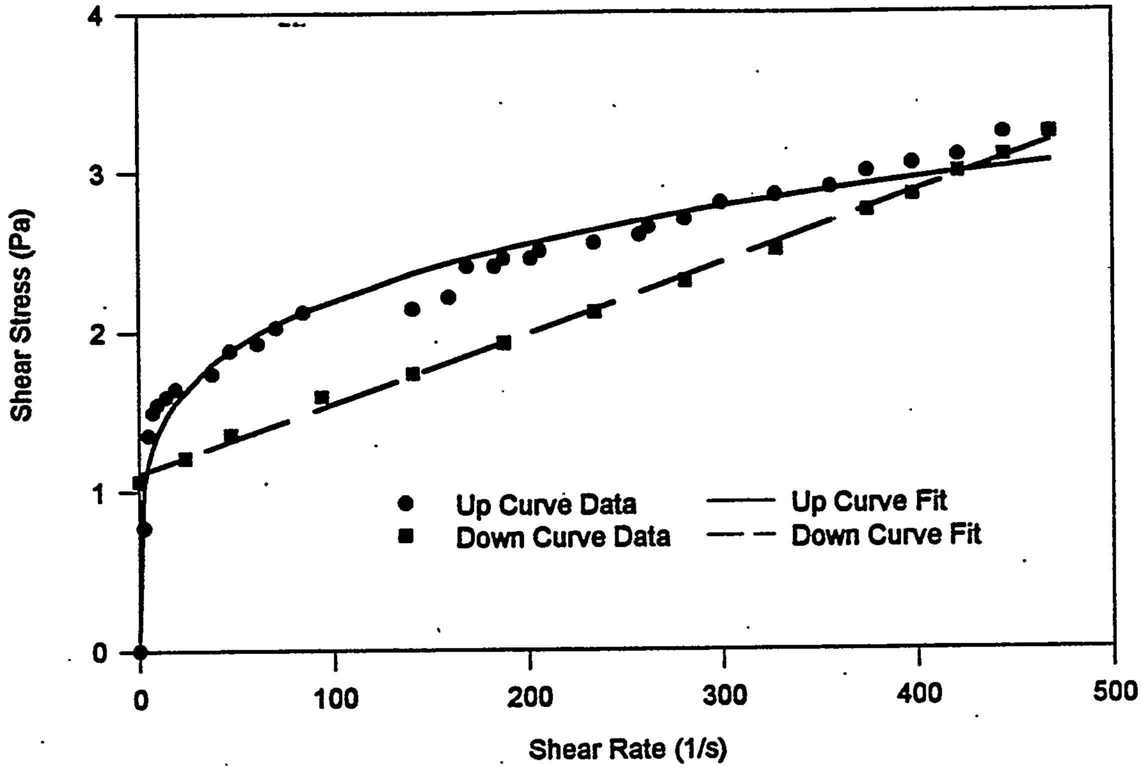


Figure D.25. Viscosity versus Shear Rate of 102-AZ Core #1 Formated Slurry (All Runs) with Bingham Curve Fit



	Average	Reproducibility
Density[a]	1.34	<0.5%
Wt-Fraction Solid[a]	0.436	<0.5%
Wt-Fraction Oxide[a]	0.384	<0.5%
g T.O/L[a]	515	
Vol-Fraction Settled Solid[a]	0.635	1%
Density Centrifuged Solid[a]	1.69	<0.5%
Density Centrifuged Supernate[a]	1.03	<0.5%
Vol-Fraction Centrifuged Solid[a]	0.483	2%
Wt-Fraction Centrifuged Solid[a]	0.603	1%
Wt-Fraction Dissolved Solid[a]	NA	
Mean Particle Size Volume Density	NA	NA
Mean Particle Size Number Density	NA	NA

Notes: a. Please refer to Table C.3. for more information

Figure D.26. Shear Stress versus Shear Rate of 102-AZ Core #1 Melter Feed Slurry (Run 2) with Yield-Pseudoplastic Model Curve Fits and Corresponding Physical Properties

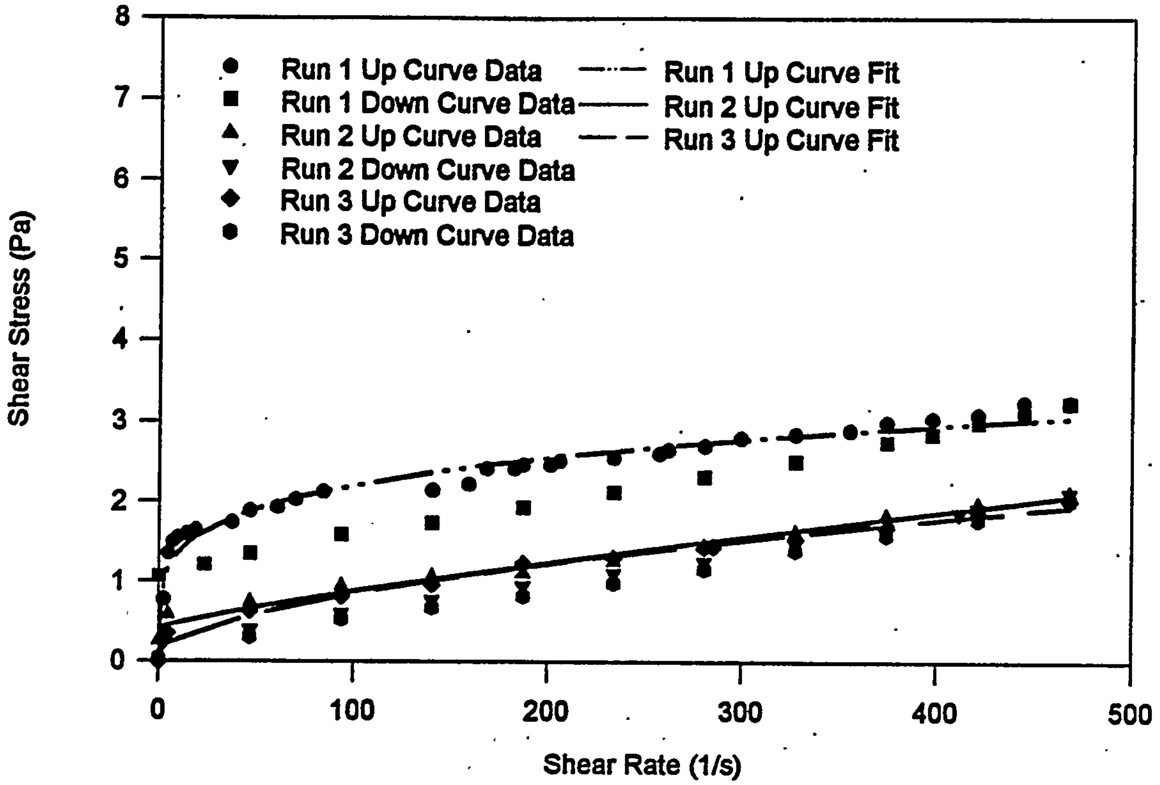


Figure D.27. Shear Stress versus Shear Rate of 102-AZ Core #1 Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

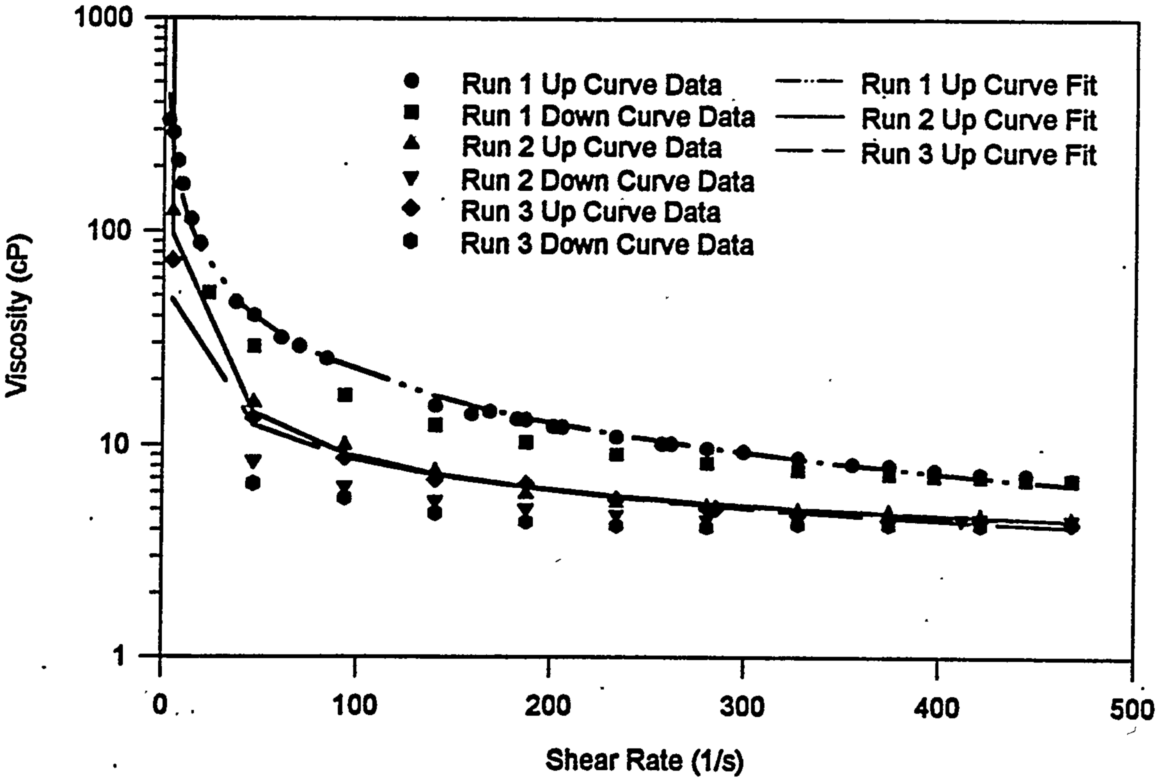


Figure D.28. Viscosity versus Shear Rate of 102-AZ Core #1 Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

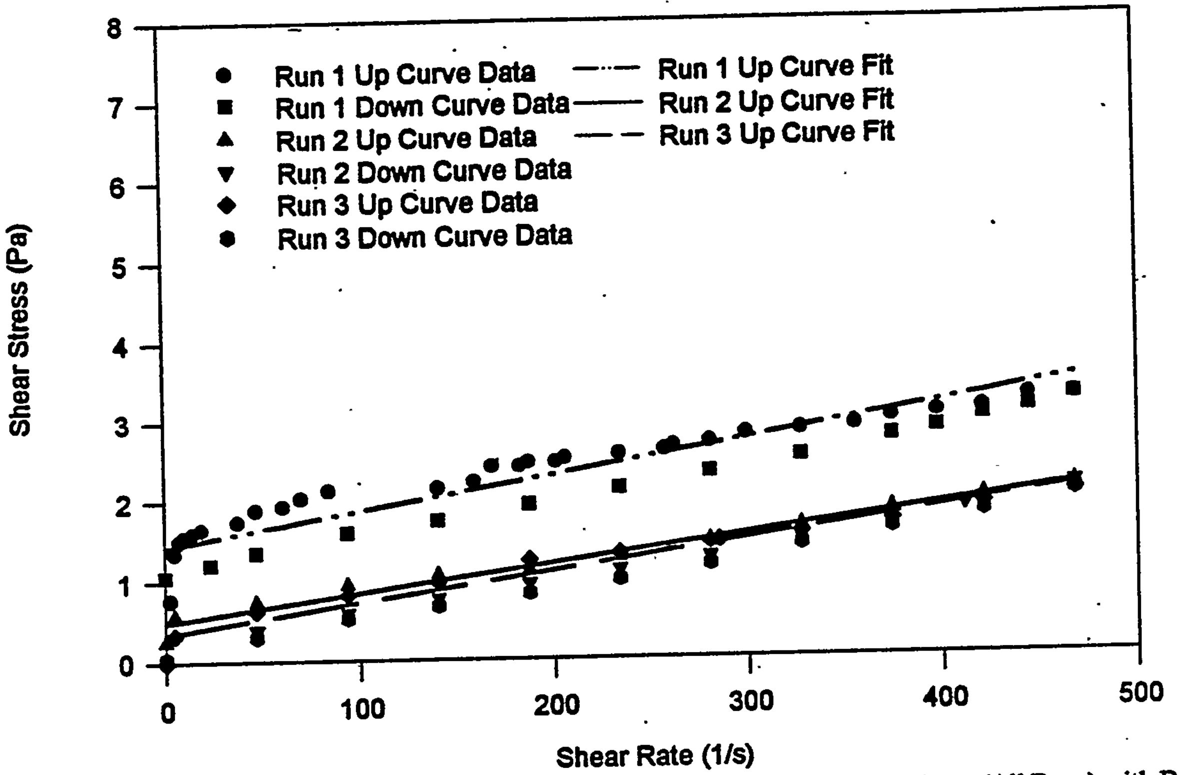


Figure D.29. Shear Stress versus Shear Rate of 102-AZ Core #1 Melter Feed Slurry (All Runs) with Bingham Curve Fits

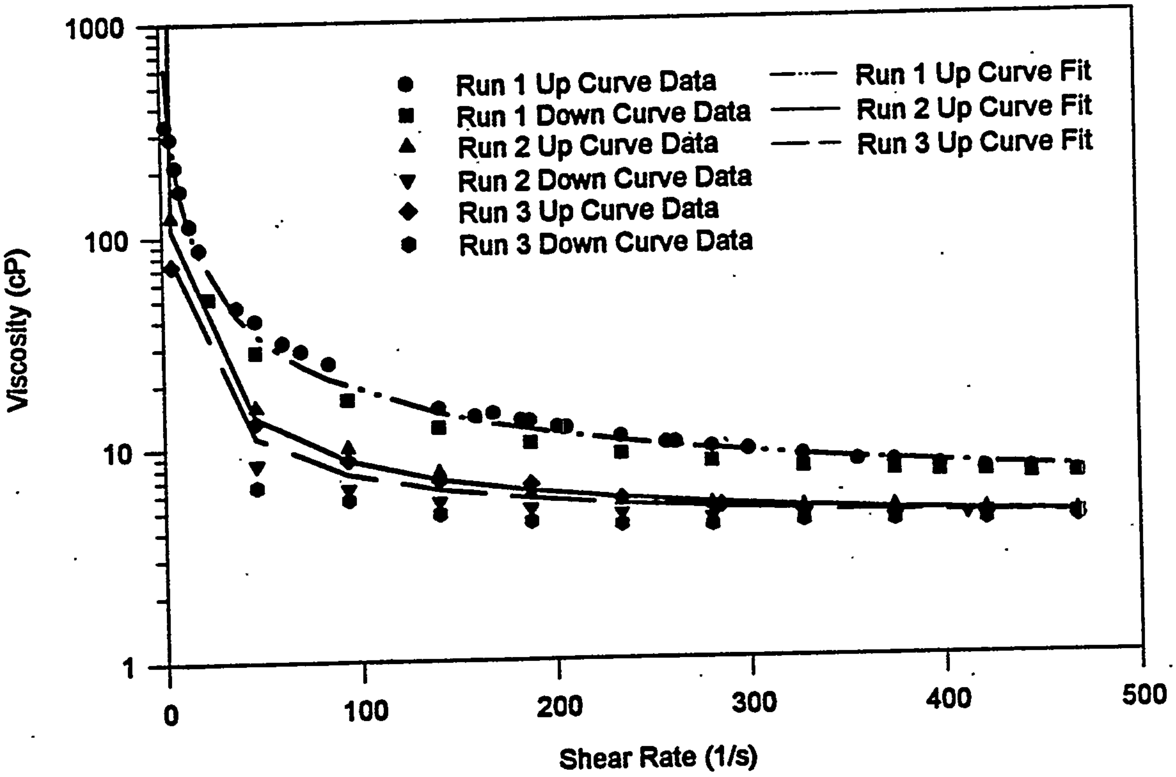
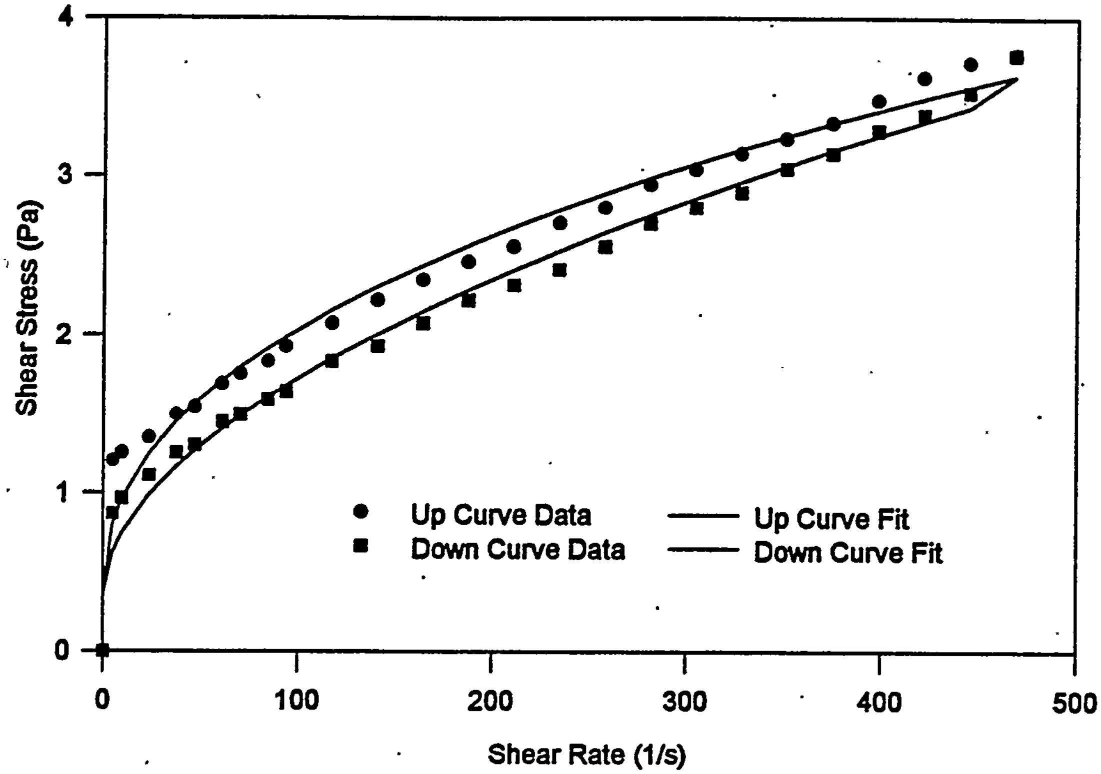


Figure D.30. Viscosity versus Shear Rate of 102-AZ Core #1 Melter Feed Slurry (All Runs) with Bingham Curve Fits



	Average	Reproducibility
Density [a]	1.11	<0.5%
Wt-Fraction Solid [a]	0.192	9%
Wt-Fraction Oxide [a]	0.145	10%
g TO/L [a]	161	
Vol-Fraction Settled Solid [a]	0.87	1%
Density Centrifuged Solid [a]	1.27	2%
Density Centrifuged Supernate [a]	1.01	<0.5%
Vol-Fraction Centrifuged Solid [a]	0.400	1%
Wt-Fraction Centrifuged Solid [a]	0.462	1%
Wt-Fraction Dissolved Solid [a]	0.024	21%
Mean Particle Size Volume Density [b]	25.57	13.27
Mean Particle Size Number Density [b]	1.30	1.38

Notes: a. Please refer to Table C.4. for more information
b. Complete particle size distribution information is provided in Appendix C

Figure D.31. Shear Stress versus Shear Rate of NCAW Simulant Formated Slurry (Run 1) with Yield-Pseudoplastic Model Curve Fits and Corresponding Physical Properties

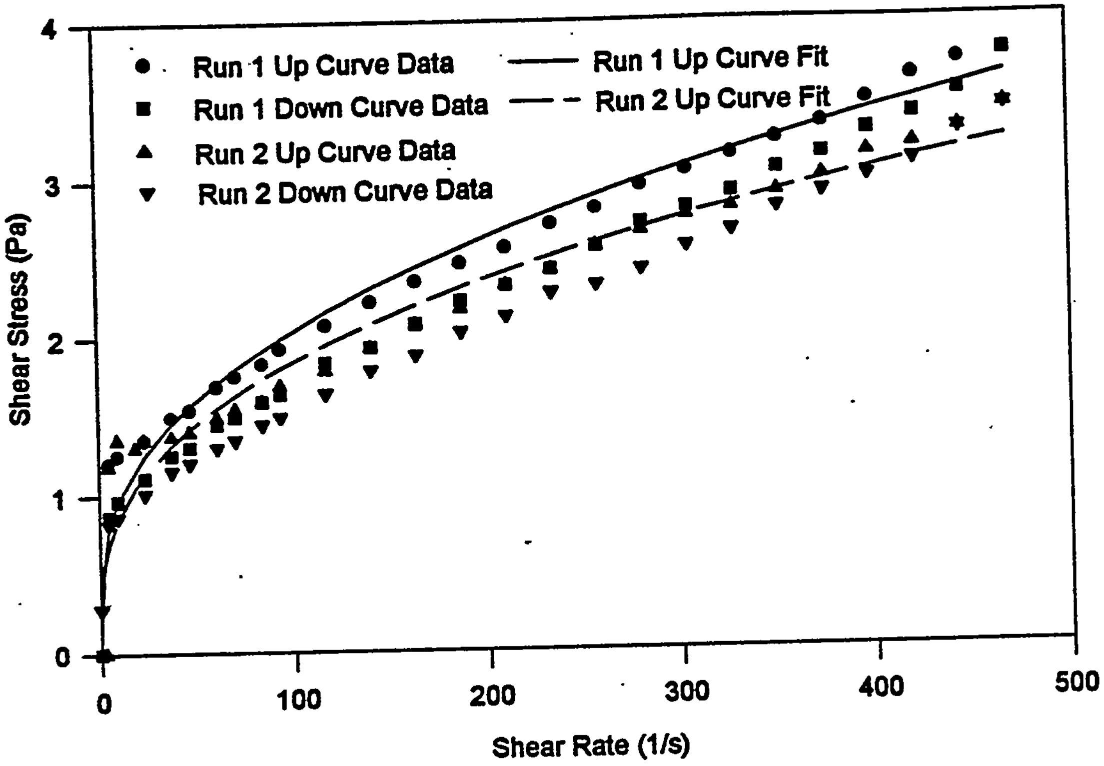


Figure D.32. Shear Stress versus Shear Rate of NCAW Simulant Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

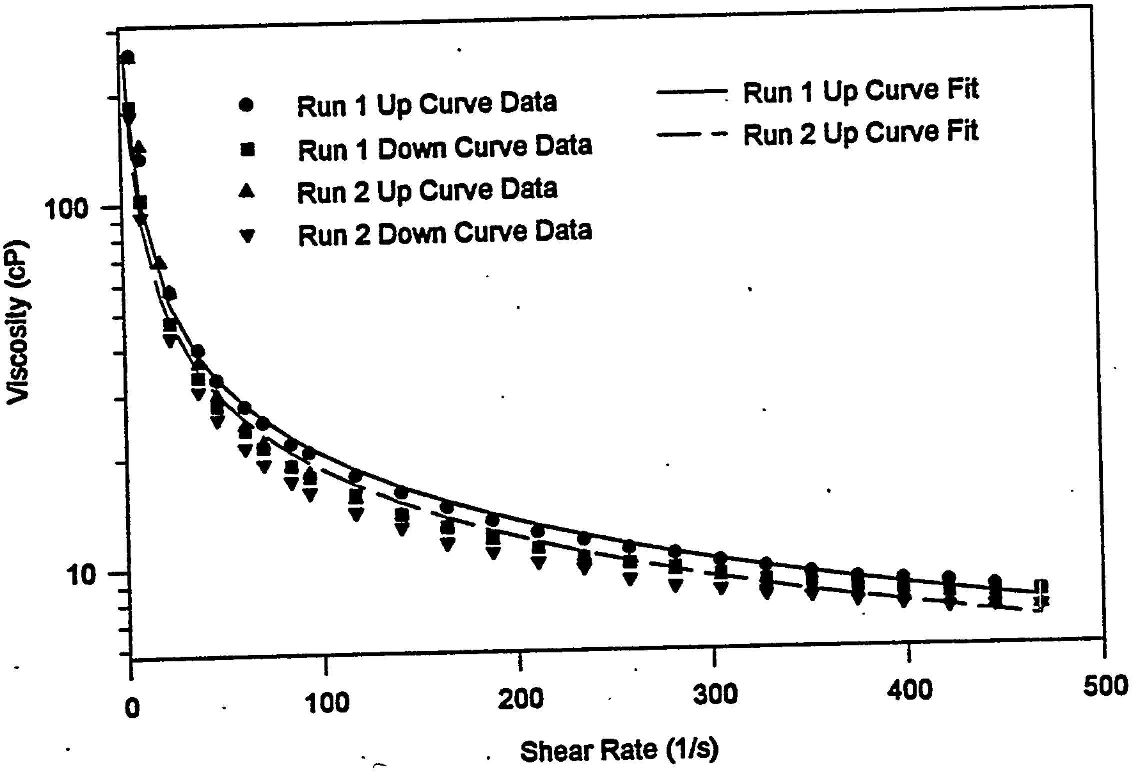


Figure D.33. Viscosity versus Shear Rate of NCAW Simulant Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

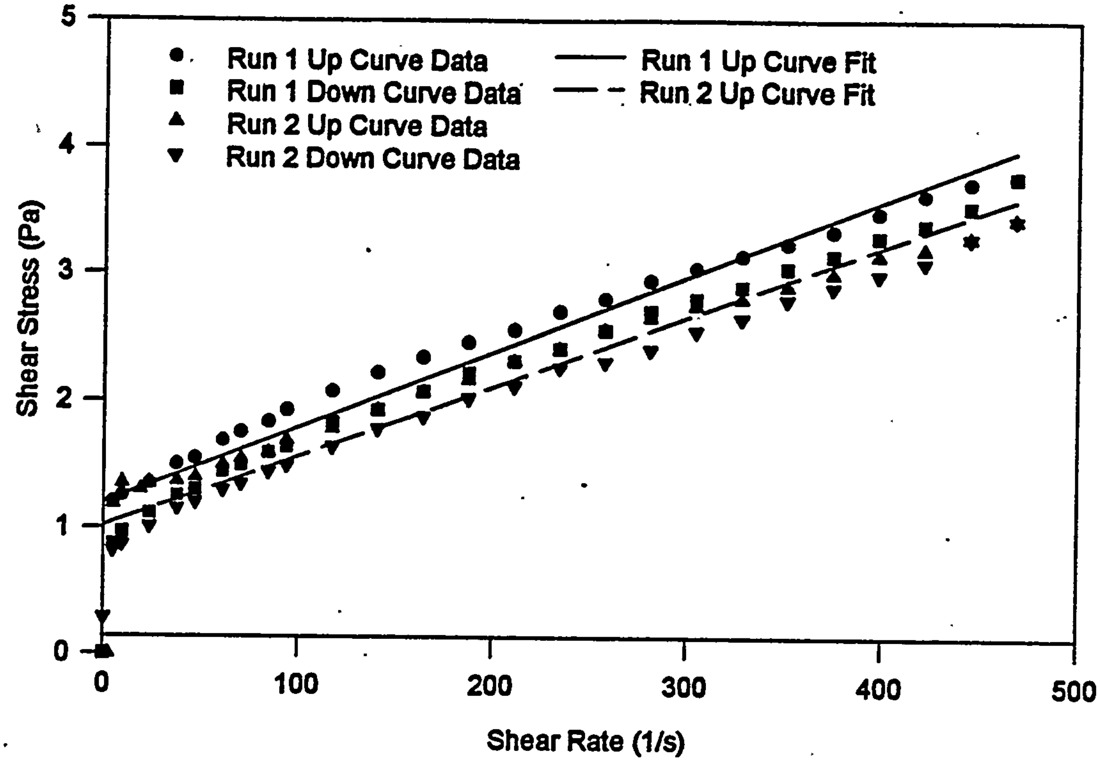


Figure D.34. Shear Stress versus Shear Rate of NCAW Simulant Formated Slurry (All Runs) with Bingham Curve Fits

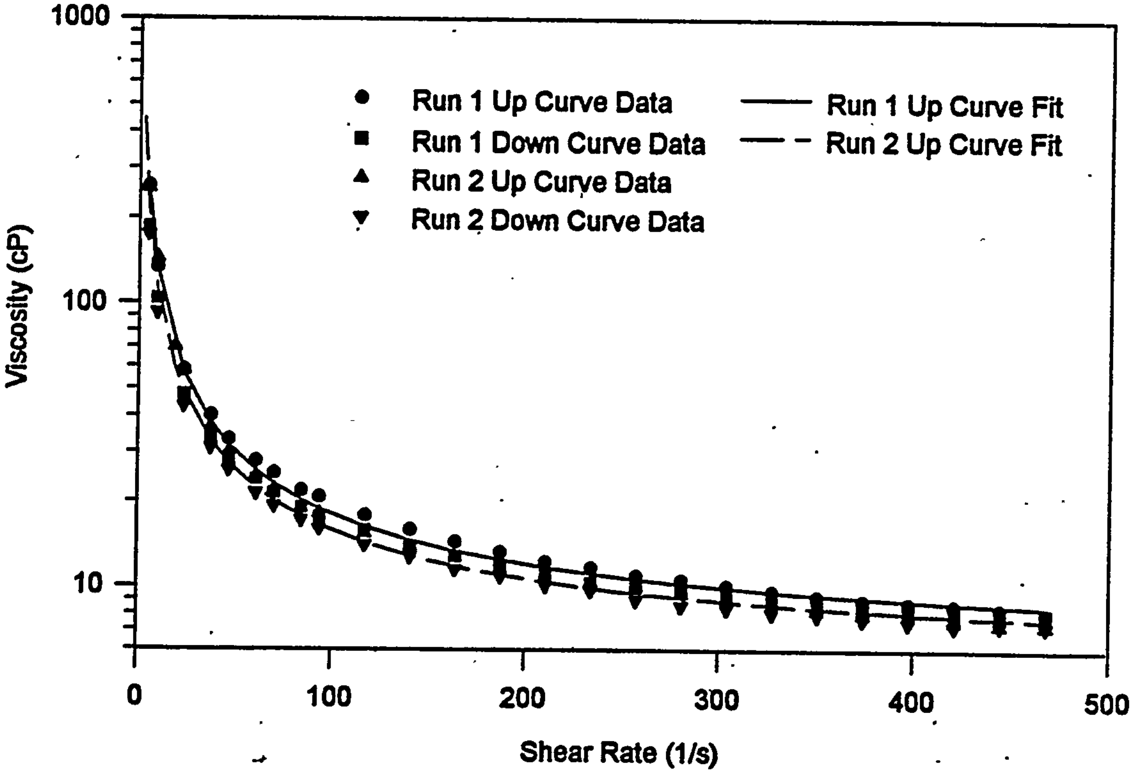
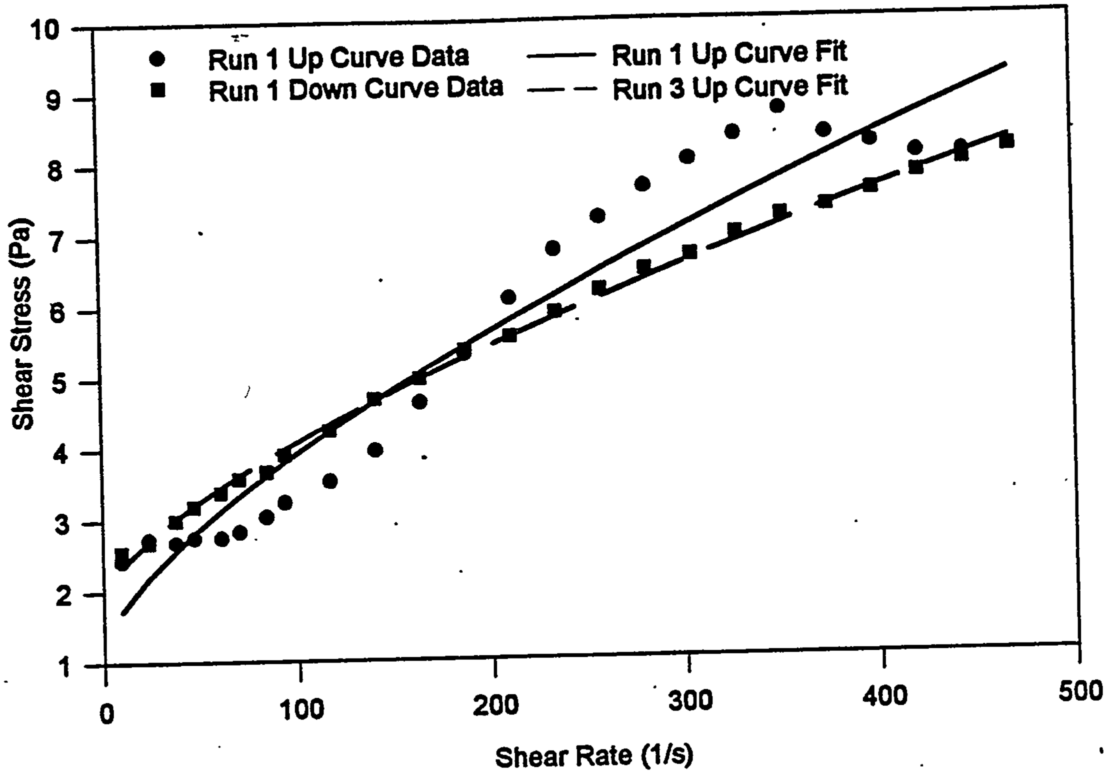


Figure D.35. Viscosity versus Shear Rate of NCAW Simulant Formated Slurry (All Runs) with Bingham Curve Fits



	Average	Reproducibility
Density [a]	1.28	<0.5%
Wt-Fraction Solid [a]	0.370	2%
Wt-Fraction Oxide [a]	0.341	5%
g TO/L [a]	438	2%
Vol-Fraction Settled Solid [a]	0.870	1%
Density Centrifuged Solid [a]	1.50	<0.5%
Density Centrifuged Supernate [a]	1.01	1%
Vol-Fraction Centrifuged Solid [a]	0.583	1%
Wt-Fraction Centrifuged Solid [a]	0.672	1%
Wt-Fraction Dissolved Solid [a]	0.014	11%
Mean Particle Size Volume Density [b]	25.57	13.27
Mean Particle Size Number Density [b]	1.30	1.38

Notes: a. Please refer to Table C.4. for more information
b. Complete particle size distribution information is provided in Appendix C

Figure D.36. Shear Stress versus Shear Rate of NCAW Simulant Melter Feed (Run 1) with Yield-Pseudoplastic Model Curve Fits

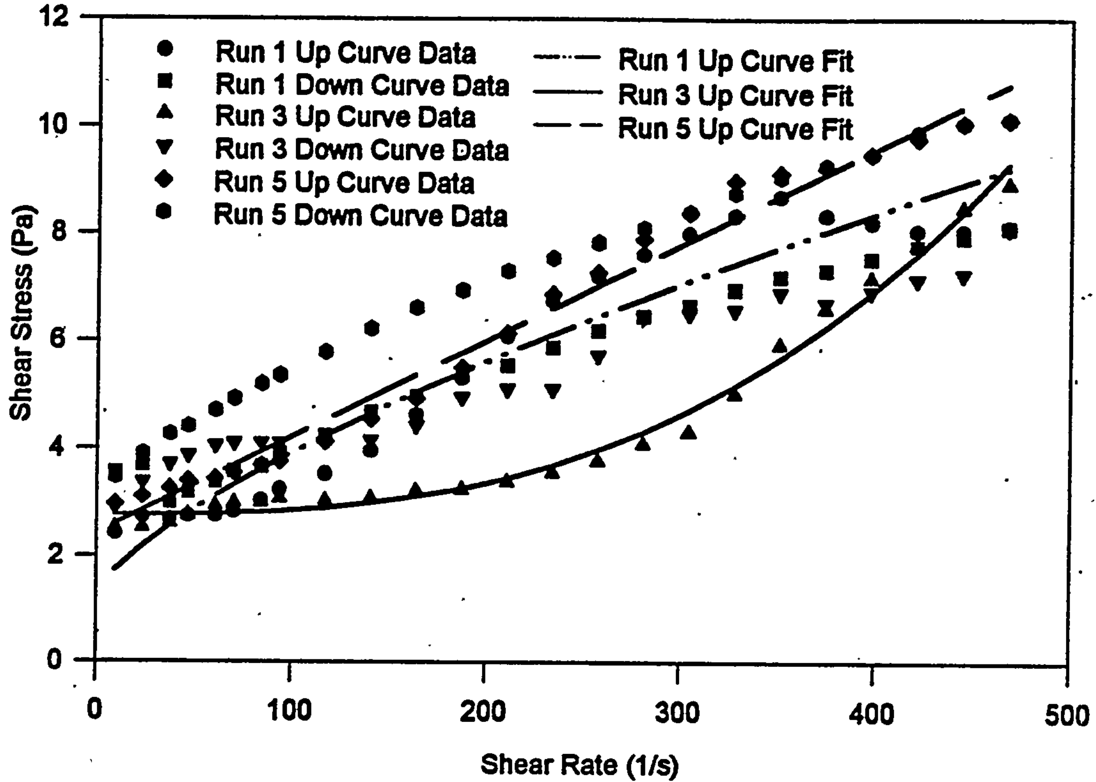


Figure D.37. Shear Stress versus Shear Rate of NCAW Simulant Melter Feed Slurry (Runs 1, 3, & 5) with Yield-Pseudoplastic Model Curve Fits

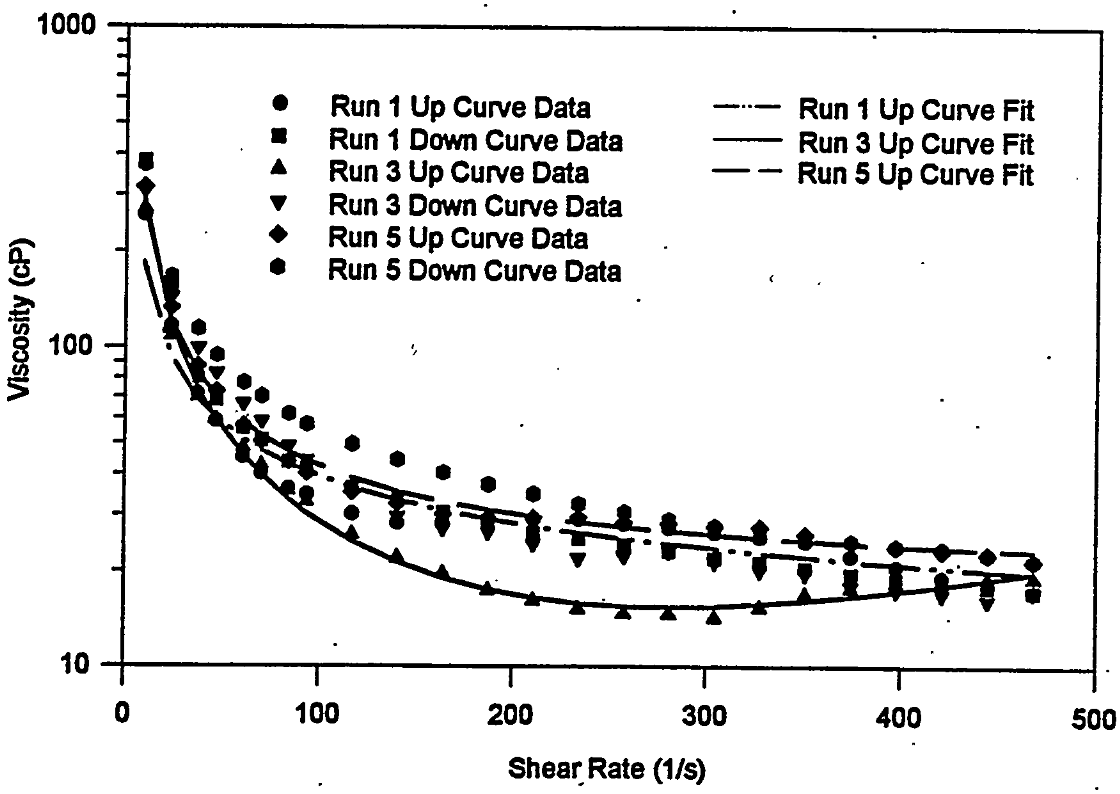


Figure D.38. Viscosity versus Shear Rate of NCAW Simulant Melter Feed Slurry (Runs 1, 3, & 5) with Yield-Pseudoplastic Model Curve Fits

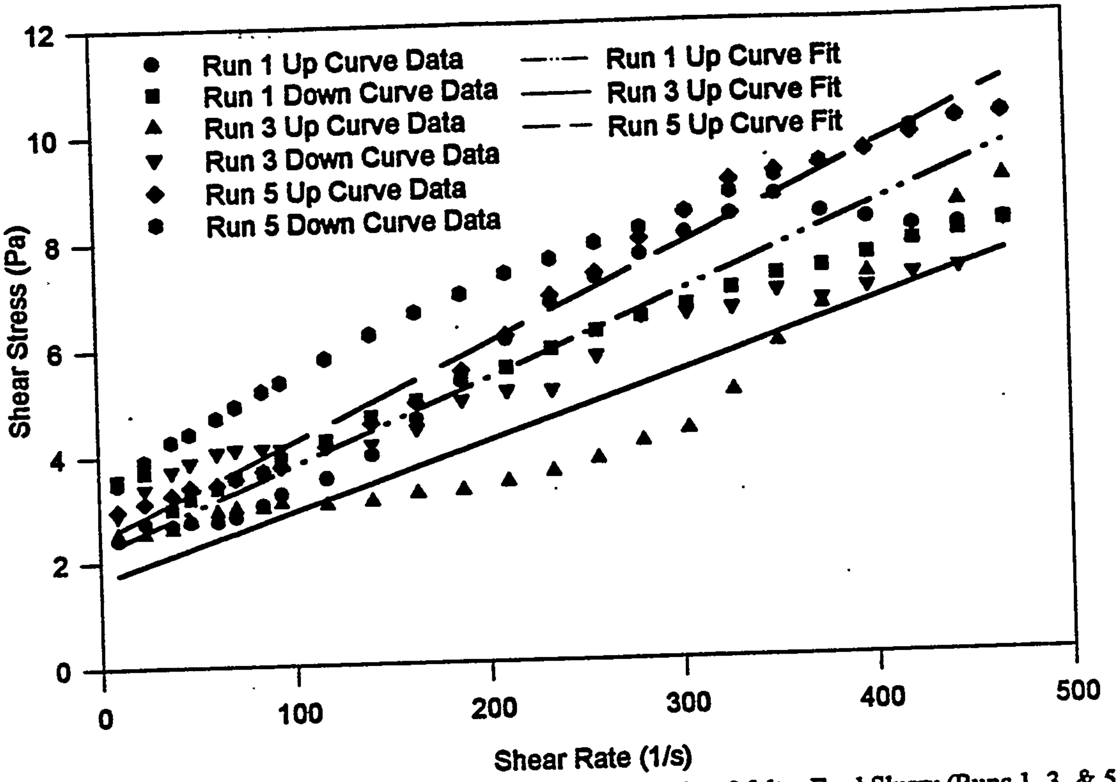


Figure D.39. Shear Stress versus Shear Rate of NCAW Simulant Melter Feed Slurry (Runs 1, 3, & 5) with Bingham Curve Fits

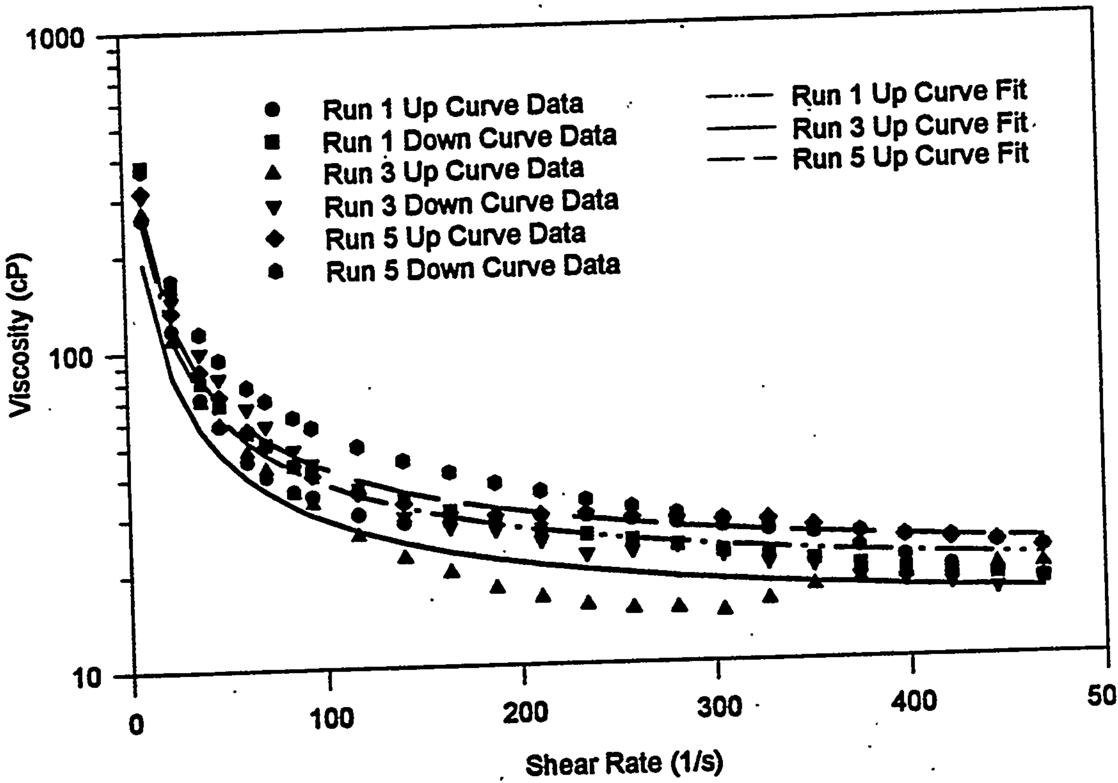
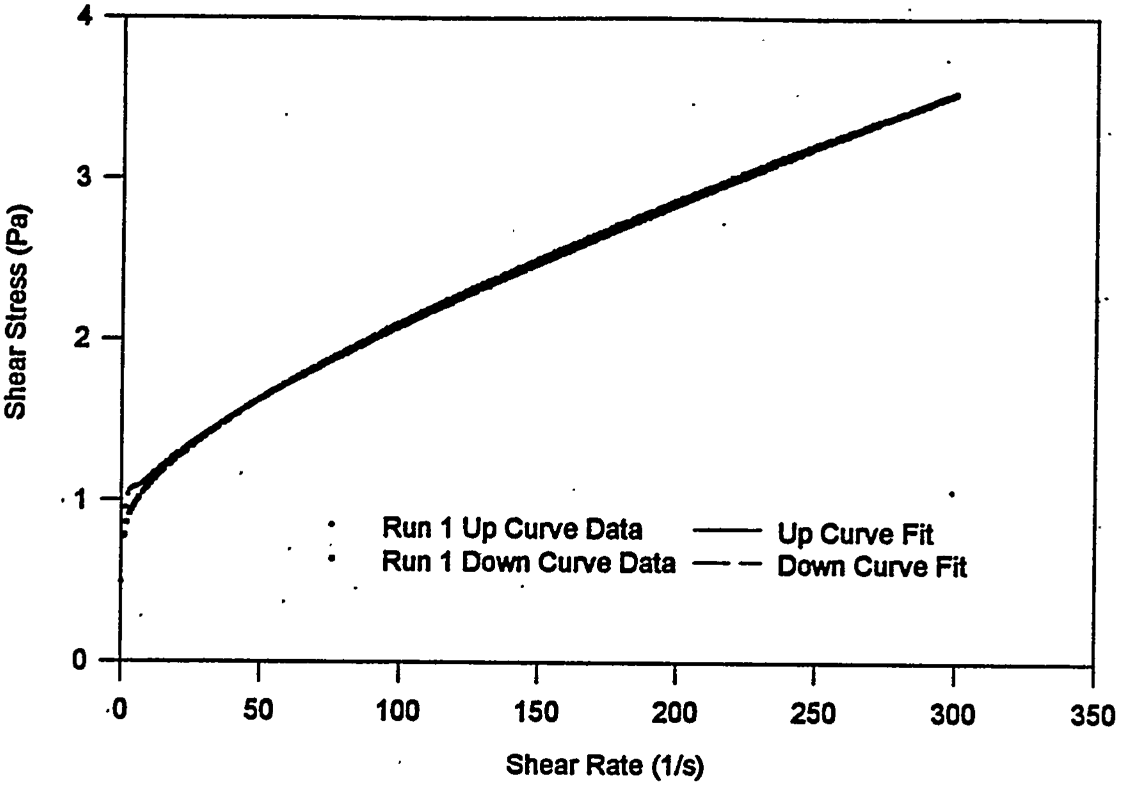


Figure D.40. Viscosity versus Shear Rate of NCAW Simulant Melter Feed Slurry (Runs 1, 3, & 5) with Bingham Curve Fits



	Average	Reproducibility
Density [a]	1.14	1%
Wt-Fraction Solid [a]	0.222	1%
Wt-Fraction Oxide [a]	0.129	<0.5%
g TO/L [a]	147	
Vol-Fraction Settled Solid [a]	0.84	<0.05
Density Centrifuged Solid [a]	1.34	1%
Density Centrifuged Supernate [a]	1.04	1%
Vol-Fraction Centrifuged Solid [a]	0.421	2%
Wt-Fraction Centrifuged Solid [a]	0.484	<0.5%
Wt-Fraction Dissolved Solid [a]	NA	
Mean Particle Size Volume Density	NA	NA
Mean Particle Size Number Density	NA	NA

Notes: a. Please refer to Table C.5. for more information

Figure D.41. Shear Stress versus Shear Rate of 102-AZ Core #1 Simulant Formated Slurry (Run 1) with Yield-Pseudoplastic Model Curve Fits and Corresponding Physical Properties

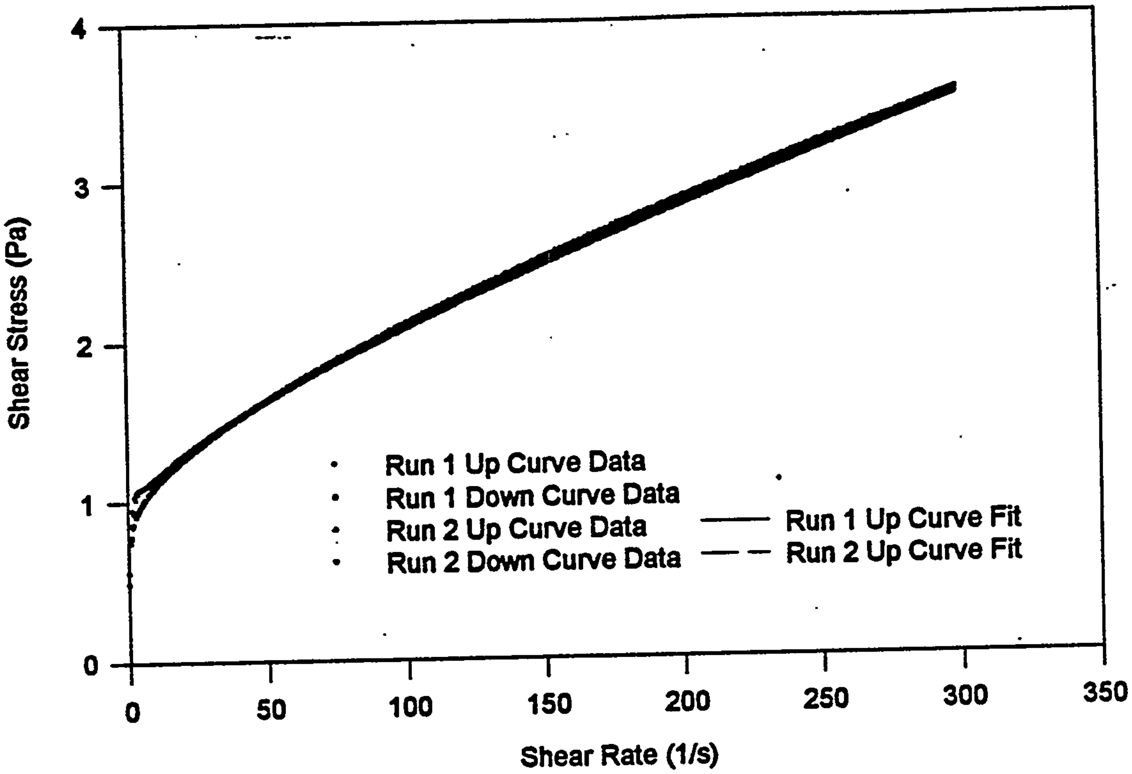


Figure D.42. Shear Stress versus Shear Rate of 102-AZ Core #1 Simulant Formated Slurry (All Runs) with Pseudoplastic Model Curve Fits

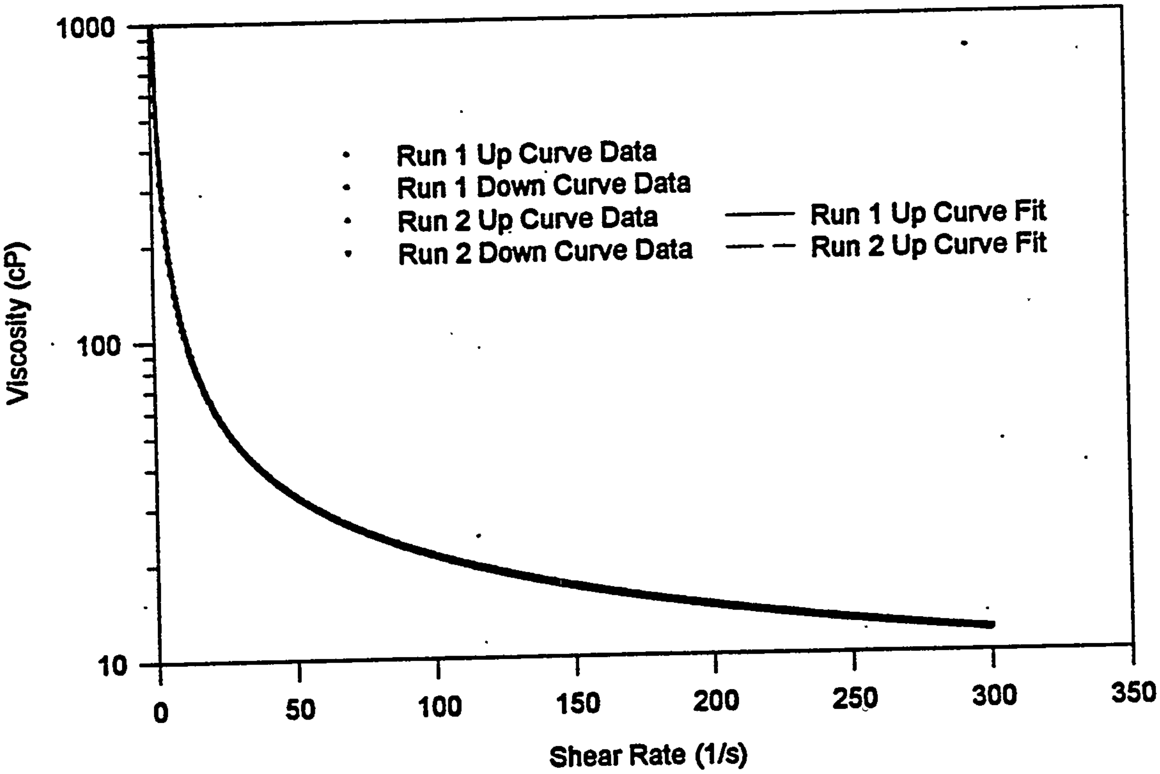


Figure D.43. Viscosity versus Shear Rate of 102-AZ Core #1 Simulant Formated Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

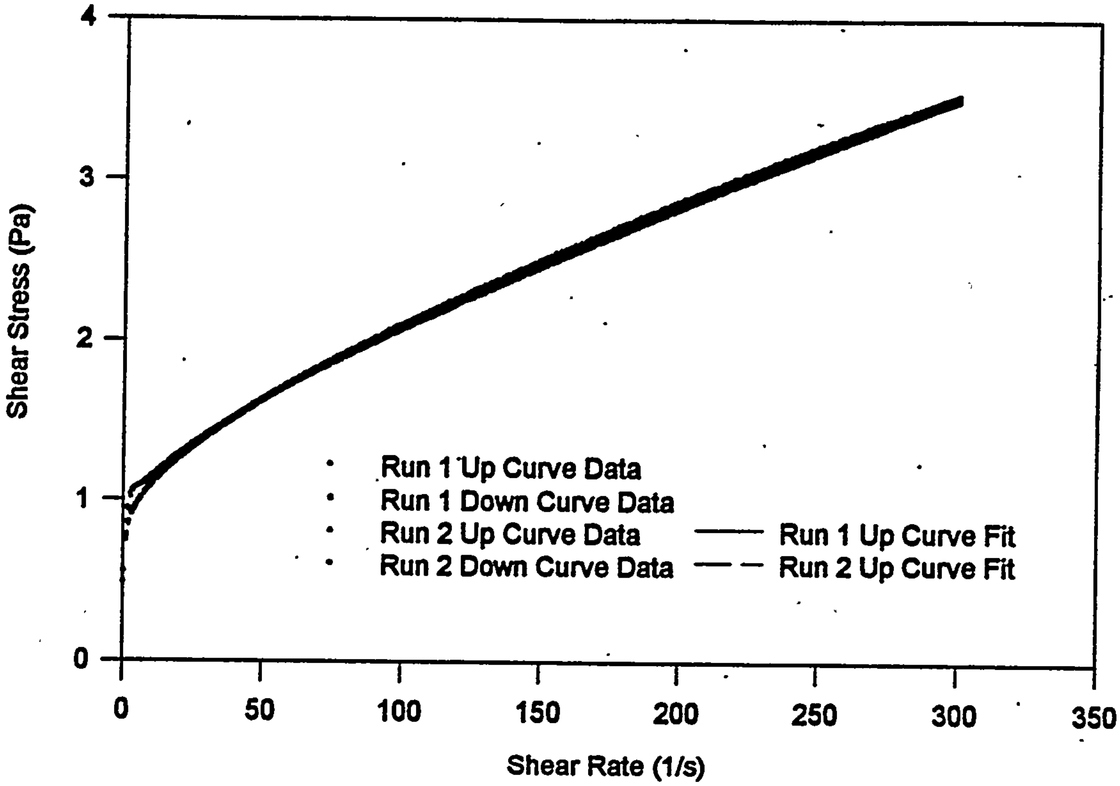


Figure D.44. Shear Stress versus Shear Rate of 102-AZ Core #1 Simulant Formated Slurry (All Runs) with Bingham Curve Fits

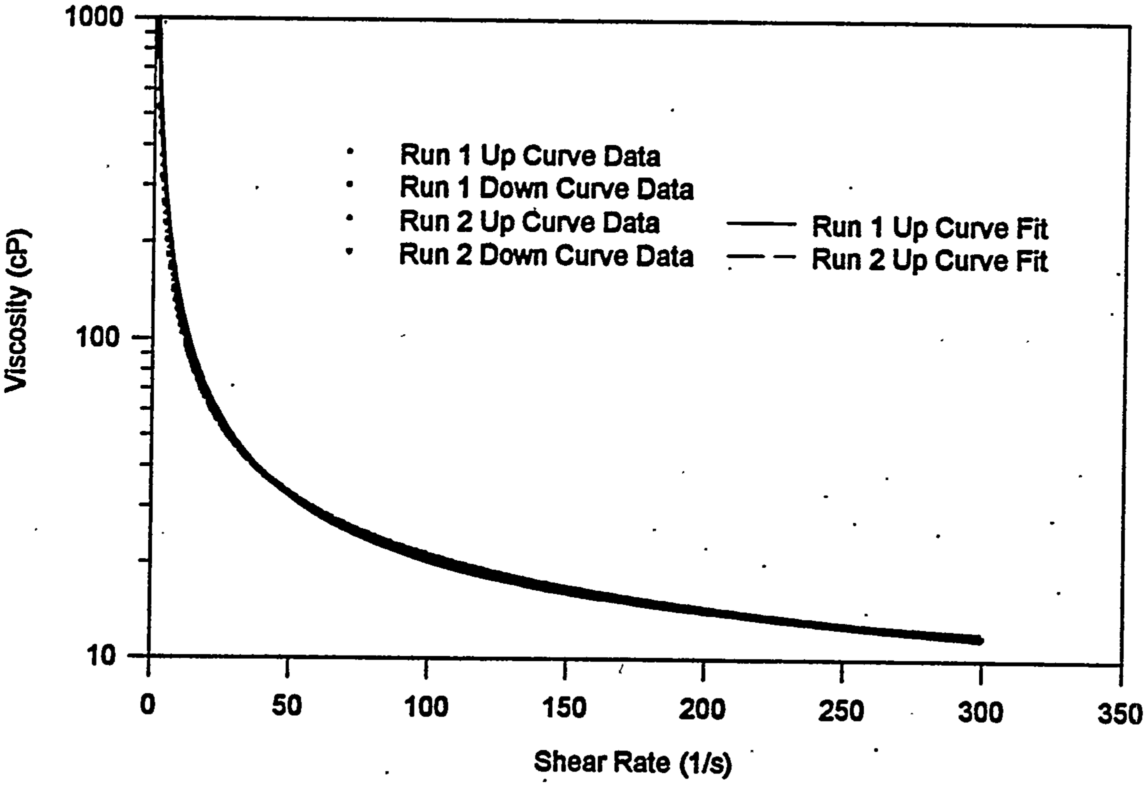
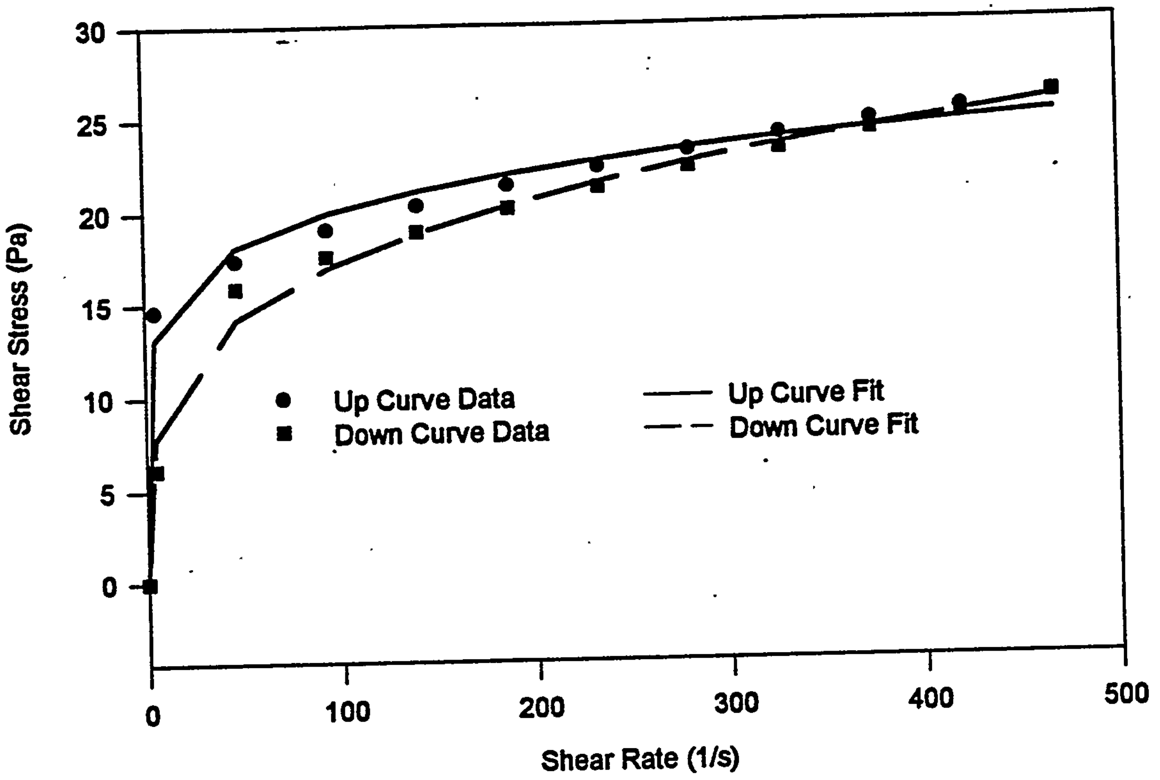


Figure D.45. Viscosity versus Shear Rate of 102-AZ Core #1 Simulant Formated Slurry (All Runs) with Bingham Curve Fits



	Average	Reproducibility
Density [a]	1.42	2%
Wt-Fraction Solid [a]	0.478	<0.5%
Wt-Fraction Oxide [a]	0.404	<0.5%
g TO/L [a]	573	
Vol-Fraction Settled Solid [a]	1.00	<0.5%
Density Centrifuged Solid [a]	NA	
Density Centrifuged Supernate [a]	NA	
Vol-Fraction Centrifuged Solid [a]	NA	
Wt-Fraction Centrifuged Solid [a]	NA	
Wt-Fraction Dissolved Solid [a]	NA	
Mean Particle Size Volume Density	NA	
Mean Particle Size Number Density	NA	

Notes: a. Please refer to Table C.5. for more information

Figure D.46. Shear Stress versus Shear Rate of 102-AZ Core #1 Simulant Melter Feed Slurry (Run 1) with Yield-Pseudoplastic Model Curve Fits

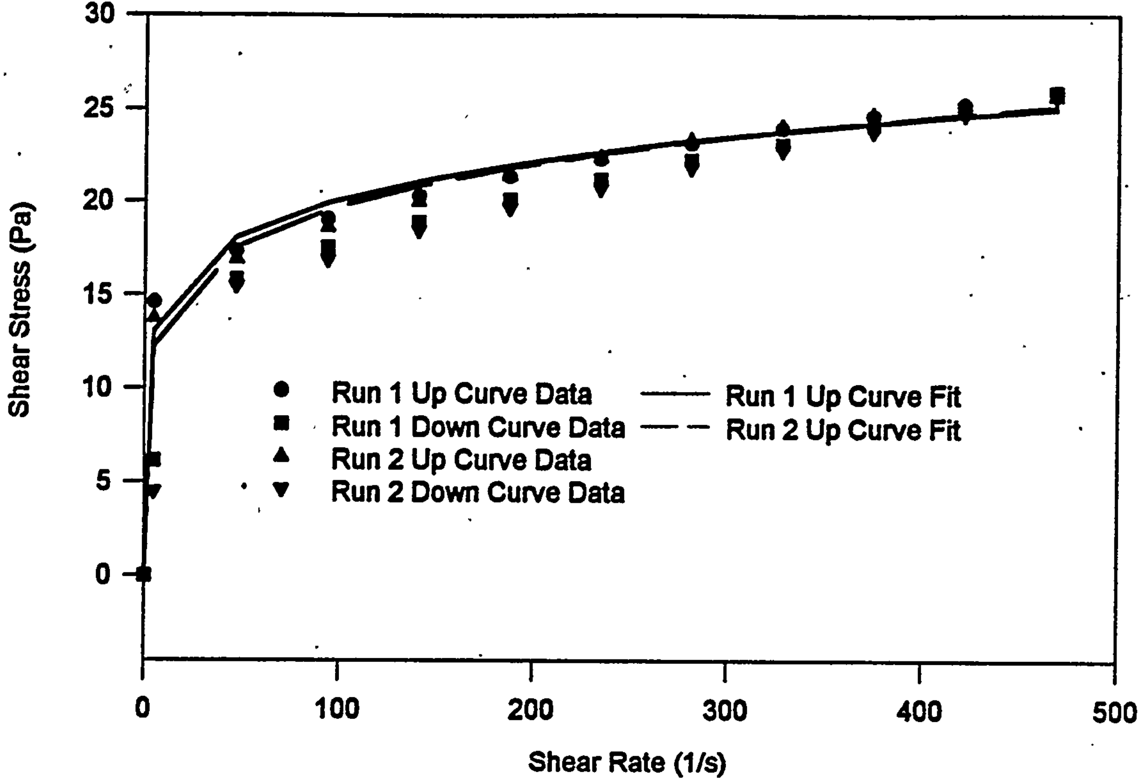


Figure D.47. Shear Stress versus Shear Rate of 102-AZ Core #1 Simulant Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

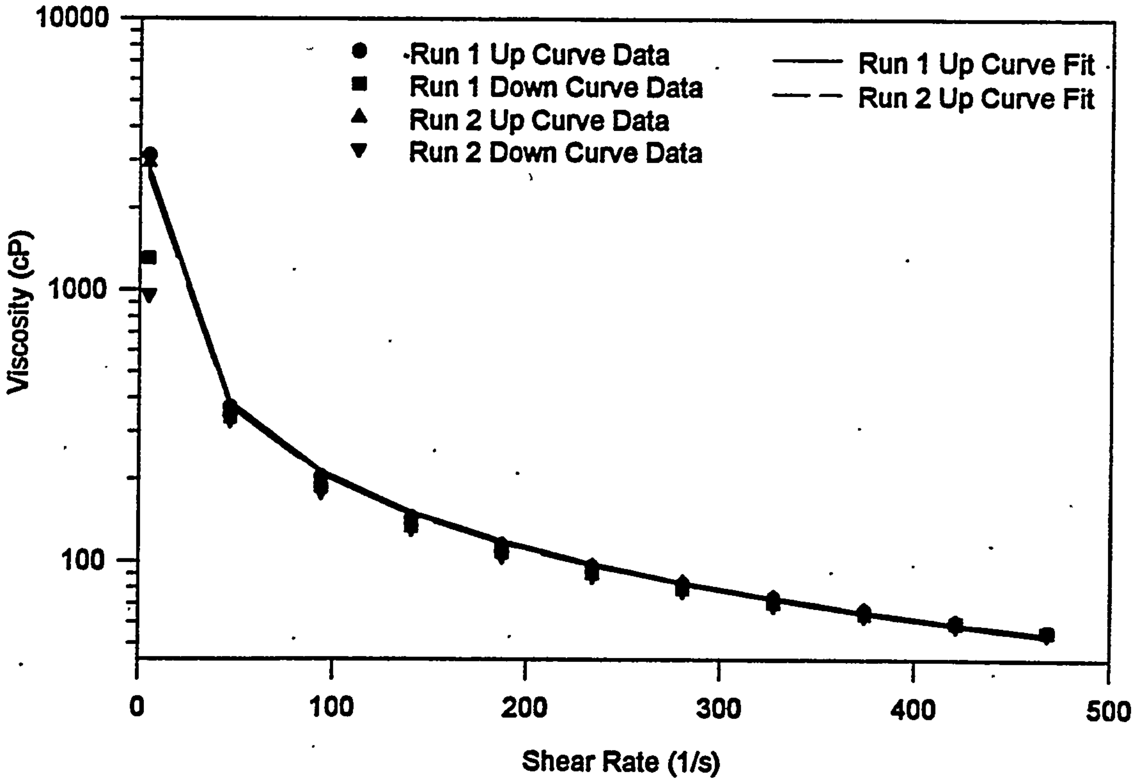


Figure D.48. Viscosity versus Shear Rate of 102-AZ Core #1 Simulant Melter Feed Slurry (All Runs) with Yield-Pseudoplastic Model Curve Fits

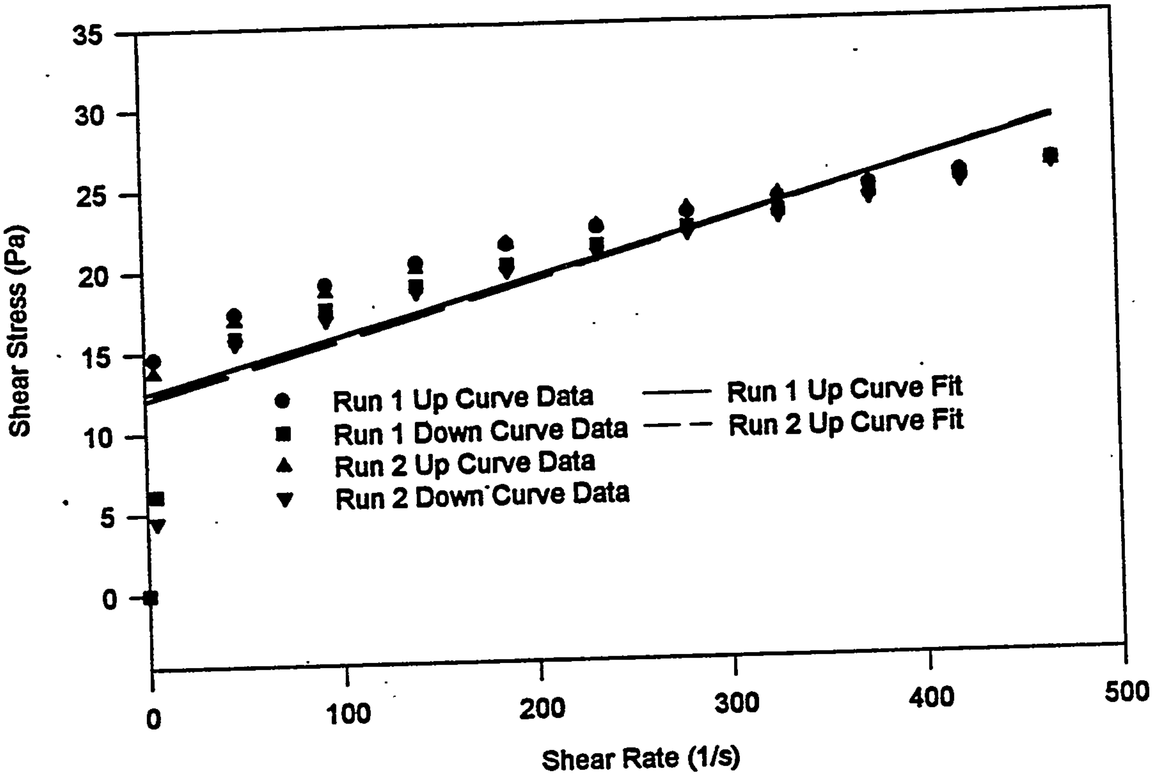


Figure D.49. Shear Stress versus Shear Rate of 102-AZ Core #1 Simulant Melter Feed Slurry (All Runs) with Bingham Curve Fits

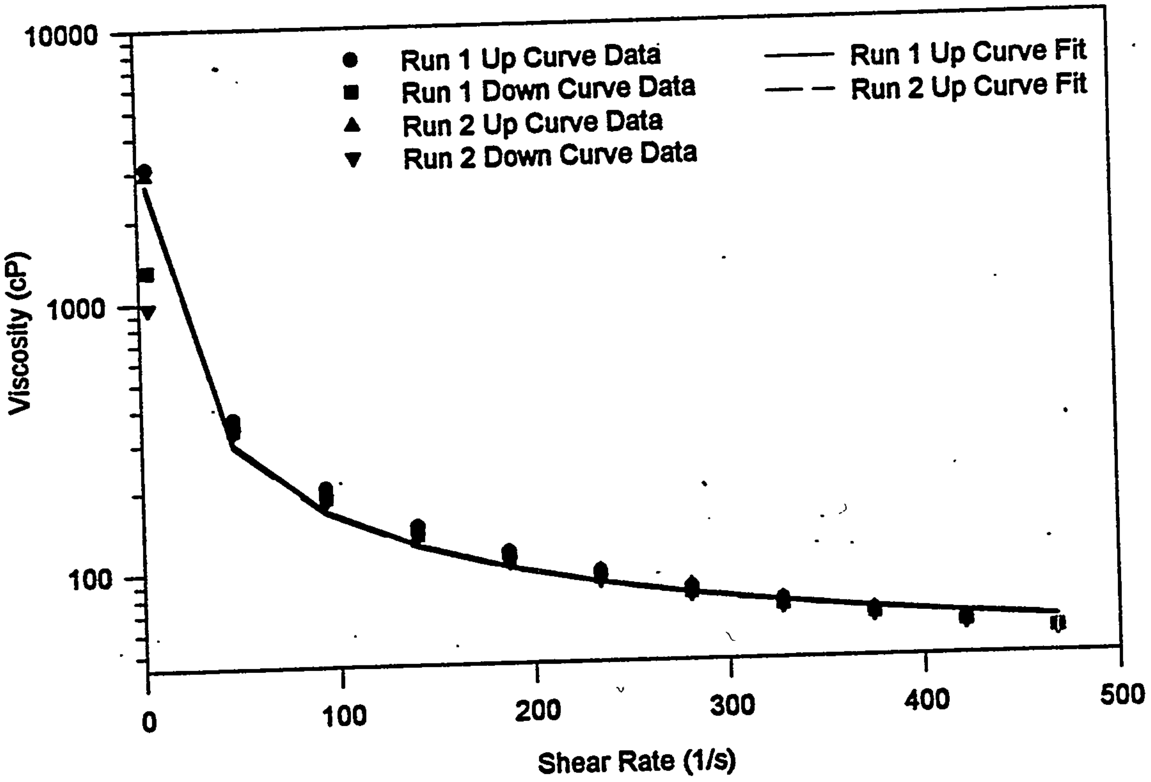


Figure D.50. Viscosity versus Shear Rate of 102-AZ Core #1 Simulant Melter Feed Slurry (All Runs) with Bingham Curve Fits

Table D.1. Table of Bingham and Yield-Pseudoplastic Curve Fits

Equations:
Yield-Pseudoplastic $\tau = \tau_0 + k\gamma^n$
Bingham Plastic $\tau = \tau_0 + n_p\gamma$

Reference	Figure	Run No. and Type	k	n	τ_0	r^2	τ_0	n_p	r^2
101-1 FS	Figure 1	R1 - up	9.25E-03	0.8293	0.0558	0.854	-	-	-
		R1 - down	1.41E-03	1.1216	3.71E-10	0.868	-	-	-
	Figures 2-5	R1 - up	9.25E-03	0.8293	0.0558	0.854	0.1638	3.08E-03	0.853
		R2 - up	6.86E-07	2.2136	0.3816	0.188	0.2986	1.10E-03	0.143
		R3 - up	0.0106	0.8305	0.1340	0.900	0.2573	3.55E-03	0.897
101-1 MF	Figure 6	R1 - up	5.2303	0.2335	2.86E-08	0.914	-	-	-
		R1 - down	2.2960	0.3229	4.3996	0.997	-	-	-
	Figures 7-10	R1 - up	5.2303	0.2335	2.86E-08	0.914	9.3782	0.0333	0.715
		R2 - up	3.5912	0.2391	9.26E-09	0.927	6.3806	0.0235	0.623
		R3 - up	2.4803	0.2434	5.63E-09	0.924	4.2537	0.0177	0.642
101-2 FS	Figure 11	R1 - up	0.0430	0.5183	0.0360	0.986	-	-	-
		R1 - down	8.57E-04	1.1600	2.01E-11	0.991	-	-	-
	Figures 12-15	R1 - up	0.0430	0.5183	0.0360	0.986	0.2323	2.00E-03	0.874
		R2 - up	3.92E-03	0.9302	0.0672	0.991	0.0626	2.47E-03	0.981
101-2 MF	Figure 16	R1 - up	1.2372	0.1594	3.28E-10	0.929	-	-	-
		R1 - down	5.07E-03	1.0095	0.9416	0.991	-	-	-
	Figures 17-20	R1 - up	1.2372	0.1594	3.28E-10	0.929	1.8301	4.07E-03	0.556
		R2 - up	0.6640	0.1796	0.0735	0.887	9.5405	3.12E-03	0.745
102-1 FS	Figure 21	R1 - up	3.63E-03	0.9046	0.0651	0.989	-	-	-
		R1 - down	6.56E-04	1.1906	4.13E-11	0.996	-	-	-
	Figures 22-25	R1 - up	3.63E-03	0.9046	0.0651	0.989	0.0850	2.01E-03	0.987
		R2 - up	2.50E-03	0.9971	0.0123	0.986	0.0132	2.46E-03	0.986
102-1 MF	Figure 26	R2 - up	0.7955	0.2155	0.0635	0.972	-	-	-
		R2 - down	4.27E-03	1.0059	1.1110	0.998	-	-	-
	Figures 27-30	R1 - up	0.7955	0.2155	0.0635	0.972	1.4289	4.39E-03	0.800
		R2 - up	9.71E-03	0.8356	0.4227	0.979	0.4925	3.50E-03	0.975
		R3 - up	0.0516	0.5821	0.0982	0.989	0.3527	3.75E-03	0.952
NCAW Sim FS	Figure 31	R1 -up	0.2323	0.4307	0.3503	0.976	-	-	-
		R1 - down	0.1134	0.5407	0.3651	0.982	-	-	-
	Figures 32-35	R1 -up	0.2323	0.4307	0.3503	0.976	1.2033	5.92E-03	0.919
		R2 - up	0.2671	0.3948	0.2023	0.945	1.0209	5.48E-03	0.880
NCAW Sim MF	Figure 36	R1 - up	0.1089	0.6989	1.2075	0.929	-	-	-
		R1 - down	0.0864	0.6979	1.9392	0.998	-	-	-
	Figures 37-40	R1 - up	0.1089	0.6989	1.2075	0.929	2.2114	0.0157	0.914
		R3 - up	2.06E-07	2.8092	2.7675	0.989	1.6551	0.0126	0.852
		R5 - up	0.0184	0.9954	2.4358	0.982	2.4476	0.0178	0.982
102-1 Sim FS	Figure 41	R1 - up	0.0414	0.7264	0.9128	0.999	-	-	-
		R1 - down	0.0555	0.6807	0.8327	1.000	-	-	-
	Figures 42-45	R1 - up	0.0414	0.7264	0.9128	0.999	1.2109	8.07E-03	0.989
		R2 - up	0.0413	0.7244	0.9197	0.999	1.2171	7.95E-03	0.989
102-1 Sim MF	Figure 46	R1 - up	10.3945	0.1418	0.1469	0.989	-	-	-
		R1 - down	5.2075	0.2597	3.05E-09	0.991	-	-	-
	Figures 47-50	R1 - up	10.3945	0.1418	0.1469	0.989	12.4420	0.0343	0.617
		R2 - up	9.5468	0.1565	0.1626	0.991	12.0685	0.0354	0.641

APPENDIX E

Summary of Test Plans and Procedures

Table E.1. Summary List of Procedures

Leaching Tests of Radioactive Glass, Technical Procedure MCC-TP-18, Rev. 0.

Leaching Tests Using the PCT Method, Technical Procedure MCC-TP-19, Rev. 1.

Operating Procedure for HWVP Off-Gas Analyzers (Radioactive Lab-Scale), PNL Technical Procedure PHTD-WTC-006-37, Rev. 0.

Procedure for Treating Slurries with Formic Acid, Adding Glass Frit and Drying the Slurry for Vitrification, PNL Technical Procedure WTC-053-2, Rev. 0.

Preparation of Special Sample Forms, MCC Technical Procedure MCC-TP-3, Rev. 0.

Laboratory Procedure for Measurement of Physical and Rheological Properties of Solution, Slurries and Sludges, PNL Technical Procedure PNL-ALO-501, Rev. 0.

Glass Preparation, MCC Technical Procedure MCC-TP-2, Rev. 0.

Procedure for Treating Slurries with Formic Acid, Adding Glass Frit and Drying the Slurry for Vitrification, PNL Technical Procedure PHTD-WTC-006-038, Rev. 0.

Procedure for Preparation of Simulated HWVP Feed for Laboratory-Scale Redox/Rheology and Radioactive Process/Product Tests, Technical Procedure WTC-006-36, Rev. 0.

This list includes all technical procedures specifically relating to the radioactive testing in this study. Standard technical procedures utilized by the analytical chemistry laboratory and CVS glass laboratory are not included in this list.

APPENDIX F

Data Reduction Methods

F.1 Rheology Calculations (Theory and Calculations)

Apparent viscosity is shear stress divided by the shear rate at a given shear rate. In a Newtonian fluid this is a linear function; therefore, the ratio is the same at every point. Only Newtonian fluids have a single number viscosity. A fluid whose apparent viscosity varies with shear rate is non-Newtonian. In these fluids, apparent viscosity defines only the flow behavior at a fixed point.

To define a complete flow behavior, it is necessary to obtain a rheogram (or flow diagram). A rheogram is a curve that plots shear stress versus shear rate over a specified shear-rate range. The shape of this curve defines the flow behavior of the fluid. If the fluid behaves as a Newtonian fluid, the curve is linear. A non-Newtonian curve is non-linear and can be difficult to fit mathematically. Most curves are a variation of a known behavior, for which mathematical models exist to fit the curves.

Theoretically, the yield stress or yield point is the minimum amount of stress required to initiate fluid movement and begin flow. In practice, the yield stress is obtained from the stress value on the graph where the slope changes from vertical to horizontal. A curve fit is used to extrapolate this intersection back to the shear-stress axis. The measured yield stress is not a true yield stress because the Haake rheometer used was a controlled-rate instrument, not a controlled-stress instrument; therefore, by definition it is forcing the material to flow for a reading to occur. A true yield stress is stress-dependent only; the yield stresses obtained by this system will be both rate- and stress-dependent. This measured yield stress is, however, often a good approximation of the desired data.

The rheometer "ramps up" the shear rate to a chosen value and records the resulting shear stress. The curve obtained from this test is called a rheogram. From a rheogram, viscosity data, yield-stress data, and flow-curve data can be obtained. There are several types of flow curves that have been well studied and have defined mathematical curve fits. These curve fits are usually used to describe and predict the flow behaviors of fluids.

Two primary curve fits, yield pseudoplastic or Herschel-Bulkley and Bingham plastic, were used in data analysis. The Bingham Plastic model is a good model for obtaining yield points. It assumes a Newtonian flow after a threshold yield stress has been overcome.

$$\tau = \tau_0 + \eta_p \gamma \qquad \text{(F.1) (Bingham Plastic)}$$

where:

τ = shear stress
τ_0 = yield stress factor
η_p = plastic viscosity
γ = shear rate.

The Herschel-Bulkley model describes a typical yield pseudoplastic. It is a modification of a power law fluid curve fit, which takes into account a yield stress. The exponential component of the equation quantifies the non-Newtonian behavior. The closer the exponent to 1.0, the more Newtonian the behavior. When n=1, the equation becomes a Bingham fit.

$$\tau = \tau_0 + k\gamma^n \qquad \text{(F.2) (Herschel-Bulkley)}$$

where:
τ = shear stress
τ_0 = yield stress factor
k = consistency factor
γ = shear rate
n = measure of deviation from Newtonian flow.

Pseudoplastic liquids show an apparent viscosity decrease as the shear rate is increased. This is known as "shear thinning" and is a common fluid behavior for emulsions, suspensions, and dispersions. Under shear conditions, particles/molecules acquire an orientation more conducive to flow, which lowers the viscosity. For a classic pseudoplastic liquid, shear thinning is reversible. As the shear rate drops, the apparent viscosity climbs with little or no lag time since the orientation of particles/molecules will be lost as quickly as it was achieved. Thixotropy is more complex because it usually involves bonding, which creates a three-dimensional structured network. This structure is easily broken down under the influence of shear, but will begin rebuilding as soon as the shear is removed. If a thixotropic liquid is measured at a constant shear rate, the apparent viscosity will drop asymptotically with time until it reaches the lowest viscosity achievable at that shear rate. This is called the sol-state. The time to rebuild varies, depending on the material. In practical terms, it takes less energy to maintain a flow than the energy it took to achieve it.

Special Notes with Respect to Data

Scatter is evident in some of the rheograms. One of the most likely causes for this is that the solids loading the slurries prevented the use of the most appropriate sensor. The measured viscosities of the slurries is below the recommended range of the sensor used over much of the shear-rate range. Data are particularly inaccurate when both the shear stress and the shear rate are low. For this reason apparent viscosity numbers below 50 s^{-1} are suspect. The larger particles may be responsible for the spiked nature of the scatter in some of the runs.

The small amounts of sample available, combined with the difficulty of hot cell measurements necessitated that duplicates of samples originally measured be rerun. These duplicates usually showed a lower yield point and viscosity. In both types of slurries, this can be a result of particle sedimentation out of the measuring gap. In the melter feeds (fritted slurries) it can also be attributed to the fact that melter feed is often a thixotropic yield pseudoplastic, which means that its rheological character changes after shear forces have been applied.

Nonradioactive laboratory-scale testing with simulants indicates that the rheology of formated slurries is fairly stable with respect to time. No significant change has been observed even when the slurries have been allowed to sit for several months (Beckette et al. 1994).

F.2 Normalized Release

Elemental and isotopic normalized releases were calculated for all glasses tested using the PCT and MCC-1 durability tests. Calculated and predicted values from simulants and models were then compared to data from the radioactive glass to determine whether or not the simulants and models accurately represented radioactive glass behavior. Although the limited quantity of radioactive glass available and the relatively high cost of performing radioactive tests limited the amount of replicate samples and tests performed, a practical statistical comparison of the simulants and models to radioactive glass was attempted. Additional statistical comparisons were made to determine if the difference in test conditions between the laboratory and the hot cell was significant.

As shown in Appendix A, Tables A.10 through A.12, radioactive glasses were tested in triplicate using both PCT and MCC-1 durability tests. Duplicate blanks were also included in each test involving radioactive glass. (A blank is a test vessel filled with leachant but with no glass specimen present.) Duplicate blanks were included primarily for redundancy in case one was spilled, but also provided a measure of variability for blank concentrations. Due to difficulties in performing such work in the hot cell, one blank was spilled in hot-cell test #4.

Simulant glasses were tested in duplicate in laboratory durability tests and in triplicate or quadruplicate in hot-cell tests. Quadruplicate samples resulted from duplicate simulant samples prepared in the hot cell and duplicate samples prepared in the laboratory but tested in the hot cell. This was performed to check for differences in preparation techniques (i.e., crushing, sieving, washing, drying) that could affect results. Durability tests performed in the laboratory included either one or no blanks.

For each sample, the normalized elemental mass loss of the i-th element was computed for the j-th specimen using the following equation

$$NL_{ij} = \frac{(C_{ij} - B_i)}{f_i\,(S_j / V_j)} \qquad \text{(F.3)}$$

where NL_{ij} = normalized loss of element i from specimen j

C_{ij} = concentration of element i observed in leachate from specimen j

B_i = concentration of element i observed in leachate from blank (averaged before subtracting when there is more than one blank)

f_i = average mass fraction of element i in the specimen

S_j = surface area of specimen j

V_j = initial volume of leachant in test vessel containing specimen j.

Note in Equation (2) that C_{ij} and S_j/V_j were measured from each specimen, whereas B_i and f_i were constant over the specimens at a particular set of test conditions. Thus, if the NL_{ij} values were substituted into the usual formula for computing a standard deviation, the uncertainty due to the B_i and f_i values would not be determined. It was necessary to use a standard deviation estimate that accounted for the variability in all of the quantities in Equation (F.3). Additionally, it would be valuable to account for long-term and inter-laboratory variabilities (i.e., variabilities resulting from analyses and testing performed at different times and by different laboratories). Although we wanted to compare results of glasses tested at different times by different technicians, sufficient data were not collected to allow calculation of these types of uncertainty.

By applying the propagation of errors method as presented by Bowen and Bennett (1988), an approximate formula for the variance of NL_{ij} was derived[a] and is given by

$$\mathrm{Var}(NL_{ij}) \cong (NL_{ij})^2 \left[\frac{\mathrm{Var}(C_{ij}) + \mathrm{Var}(B_i)}{(C_{ij} - B_i)^2} + \frac{\mathrm{Var}(f_i)}{f_i^2} + \frac{\mathrm{Var}(S_j/V_j)}{(S_j/V_j)^2} - \frac{2\,\mathrm{Cov}(C_{ij}, S_j/V_j)}{(C_{ij} - B_i)(S_j/V_j)} \right]$$

Each term in Equation (F.4) provides a measure of the contribution of a particular source of random error to the variance of NL_{ij}. $\mathrm{V\hat{a}r}\ (NL_{ij})$ is a measure of the single laboratory variance of NL_{ij}; that is, its square root (the standard deviation of NL_{ij}) is a measure of the extent to which a single laboratory can be expected to reproduce its own results when using replicate specimens of the specified glass under a particular set of conditions. Note that this quantity is not an indication of the capability of a laboratory to reproduce its own results when the test is repeated at different times, or of the expected agreement among the results from multiple laboratories that apply the test method under prescribed similar conditions.

An estimate of Var (NL_{ij}) is given by

$$\mathrm{V\hat{a}r}\ (NL_{ij}) = NL_{ij}^2 \left[\frac{\mathrm{V\hat{a}r}\ (C_{ij}) + \mathrm{V\hat{a}r}(B_i)}{\mathrm{Ave}\ (C_{ij} - B_i)^2} + \frac{\mathrm{V\hat{a}r}\ (f_i)}{f_i^2} + \frac{\mathrm{V\hat{a}r}\ (S_j / V_j)}{\mathrm{Ave}\ (S_j / V_j)^2} \right] \qquad \text{(F.5)}$$

(a) Olson, K. M., M. L. Elliott, J. W. Shade, and H. D. Smith. 1990. *Letter Report: Fabrication, Characterization, and Evaluation of West Valley Sludge Glass-1, A Fully Radioactive Glass Made with High-Level Waste from the West Valley Demonstration Project*. Prepared by the Pacific Northwest laboratory, Richland, Washington.

where

NL_{ij} = the average (over replicate specimens) of the NL_{ij} values

$\hat{\text{Var}}(C_{ij})$ = the sample variance of the C_{ij} values

$\hat{\text{Var}}(B_i)$ = the sample variance of the B_i values

$\text{Ave}(C_{ij} - B_i)$ = the average (over replicate specimens of the blank-corrected concentration values

$\hat{\text{Var}}(f_i)/f_i^2$ = the relative sample variance of f_i, which can be computed directly from replicate glass analytical data or from prior computations of relative standard error (RSD). The value is equal to the square of the RSD of the mean wt% oxide value for the i-th element

$\hat{\text{Var}}(S_j/V_j)$ = the sample variance of the S_j/V_j values over the replicate specimens

$\text{Ave}(S_j/V_j)$ = the average (over the replicate specimens) of the S_j/V_j values

Note that there is a direct correspondence between the sample estimates in Equation (F.5) and the quantities in Equation (F.4), with one exception. The last term in Equation (F.4) involves Cov $(C_{ij}, S_j/V_j)$, the covariance of C_{ij} and S_j/V_j over the replicate specimens at a set of test conditions. For computational convenience, this term was neglected, which will result in slight over-estimation of the standard deviation of the NL_{ij} values. This is because Cov $(C_{ij}, S_j/V_j)$ is positive; that is, when S_j/V_j increases, the concentration of the i-th element in the leachate should increase. Therefore, including an estimate of this term in Equation (F.5) would result in subtraction of a positive quantity, yielding a smaller standard deviation estimate than the computationally simpler estimate given by the above form of Equation (F.5).

The estimated standard deviation for NL_{ij} is calculated as the square root of the variance computed from Equation (F.5). Estimated relative standard deviations are calculated as

$$RSD_{ij} = \left(\hat{\text{Var}}(NL_{ij})\right)^{0.5} / \overline{NL_{ij}} \qquad \text{(F.6)}$$

and percent relative standard deviations (%RSDs) is the same value expressed in percent.

Simplifying assumptions were made in calculating $\hat{\text{Var}}(NL_{ij})$ when insufficient data were present to make a complete calculation or when the effect on $\hat{\text{Var}}(NL_{ij})$ was determined to be insignificant.

(1) Some of the laboratory tests did not include blank samples, preventing the calculation of $\hat{\text{Var}}(B_i)$. This contribution was ignored, because it is insignificant when compared to $\hat{\text{Var}}(C_{ij})$ and $\hat{\text{Var}}(f_i)$ in the major components reported in laboratory tests (i.e., B, Li, Na, Si). One

exception to this is silicon, in the case of fused-silica-lined leach containers. For this reason, the blank samples were included in all tests conducted with fused-silica liners.

(2) Glass compositions were not measured for the three core sample simulants. In general, "as-batched" compositions are expected to be more accurate than measured compositions and are therefore used for f_i in the Equation (F.3) calculations. Results from six replicate analyses of CVS1-19 glass (Hrma, Piepel, 1994, Table A.10) show RSDs for B, Li, Na, and Si between 2 and 5 percent. A conservative value of 5% RSD was chosen in estimating $\hat{Var}(f_i) / f_i^2$ for these three core sample simulants (i.e., $\hat{Var}(f_i) / f_i^2$ is estimated at 0.0025).

(3) Only single radiochemical analyses were performed on each of the radioactive glasses. In the absence of replicate data, analytical error estimates for counting, calibration, and handling were used to calculate $\hat{Var}(f_i) / f_i^2$ (see Appendix B, Tables B.1 through B.3 for actual values).

(4) Variance contributions from surface area-to-volume ratio were found to be insignificant (no change to RSD to three significant digits) when compared to other sources of variability and were consequently neglected. Similarly, this justifies neglecting the Cov (C_{ij}, S_j/V_j) term in Equation (F.4).

Statistical comparisons of core sample normalized release to those of the simulants were made. Hypothesis tests are performed to compare the mean ln transformed normalized releases (mean ln NL_{ij}) for B, Li, and Na, using their respective ln transformed standard deviations (SD mean ln NL_{ij}). The t-distribution was used to compare two means calculated from small samples sizes from populations with unknown variances. Implicit assumptions of this test were as follows: (1) normality for both populations; (2) populations with equal variances; and (3) statistically independent observations. The natural log transformed data were used instead of the untransformed data to satisfy the normality assumption of the t-test. Distributions of untransformed normalized release measurements are known to be non-normal (i.e., skewed to the higher releases). Transforming the data to the natural log form produced a normal distribution, which can be used for statistical comparison. The mean ln (NL_{ij}) represented more of a median than a mean of the data, but in most cases was very close to the mean. The assumption of equal variances was reasonable but is not verified.

Calculation of the t statistic and degrees of freedom for performing a t-test was as follows:

$$t = \frac{\overline{\ln(NL_{ij})_1} - \overline{\ln(NL_{ij})_2}}{\sqrt{\frac{(n_1 - 1)\hat{Var}(NL_{ij})_1 + (n_2 - 1)\hat{Var}(NL_{ij})_2}{n_1 + n_2 - 2}} \cdot \sqrt{\frac{n_1 + n_2}{n_1 n_2}}} \qquad \text{(F.7)}$$

where n_1 = number of replicate leachate samples for radioactive glass
n_2 = number of replicate leachate samples for simulant glass.

Equation (F.7) is not strictly correct because the means being compared are actually calculated numbers that represent several sources of variability and the degrees of freedom were determined only from the number of leachate analyses. However, this method approximated a statistical comparison between sample means and was adequate for our application.

Similar comparisons of sample means were made between samples of similar glasses tested under different conditions (e.g., differing leach containers). In cases where the data being compared were generated from a single source of glass, the $V\hat{a}r\ (f_i)$ was ignored in equation (F.5). This was justified since the difference in the data was not in any way associated with analytical uncertainty of the glass.

Ninety-five percent confidence intervals were calculated for each of the ln transformed means

$$Pr\left[\overline{\ln NL_{ij}} - t_{n-1,\alpha/2} * \frac{\sqrt{V\hat{a}r(NL_{ij})}}{\sqrt{n}} < \mu < \overline{\ln NL_{ij}} + t_{n-1,\alpha/2} * \frac{\sqrt{V\hat{a}r(NL_{ij})}}{\sqrt{n}}\right] = 1 - \alpha$$

using the following equation

The mean ln NL_{ij}s and confidence intervals were then untransformed to be represented graphically. Although the untransformed mean ln NL_{ij} values were slightly lower than the mean NL_{ij} values, the two were probably not discernibly different in graphical form and the confidence limits can be represented.

APPENDIX G

Description of Analytical Methods

G.1 Physical Characterizations

In addition to the quantitative physical characterizations described below, the physical appearance of the waste was documented after extrusion and throughout the processing steps. The descriptions included physical state (solution, slurry, or sludge), color, ability to hold its shape, and any other observable physical characteristics.

G.1.1 Density

Solid samples were placed in preweighed, volume-graduated, centrifuge tubes where they were weighed and then centrifuged for 1 h at >1000 gravities to remove voids. This ensured accurate volume measurements and allowed division of the sample mass by the sample volume to obtain density.

Liquid samples were placed in a preweighed volumetric flask up to the volume line, weighed, and the density was calculated.

Pre-weighed glass bar samples were placed in a graduated cylinder, volume measured from displacement, and density calculated.

G.1.2 Solids Settling Rate and Volume Percent Settled Solids

Settling rates and vol% settled solids measurements were conducted in preweighed, volume-graduated, centrifuge tubes where total volume and the volume of settled solids (i.e., level of visual interface were recorded periodically. The cross-sectional area in the upper portion of the centrifuge tube was constant, thus allowing the conversion of settling rate data from mL/h to cm/h.

After the settling rates were determined, the vol% settled solids were calculated by dividing the final settled solids volume by the total sample volume.

G.1.4 Volume Percent and Weight Percent Centrifuged Solids and Supernatant

Following the settling-rate measurements above, the samples were centrifuged for 1 h at >1000 gravities. Total sample volume and solids volume were determined using the graduations on the centrifuge tubes, thus allowing the volume percentages of both solids and supernatant to be calculated.

Weight percentages of the centrifuged solids and supernatant were determined by decanting or pipeting the centrifuged supernatant into a preweighed vial. This allowed weighing supernatant and the solids remaining in the centrifuge tubes independently. The weight percentages of each were then calculated.

G.1.5 Weight Percent Total Solids, Total Oxides, and Dissolved Solids

Samples were placed into preweighed crucibles, weighed, and allowed to air-dry overnight to remove free liquid to prevent splattering in the oven. Then the samples were transferred to a drying oven at 105 ± 5°C where they were dried for 24 h. The dried samples were removed from the oven, placed in a desiccator to cool to room temperature, reweighed, and the weight percent total solids was calculated.

Next, the dried solids were placed in a furnace at 1025 ± 25°C for 1 h to convert the chemicals in the sample to their stable oxide form. The samples were removed from the furnace, placed in a desiccator to cool to room temperature, reweighed, and the weight percent total oxides was calculated.

Determinations of the weight percent dissolved solids were performed with supernatant samples in the same manner as described above for the measurement of wt% total solids slurry samples. The percent dissolved solids in the supernatant was multiplied by the percentage of supernatant in the sample (1 - wt% centrifuged solids) to arrive at the percentage of dissolved solids in the total sample.

G.1.6 Particle Size

A Brinkman Model 2010 Particle Size Analyzer was used to determine the distribution of particle sizes in the solid samples. This instrument determines particle sizes in the range 0.5 μm to 150 μm by measuring the time required for a rapidly moving laser beam to traverse selected particles maintained in stirred suspension in a glycerin/water mixture. Two measurements on the same sample several days apart yielded nearly identical results, indicating that the glycerin/water mixture does not partially dissolve the sample and thus reduce the particle sizes.

G.1.7 Shear Stress Versus Shear Rate

Shear stress versus shear rate data were used to evaluate the viscosity of a fluid. The data were generated in the form of a rheogram or flow curve, which is a plot of shear stress as a function of shear rate. The rheograms were obtained at room or hot cell temperature using a Haake RV 100 viscometer equipped with an M5 measuring-drive head and the MV1 sensor system.

The measurement of viscosity with this instrument required that the sample be placed in the gap between two coaxial cylinders. About 40 mL of sample was thoroughly agitated and transferred into the cylinders. When the system was set in motion, a viscosity-related torque, caused by the sample's resistance to shearing, acted on the inner cylinder. This torque deflected a measuring device which was correlated to the shear stress value. A slurry with a yield stress "clamped" the rotor to the cup until the applied torque exceeded the yield stress.

G.2 Chemical Characterization

Chemical and radiochemical analyses were performed on both liquids (e.g., supernatant) and solids (e.g., sludge). Analyses of liquid samples were performed directly on the liquids or dilutions thereof. Analyses of solid samples were performed on solutions prepared from the solids according to the following methods.

Solid samples were prepared for ICP/AES analyses and most of the radiochemical analyses by fusing the solids in molten salt and then dissolving the fused solids in hydrochloric acid. Two fusions were made; one used Na_2O_2 in a Zr crucible (so-called Na/Zr fusion) and the other used a mixture of

KNO_3 and KOH in a Ni crucible (so-called K/Ni fusion). These two separate fusions were required to allow determination of Na, K, Ni, and Zr in the samples. Following fusion, the samples were dissolved in HCl solution and diluted to a known volume.

Solids samples were prepared for IC and tritium analyses by leaching a weighed amount (~1 g) of solids in a measured volume (~100 mL) of water for 1 h while agitating ultrasonically. The leachate was then filtered using 0.45-μm filters and analyzed. The rationale for this method of preparation is that all of the anions that can be analyzed by the IC method, as well as the tritium, were expected to be dissolved nearly quantitatively from the solids by the water leaching treatment whereas fusion would have volatilized or destroyed some of the analytes.

G.2.1 pH

Standard laboratory procedures were used to measure the pH of supernatants and slurries without any pretreatment. Before use, the pH electrode was calibrated employing a two-standard method that took into account the temperatures of the standards and samples.

G.2.2 Carbon Analyses

Carbon analyses were performed directly on both solid and liquid samples. Liquid samples were analyzed by the ultraviolet-catalyzed persulfate oxidation method. First, they were acidified to drive off the inorganic carbon as CO_2, which was swept away in a gas stream and measured in an infrared detector. Next, potassium persulfate ($K_2S_2O_8$) was added to the liquid samples where oxidation of the organic carbon to CO_2 was catalyzed, at ambient temperature, by ultraviolet light. The CO_2 was swept away in a gas stream and measured in an infrared detector, just as with the inorganic carbon.

Solid samples were analyzed by one of two methods, the total combustion method and the hot persulfate oxidation method.

In the total combustion method, the organic carbon was oxidized in an oxygen gas stream at 600°C. The CO_2 thus formed was swept away by the gas stream and measured by a coulometric detector. Total carbon, both organic and inorganic, was determined by oxidizing the sample in an oxygen gas stream at 1000°C, which not only oxidized the organic carbon but also decomposed the inorganic carbonates to yield CO_2. Again, the CO_2 was swept away by the gas stream and measured by a coulometric detector.

A hot persulfate oxidation method was used to analyze some of the solids samples. It was similar to the UV-catalyzed oxidation method described above for liquids except that, after driving off the inorganic carbon with acid treatment, the organic carbon was oxidized by silver-catalyzed persulfate at 90°C. Both the inorganic CO_2 and organic CO_2 were swept away in a gas stream and measured in a coulometric detector.

As noted, some of the solid samples were analyzed by the combustion method and others by the persulfate method. After development and some experience with the persulfate method, it was generally considered to yield more satisfactory results. However, because of the mix of methods and initial inexperience with the persulfate method, some unsatisfactory results were obtained and have not been rerun. Thus, the reported data were not entirely complete and consistent.

Carbon-14 analyses were conducted on separate samples to which silver-catalyzed $K_2S_2O_8$ and sulfuric acid were added simultaneously and heated to 90°C. Organic carbon-14 was thereby oxidized to $^{14}CO_2$ which, along with any inorganic $^{14}CO_2$, was swept with carrier gas into a NaOH trap where it was quantitatively absorbed and analyzed using a liquid scintillation counter.

G.2.4 ICP/AES Analyses

ICP/AES analyses were performed directly on liquid samples or dilutions thereof. In the case of solid samples, both fusions were analyzed by ICP/AES to circumvent the obvious interferences by Na and Zr in the Na/Zr fusion and by K and Ni in the K/Ni fusion.

G.2.5 IC Analyses

IC analyses were performed directly on liquid samples or dilutions thereof. As stated in Section G.2, in the case of solid samples, IC analyses were performed on leachates from the solids.

G.3 Radiochemical Characterization

In the following descriptions, "prepared solutions" refers to the solutions prepared as described in Section G.2. Frequently, the radioactivities of the prepared solutions were too high and required further dilution before analysis. In the case of samples prepared from fusions, as described in Section G.2, radiochemical analyses were sometimes performed on both the Na/Zr and the K/Ni fusions and other times on just one of the fusions.

G.3.1 Gamma Energy Analysis

Gamma energy analysis (GEA) was used to measure the concentrations of most gamma-emitting isotopes in the prepared solutions.

G.3.2 Total α and Total β

Aliquots of prepared solutions were evaporated onto discs and then counted on α or β proportional counters, as appropriate.

G.3.3 Alpha Energy Analysis

Alpha energy analysis (AEA) was used to determine the concentrations of $^{243+244}Cm$, ^{241}Am, $^{239+240}Pu$, and ^{237}Np. Aliquots of prepared solutions were evaporated onto discs and counted with a solid-state α detector.

G.3.4 Total Pu and Pu Isotopes

To determine total Pu concentrations and Pu isotopic compositions, the Pu was first separated by ion exchange. The total α activity was measured using an α proportional counter, and AEA was used to determine the relative contributions of the different Pu α emitters. When a Pu isotopic analysis was requested, the relative concentrations of all of the different Pu isotopes were determined by mass spectrometry. Pu isotopic determinations were normally done only on samples with total Pu concentrations >0.4 μg/mL. Lower concentrations risked contamination and interference by ^{238}U, which was normally present in much higher concentrations. If a small amount of ^{238}U was carried along with the Pu in the ion exchange separation, the Pu isotopic results would be invalidated.

G.3.5 Total U and U Isotopes

Total U was measured on the prepared solutions using laser fluorimetry. Uranium isotopic composition was determined by mass spectrometry following separation of the uranium from solution using ion exchange.

G.3.6 Tritium Analyses

Because tritium was expected to be present in the form of water, water from the prepared solutions was purified by double distillation and then analyzed by liquid scintillation counting.

G.3.7 Other Radioisotopes

Each of the remaining radioisotopes (^{129}I, ^{99}Tc, ^{90}Sr, and ^{79}Se) to be measured was separated individually from the prepared solutions using either precipitation, ion exchange, or distillation methods and then measured by beta or gamma counting, or liquid scintillation counting.