**INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROSCOPY GLOVEBOX ASSEMBLY SYSTEM
AT THE WEST VALLEY DEMONSTRATION PROJECT**

Topical Report

By
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January 2000

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Prepared by
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ABSTRACT

The inductively coupled plasma/atomic emission spectroscopy [ICP/AES (ICP)] system for elemental analyses in support of vitrification processing was first installed in 1986. The initial instrument was a Jobin Yvon (JY) Model JY-70 ICP™ that consisted of sequential and simultaneous spectrometers for analysis of nonradioactive samples as radioactive surrogates. The JY-70 ICP™ continued supporting nonradioactive testing during the Functional and Checkout Testing of Systems (FACTS) using the full-scale melter with “cold” (nonradioactive) testing campaigns. As a result, the need for another system was identified to allow for the analysis of radioactive samples. The Mass Spec (Spectrometry) Lab was established for the installation of the modified ICP system for handling radioactive samples. The conceptual setup of another ICP was predicated on the use of a hood to allow ease of accessibility of the torch, nebulizer, and spray chamber, and the minimization of air flow paths. However, reconsideration of the radioactive sample dose rate and contamination levels led to the configuration of the glovebox system with a common transfer interface box for the ICP and the inductively coupled plasma-mass spectrometer (ICP-MS) glovebox assemblies. As a result, a simultaneous Model JY-50P ICP™ with glovebox was installed in 1990 as a first generation ICP glovebox system. This was one of the first ICP glovebox assemblies connected with an ICP-MS glovebox system. Since the economics of processing high-level radioactive waste (HLW) required the availability of an instrument to operate 24 hours a day throughout the year without any downtime, a second generation ICP glovebox assembly was designed, manufactured, and installed in 1995 using a Model JY-46P ICP™. These two ICP glovebox systems continue to support vitrification of the HLW into canisters for storage. The ICP systems have been instrumental in monitoring vitrification batch processing. To date, remote sample preparation and compositional analysis of key parameters, i.e., greater than 0.5% weight percent metal oxides, during batch processing (waste initials, glass formers, waste plus glass formers, and glass) has verified the composition to be within the acceptance criteria and generated the necessary data for the Waste Qualification Report.

1.0 INTRODUCTION

In 1982 West Valley Nuclear Services Company (WVNS) was awarded a contract by the U. S. Department of Energy (DOE) to solidify high-level waste (HLW) at the West Valley Demonstration Project (WVDP). This Demonstration Project was established for, among other things, the vitrification of the 600,000 gallons of radioactive HLW generated by Nuclear Fuel Services (NFS) while reprocessing spent nuclear fuel from 1966 to 1972. The HLW was stored in Tanks 8D-2 and 8D-4 and was composed of precipitated sludge and an alkaline supernatant. WVNS decontaminated the supernatant using an ion-exchange resin, zeolite, to remove cesium-137 (^{137}Cs) and solidify the resulting low-level radioactive waste as cement in drums. The ^{137}Cs -loaded zeolite was added to the HLW sludge. The mixture is currently being vitrified to produce canisters containing HLW glass that are suitable for storage in a federal repository. The vitrification process involves mixing the HLW with cold chemicals (glass formers) in the Concentrator Feed Makeup Tank (CFMT) and transferring the slurry to the Melter Feed Hold Tank (MFHT) prior to introduction to the melter which operates at 1,150° C. The vitrified melt is poured into 10' x 2' stainless steel canisters as glass logs. The canisters are sealed afterwards. The vitrification plan identified the generation of approximately 300 canisters from processing 100 batches of 8D-2 HLW. The plan also required the determination of the process recipe composition to produce a durable glass for storage in the federal repository. This required several test runs for the determination of the composition of the various waste and glass-former streams for operation of the process control plan. The composition at every stage of development was confirmed by analyses of key parameters. The confirmatory analyses led to the composition specified in the Waste Qualification Report, WVDP-186.

Several types of analytical instruments were evaluated by the Analytical and Process Chemistry (A&PC) Department for confirmatory analyses. The apparatus that had the most flexibility for providing accurate, reliable, and fast analytical support was the inductively coupled plasma/atomic emission spectroscopy (ICP) which possessed both simultaneous and sequential capabilities for the analysis of any analyte(s). Thus, the ICP system became the primary support for vitrification analysis. During simulated (nonradioactive) cold testing, cesium analysis in surrogate slurry posed a problem on the ICP system due to the cesium's low ionization potential. This was resolved by analyzing cesium on the atomic absorption spectrophotometer (AAS). However, in the vitrification campaign, the AAS is not required since the radioactive ^{137}Cs is being determined by gamma spectroscopy.

The first ICP instrument used at the WVDP was a Jobin Yvon (JY) Model JY-70 ICP™ that was composed of a 32-element simultaneous spectrometer (JY-32™) and a sequential monochromator (JY-38™). This system was installed in 1986 in the former ICP Lab for cold testing and operated as a nonradioactive instrument. Later on, custom designed systems were added for handling radioactive samples.

2.0 ICP SYSTEM FOR RADIOACTIVE OPERATIONS

The Societe Generale Pour Les Techniques Nouvelles (SGN) pneumatic system was installed in 1987 connecting the Supernatant Treatment System (STS) sampling aisle to the A&PC Sample Storage Cell (SSC). Due to the dose rate requirements, this system was installed to allow the remote transfer of radioactive samples in-cell. At the same time, a partial installation of a similar pneumatic transfer system was extended to the ICP Lab for interlaboratory HLW sample transfer. The transmitting end to the ICP Lab came from the Analytical Cell (AC) Aisle glovebox located between ACs 4 and 5. While this system of transferring samples remotely was installed and operational for the STS in 1987 and later for the vitrification pneumatic transfer system in 1996, the interlaboratory pneumatic transfer system was never put into operation due to safety considerations.

Originally, the existing ICP system, namely JY-70™, was considered for radioactive samples based on the initial anticipated dose and contamination level requirements that dictated the use of a radioactive fume hood for the JY-70™ along with the configuration of the torch box assembly. However, it was determined that the size and layout of the system with the right angle configuration of the simultaneous and sequential optical paths with the radio frequency (RF) generator were not conducive to the hood dimensions. This hood layout did not provide for ease of torch use and spectrometer accessibility. Hence, the evaluation of a dedicated laboratory room for the new generation ICP system that would address contamination control and operate safely was initiated. This evaluation also addressed the need for isotopic analysis by ICP/MS (mass spectrometer). As a result, another laboratory area (Mass Spec. Lab) was selected, where the ICP and ICP/MS hood and later glovebox apparatus would be located.

2.1 Engineering Controls

Starting from a partially empty laboratory in 1989 the Mass Spec. Lab area was set up to support not only the ICP assembly for slurry and glass compositional analyses, but also the ICP-MS assembly for slurry and glass radionuclide analyses that had been defined at that time. The design and layout of the system were originally done with a physical separation between the two assemblies, each with its own dedicated hood design concept as verified by Radiological Engineering (RE). However, the potential contamination concerns were reevaluated by RE which determined the need for a glovebox instead of a hood with each ICP assembly.

Therefore, the design and fabrication requisition for the ICP systems were modified to include the glovebox requirements. The changes were mainly for the glovebox air flow patterns for the torch and the glove ports' spatial requirements for physically handling all the hardware inside the glovebox. In addition, the layout of the instruments and gloveboxes was changed to allow a common transfer box interface at the end of each glovebox. This layout also allowed remote sample transfer flexibility to the gloveboxes using a pneumatic sample transfer system access between the AC Aisle glovebox and the ICP or ICP-MS glovebox. Although considered, the SGN transfer system design on the gloveboxes was not implemented due to cost and safety considerations. Instead, permission was obtained from RE to use the transfer drawer between Sample Cell 2 (SC2) and AC1 as the alternative procedure for removing cell items. As a result, the canister transfer system became the primary transfer source from the AC aisle glovebox to the ICP glovebox system.

3.0 FIRST GENERATION GLOVEBOX FOR ICP

In order to set up a “catch-all” system with wide capabilities, the design included the addition of several analytes to the spectrometer’s polychromator since the mask slits and photomultiplier tubes (PMTs) would be easier to add at the manufacturing plant than in the field. At that time the decision was to utilize a simultaneous instrument because the analysis turnaround time would be faster for 25 or 42 elements compared to using a sequential apparatus. This decision would also use less sample volume and minimize the handling time.

3.1 Specifications

The specifications for the ICP glovebox system involved simultaneous analysis of 42 analytes (Ag [silver], Al [aluminum], As [arsenic], B [boron], Ba [barium], Ca [calcium], Cd [cadmium], Ce [cerium], Co [cobalt], Cr [chromium], Cu [copper], Fe [iron], Hg [mercury], K [potassium], La [lanthanum], Li [lithium], Mg [magnesium], Mn [manganese], Mo [molybdenum], Na [sodium], Nd [neodymium], Ni [nickel], P [phosphorous], Pb [lead], Pd [palladium], Pr [praseodymium], Rh [rhodium], Ru [ruthenium], S [sulfur], Sc [scandium], Se [selenium], Si [silicon], Sm [samarium], Sn [tin], Sr [strontium], Te [tellurium], Th [thorium], Ti [titanium], U [uranium], Y [yttrium], Zn [zinc], and Zr [zirconium]) using a corrosive mixture matrix of 5% nitric acid (HNO_3), 5% hydrofluoric acid (HF), and 1 mL (30%) hydrogen peroxide (H_2O_2); a provision for cleaning the glovebox lens; the use of Central Research Laboratories’ (CRL) 203 millimeter ([mm] 8") glove ports and a 190 mm (7.4") Double Door Sealed System™ with canisters for transferring items without going through the common transfer interface box door; and the use of peristaltic pumps with provision for an autosampler. Also, specifications were incorporated for lights; inlet and outlet exhaust filtration; cooling systems for the load coil and plasma exhaust gases; vacuum interface systems for gases, water, and electrical cables; a common passthrough system; and shutdown capability of the plasma torch if the argon gas flow or negative exhaust was lost.

3.2 Scope of Analysis

The number of analytes was predicated on the initial vitrification process campaign requirements (Al, B, Ba, Ca, Ce, Co, Cr, Cu, Fe, K, La, Li, Mg, Mn, Mo, Na, Nd, Ni, P, Pd, Pr, Rh, Ru, S, Sc, Si, Sm, Sn, Sr, Te, Th, Ti, U, Y, Zn, Zr) developed by Process Development Engineering. Additional analytes (Ag, As, Cd, Hg, Pb, Se) were requested due to EPA (Environmental Protection Agency) TCLP (Toxicity Characteristic Leaching Procedure) derivative-type analyses that may be required.

3.3 Overall Dimensions

The glovebox overall dimensions were based partly on utilizing a pneumatic sample transfer system provided by SGN in 1987. This flange position for the sample transfer station was located on the top of the glovebox requiring an opening dimension of 235 mm (9.25") x 324 mm (12.8"). The flange was located to minimize interference while accessing the glovebox through the transfer box panel/door and canister transfer system. The plasma torch height and depth adjustment required an indentation in the corner near the spectrometer location (see Figure 1).

Figure 1. Plasma Torch Location on Glovebox Indentation

Figure 2. Location of Glovebox
Transfer Canister Cell Flange

Figure 3. JY-50PTM Glovebox Assembly

The double door transfer canister flange system was located diagonally opposite the torch box/peristaltic pump sample analysis positioning area (see Figure 2). The future requirement for an autosampler to minimize dose exposure also demanded a footprint. Therefore, the external dimensions of the glovebox were approximately 2000 mm (78.7") long, 900 mm (35.4") deep at bottom, 600 mm (23.6") deep at top, and 1050 mm (41.3") high. The glovebox was mounted on a tubular frame chassis (see Figure 3) with 10 degree sloped window sides for easier access inside the glovebox. One end of the glovebox was connected to the common transfer box door, 540 mm (21.2") wide and 390 mm (15.4") high, with the overall transfer box dimensions of 720 mm (28.3") long, 520 mm (20.5") high, and 856 mm (33.7") wide. The opposite glovebox end was for the optical channel pathway between the torch box plasma location (485 mm [19.1"] depth from front) inside the glovebox and the optical emission lens entrance to the simultaneous spectrometer. The spectrometer chassis and overall dimensions were 990 mm (39.0") wide, 864 mm (34.0") deep, and 1346 mm (53.0") high with an optical path channel dimension between the glovebox and spectrometer of 229 mm (9.02"). The total optical path length from the plasma to the primary slit is 838 mm (33.0").

3.4 Special Considerations

The JY-50PTM simultaneous spectrometer incorporated the polychromator which used a Paschen-Runge assembly configuration with the input slit, holographic diffraction grating, and output slits on the Rowland circle. The 3600 groves/mm grating for the spectrometer, with a 0.5-meter (m) focal length and a 1200 grooves/mm flat field grating with a focal length of 250 mm for the alkalis, were provided for the analyte choices. The use of corrosive acids required the utilization of a HF-resistant (dismountable) torch and a Ryton™ Scott-type spray chamber with the application of a pneumatic corrosion resistant Hildebrand™ nebulizer.

Figure 4. Transfer Interface Box

The transfer interface box (see Figure 4) had two 152 mm (6") glove port gloves and a top viewing window for transfer of material to and from the gloveboxes. The glove ports were manufactured by CRL™. A CRL™ glove transfer boot that allows the glove to be changed without breaking vacuum is used for replacing gloves that are damaged.

3.6 Autosampler Provisions

Provisions were made to utilize a small autosampler that was compatible with the glovebox door openings. Because of the number of wires required for the autosampler at that time and the other wires for power inside the glovebox, the installation of two vacuum compatible electrical interfaces allowed for a total of 36 wires to be used in the glovebox (see Figure 5). The use of nominal 20 mL scintillation vials as a way of providing samples from the analytical cells for analysis required a 14-rack system with the Gilson 221™ Autosampler (see Figure 6).

Figure 6. Gilson 221™ Autosampler—14-Rack Dispenser

3.5 Glove Ports

In order to provide flexibility for working inside the glovebox, 203 mm (8") diameter glove port gloves were installed, with a total of six glove ports in the rear and eight glove ports on the working front. The decision not to install another SGN pneumatic transfer system and other reconfigurations allowed the top center glove port on the back and the two top center glove ports on the front of the glovebox to be replaced with windows to provide more viewing area and less glove interference.

Figure 5. Electrical and Utility Vacuum Pass-throughs

3.7 Double Door Canister Transfer System

The CRL 190 mm double door canister transfer system was chosen because the internal dimensions (I.D.) were approximately 7" diameter and 9" high. A polyvinyl chloride (PVC) liner is used in each canister to minimize canister contamination and limit dose rates from beta emitters. This capacity allowed the transfer of samples, calibration and quality control (QC) standards (Std.), waste bottles, decontamination reagents, wipes, smears, material, and equipment from any hood and glovebox with similar transfer canister systems. In addition, this type of transfer design helped minimize contamination on the canister's external surface. The canister transfer is the primary means of transferring samples among the Radiochemistry Lab, ACs, and ICP gloveboxes.

3.8 Peristaltic Pump

Initially, two separate peristaltic pumps were used in the ICP glovebox scheme. One single-channel pump was used for sample introduction and another single-channel pump was used for waste products from the spray chamber. After two failures of the single-channel pumps, the Gilson Model Minipuls™ 3 two-channel peristaltic pump was installed. The two-channel peristaltic pump was easier to use from a power and footprint limitation.

3.9 Glovebox Lighting

Fluorescent lighting was used externally at the top of the front and back glovebox windows to provide lighting inside.

3.10 Inlet and Outlet Filtration

With the anticipated use of radioactive samples in 1991, the concern was to minimize glovebox exhaust manifold contamination in the Mass Spec. Lab since the Mass Spec. Lab manifold exhaust extension was newly installed in 1990. The glovebox in-line exhaust filter was installed to prevent downstream contamination of the new exhaust manifold system. The size capacity was 425 lpm ([liters per minute] 15 cubic feet per minute [cfm]). Additionally, a backflow preventer was installed in the glovebox inlet filter in case vacuum was lost or positive pressure resulted from the exhaust.

3.11 Interface System for Utilities

With the glovebox under vacuum, provisions were made for the power to the autosampler, peristaltic pumps, photocell for the torch RF feedback, electronic igniter, etc. The connections were provided by two vacuum electrical interfaces, each with 18 connectors. In addition, a glovebox vacuum utility interface was required for the water and gas lines to the glovebox (see Figure 5). The utility interface provided pass-throughs for the nitrogen purge for the optical path channel, argon gas lines to the torch, the sheath gas adapter, the nebulizer, and the cooling water lines to the plasma heat exchanger and the load coil. These connections were by Swagelok™ double quick-connects with a total capacity of ten ports.

3.12 Installation and Applications

The system was installed in the latter part of 1990. One of the challenges was the use of high efficiency particulate air (HEPA) filters in the glovebox with the implicit annual filter testing requirements. Interpretation was finally clarified so that the HEPA filters could be used without testing the HEPA filters annually. Closed loop systems were used for the cooling water for the load coil and the heat exchanger. The glovebox opening for the SGN pneumatic system was sealed with a herculite cover under the flange blank. This would allow a future option for installation of the SGN drop tube with minimal contamination. The alignment of the plasma torch system in the glovebox with the lens grating and PMTs in the spectrometer was done with a laser that was mounted on a demountable shelf leveler inside the glovebox.

4.0 LESSONS LEARNED WITH THE FIRST GENERATION ICP GLOVEBOX SYSTEM

- 4.1 Misalignment of the torch channel center point with the spectrometer grating/PMT center was due to movement of the spectrometer. The footings needed to be stabilized.
- 4.2 The distance from the glove ports to the torch needed to be shorter so that analysts could easily disassemble and change the torch system.
- 4.3 Relocation of the double door transfer system to the front of the glovebox was required to minimize the analyst's movement.
- 4.4 A drop tube for nonradioactive material, reagents, and calibration and QC Stds. was required to save transfer time and minimize the canister transfer and Radiological Control Technicians' (RCTs) support.
- 4.5 The relocation of lights was needed to decrease the glare.
- 4.6 No dry wiping or dry smears on the glovebox polycarbonate surfaces was allowed in order to eliminate scratching the surface. A wet wipe or wet smear is used instead.
- 4.7 A separate closed loop system for the load coil and for the condenser was required for easier control.
- 4.8 Optical path channels were required to be modified due to of the lens cleaning requirements.
- 4.9 An easier way to remove and install the demountable torch assembly in the torch box was required for reproducible alignment, such as a cassette system.
- 4.10 Climate control on the polychromator was required to minimize cycling ambient temperature effects in the Mass Spec. Lab.
- 4.11 Resetting of an automatic cutoff for power/gases was required.
- 4.12 Lowering the glovebox wall by decreasing the height between the glovebox window and the floor in order to view the glovebox floor was required for ease of observing containers and other apparatuses.

5.0 SECOND GENERATION GLOVEBOX FOR THE ICP

The backup ICP system was deemed necessary in order to support the vitrification milestone requirement that may not be met due to spectrometer malfunctions. Therefore, efforts were made to procure another custom designed ICP glovebox system with similar features. The existing Mass Spec. Lab, which already contained the ICP JY-50P™ spectrometer and VG Elemental Plasmaquad PQ2+™ ICP/MS glovebox systems with a common transfer interface box, was configured to accommodate the second generation new ICP spectrometer glovebox system. As a result of the restricted area available, the glovebox, along with the transfer interface box, had smaller overall dimensions, specifically, length (457 mm [18"] less), to fit in the existing space as shown in Figure 7, left side. The glovebox was designed to include the lessons learned from the first generation system (see Section 4 for details). The new system was modified as follows: the torch box was located closer to the analyst for replacement and repair; a drop tube was included along with a decrease in glovebox wall height; relocation of the transfer canister to the front of the glovebox on the same plane as the analyst for ease of sample transfers; and other utility changes. The new glovebox system containing the JY-46P™ spectrometer was received in 1995 and made operational in the spring of 1996. The first generation JY-50P ICP™ glovebox system was updated to operate with the same software as the second generation JY-46P ICP™ glovebox system. This minimized cross-training on two separate systems.

Figure 7. JY-46P ICP™ Glovebox (Left side)

5.1 Layout

The second generation glovebox overall dimensions were significantly smaller: approximately 1700 mm long (67"), 740 mm (29.1") deep on the bottom, 440 mm (17.3") deep at the top, and 1050 mm (41.3") high, mounted on a tubular frame chassis (See Figure 7). One end was connected to the transfer box door opening 360 mm (4.2") wide and 560 mm (22.0") long, and the opposite end was for the optical channel pathway between the torch box area inside the glovebox and the emission entrance to the spectrometer.

Figure 8A. JY-50P™ Glovebox Retrofitted Drop Tube (Top view)

Figure 8B. JY-50P™ Glovebox Retrofitted Drop Tube (Bottom view)

Figure 9. JY-46P™ Drop Tube

There was no top-mounted flange required for the pneumatic sample transfer system so the lights were located on the top. The shorter depth (313 mm [12.3"] from the front) to the torch box area allowed easier analyst access to the torch assembly system. Although smaller dimensions were advantageous for the existing laboratory footprint, the JY-46P™ system posed some problems, such as: a. The plasma being sensitive to rapid hand and arm movement causing “blow-out.” b. Additional canister transfers being required due to the restricted floor area since there was less space for all the samples, calibration Stds, and QC Stds. required for analyses.

5.2 Drop Tube for Nonradioactive Items

The use of a drop tube on the JY-46P™ system for nonradioactive material (e.g., calibration and QC stds., nonradioactive samples, etc.) proved to be very useful and saved time. As a result, the JY-50P™ system was retrofitted with a drop tube on the exiting pneumatic flange area plate of the JY-50P™ glovebox (see Figure 8A, top view) (see Figure 8B, bottom view) that was similar to the vendor’s design on JY-46P™ for 125 mL bottles (See Figure 9).

5.3 Glovebox Lighting

Fluorescent lights were installed on the top of the glovebox since no SGN flanges were required.

5.4 Torch Platform Modification

The use of the new JY torch cassette assembly was installed with a spring-assisted lever to support the spray chamber. The assembly design allowed better support alignment of the torch, spray chamber, and gas sheath attachment. The JY-50P™ was retrofitted with the cassette system after removing the existing plates for the torch holder and installing the cassette support plate (see Figure 10).

5.5 Interface System for Utilities

The interface requirements for the utilities were the same as in Section 3.10.

5.6 Analysis Requirements

The standby ICP is a simultaneous instrument that allows the detection and analysis of 25 analytes. This number of analytes was predicated on the final vitrification process campaign requirements (Al, B, Ba, Ca, Ce, Cr, Fe, K, Li, Mg, Mn, Na, Nd, Ni, P, Rh, S, Sc, Si, Sr, Th, Ti, U, Zn, Zr) developed by Process Development Engineering. The 3600 groves/mm grating for the spectrometer and a flat field grating for the alkalis were provided for the analytes. The use of corrosive HF required the utilization of an HF-resistant (demountable) torch, a Ryton™ Scott-type spray chamber, and the use of a HF corrosion-resistant Hildebrand™ nebulizer.

Figure 10. JY-50P™ Glovebox Retrofitted Torch Cassette

6.0 ICP GLOVEBOX COMMONALITIES

The cooling systems, gas supply systems, operating parameters, transfer interface boxes, polychromator climate control, spectrometer security location, and operations are part of the commonalities of the ICP glovebox assemblies.

6.1 Cooling Systems

Two independent cooling systems for the load coil and for the heat exchanger above the torch were installed for the JY-46P™ (see Figure 11A) and JY-50P™ (see Figure 11B) glovebox systems.

Figure 11a. Cooling Systems for JY-46P™

Figure 11b. Cooling Systems for JY-50P™

6.2 Gas Supply System

Liquidified argon and nitrogen 100 L cylinders, with dedicated manifold lines and automatic switch over systems between service and reserve cylinders' banks, are used to provide a continuous gas supply for the ICP glovebox assemblies.

6.3 Operating Parameters

The operating parameters for the JY-50P™ and JY-46P™ are:

Table 1. ICP Operating Parameters

Parameter	JY-46P™	JY-50P™
Output Power	1.0 kW	1.0 kW
Reflected Power	Automatic	Automatic
Glovebox Vacuum (neg.)	0.2-0.9" water	0.2-0.9" water
Argon Gas Flow	10-12 L/min.	10-12 L/min.
Sheath Gas Flow (G1)	0.10-0.40 L/min.	0.10-0.40 L/min.
Nebulizer (Hildebrand™)	2.5-3.2 bar	2.5-3.2 bar
Vacuum Alarm Set	0.1" water	0.1" water

The RF generator operates at a frequency of 40.68 MHz as a free running, tuned line oscillator.

6.4 Nitrogen Flow

The use of nitrogen gas in the glovebox and in the spectrometer provides an inert atmosphere that will allow purging the optic channels and polychromator area in order to permit the observation of wavelengths below 200 nanometers. Dedicated nitrogen purge lines are used for the optical channel (2 L/min.) in each glovebox and for the polychromator (4 L/min.) and lens channels in the spectrometer and spectrometer flow paths.

6.5 Transfer (Interface) Box

In addition to the transfer canisters (see Figure 12) to move material and samples from and to the gloveboxes, each glovebox has a transfer (interface) box in which large apparatus pieces such as the autosampler and plasma condenser can be moved into and out of the glovebox. The overall dimensions of each transfer box are approximately: 787 mm (31") high, 686 mm (27") deep, and 711 mm (28") wide for the JY-46P™ transfer interface box (see Figure 13); 520 mm (20.5") high, 850 mm (33.5") deep, and 711 mm (28") wide for the JY-50P™ transfer interface (see Figure 4) box. This glovebox also serves as a common interface with ICP/MS. Each transfer box has two 152 mm (6") glove port gloves for transferring material in to and out of the glovebox. Viewing windows of polycarbonate were located on the top of the boxes. In addition, the wall holding the glove ports of the second generation JY-46P™ glovebox system is polycarbonate glass which allows better viewing in the transfer box.

Figure 13. JY-46P™ Transfer Interface

Figure 12. Double Door Canister

6.6 Climate Control

Climate control was retrofitted on the JY-50P™ to minimize lab temperature perturbations of the polychromator and to conform with the installed climate control on the JY-46P™.

6.7 Spectrometer Location Security

The JY-50P™ spectrometer chassis had been misaligned due to inadvertent movement with construction and modification of the exhaust manifold system and the duct work installation. Both spectrometers' optical paths were aligned by laser to the glovebox plasma torch location and then diagonal angle braces were attached to floor so that the spectrometer feet could not be moved. An additional advantage with the JY-46P™ spectrometer alignment was that the laser was used in the spectrometer system rather than in the glovebox to minimize radioactive contamination of the laser apparatus.

6.8 Operations

The two ICP glovebox systems have been operating continuously since May 1996 with routine maintenance, preventive maintenance, and service checks. The operation of two systems has made it convenient at times to dedicate one for investigative or development work while the other continues to provide routine vitrification analysis support. Both ICP glovebox systems have an automatic plasma shutdown in case of the loss of glovebox vacuum.

7.0 ICP ANALYSIS OF VITRIFICATION SAMPLES

7.1 Introduction

The processing of HLW at the WVDP was initiated in June 1996. The transfer of the HLW takes place as a batch transfer sequence from the HLW tank with the melter being continuously fed. The A&PC Department developed remote analytical cell dissolution methods for the various process streams: waste initial (WI) composed of the HLW contents from Tank 8D-2 and from the submerged bed scrubber (SBS), cold chemical (glass formers [GF]), waste plus glass formers (WGF), and the final glass shard (SHD) product. The radioactive sample aliquot quantity and dilution factor were utilized to optimize the analyte detection range concentration and minimize the dose rate to the analyst while transferring and analyzing samples in the ICP gloveboxes. Dose rates for each sample were limited to 10 mR/hr with the window closed and 50 mR/hr (later changed to 100 mR/hr) with the window open. Occasionally, higher dose sample readings were permissible with a special Radiation Work Permit (RWP). Improvements in remote sample handling and analyses have decreased the sample turnaround time from 52 and 62 hours to <30 hours, respectively for the various parameters required for the initial waste (WI) slurry and waste plus glass formers (WGF) slurry. SHD analysis is performed as requested from a random 10% of the total number of canisters (275) and from process control needs. The SHD analytes required are those that must be reported at a metal oxide weight of 0.5% and greater with an additional nine analytes analyzed for process control trends.

7.2 Experimental Section

7.2.1 Standards

The control standards are made up in 4% nitric acid (HNO_3) [trace metal concentrated acid diluted with 18 M -cm water]. The calibration standards and calibration control standards from different lots are purchased as certified multi-element solutions. National Institute of Standards and Technology (NIST) certified standards are used for single and multiple element solutions when necessary in the 4% HNO_3 matrix. Inter-element correction and check standards are utilized to determine the inter-element correction (IEC) factors.

7.2.2 Nebulizers and spray chambers

A Hildebrand™ nebulizer and corrosion resistant Ryton™ Scott-type spray chamber are used with a two-channel Gilson Minipuls 3™ peristaltic pump. The nebulizer pressure was set at 2.5 bar with the sheath gas flow at 0.15 L/ min.

7.2.3 Inductively coupled plasma emission spectrometer

JY/Horiba Model JY-50P™ and JY-46P™ optical emission polychromator spectrometers were used for simultaneous analyses of 15 analytes for vitrification process feed slurry samples and 24 analytes for glass shard samples. Refer to Section 6.3 for operating parameters.

7.2.4 Sample transfers

Samples were transferred from the ACs via the sample transfer drawer into the Analytical Aisle glovebox for transport through the CRL double door canister system to the respective spectrometer glovebox. All sample preparations and dilutions were accomplished in-cell remotely using two-finger CRL Model G-HD™ remote manipulators.

7.2.5 Reagent and standard transfers

The ICP standards, 4% nitric acid, and any other nonradioactive samples were introduced into the gloveboxes via the respective drop tubes.

7.2.6 Sample dissolution reagents

Trace metal purity concentrated (conc.) HF and HNO₃; reagent grade 30% hydrogen peroxide (H₂O₂); boric acid, powder (99.5% or better reagent grade); reagent grade potassium hydroxide, KOH (25% w/v solution); oxalic acid dihydrate, reagent grade, 0.01 M solution.

7.2.7 Acid dissolution

WI and WGF samples were analyzed from their respective process streams after remote aliquoting from the individual sample round bottomed glass bottles (Hydragard™ type) received in the A&PC SSC area. The samples were pneumatically transferred from the sampler apparatus in the Vitrification Cell via the sample transfer cell in the Vitrification Facility. A representative aliquot, while stirring in the sample bottle (minimum of 0.3 g for WI and 0.15 g for WGF), was taken and transferred to a tared 120 mL vented Teflon™ digestion vessel with caps and pressure relief devices (Savaille™ #578). The sample was heated in an oven at 100°C for one hour with 5 mL HNO₃ and 5 mL HF, followed by 1 mL H₂O₂ (30%) addition then the contents were transferred to a weighed bottle and diluted to 100g. A secondary dilution was prepared for ICP analyses to minimize matrix effects. Post-boric digestion was performed on the primary dilution (0.55 g boric acid added to ~20 g aliquot) for the determination of Th in slurry and for the determination of Ba, Ce, Nd, and Th in glass samples.

Similarly, the dissolution of GF samples were performed using a minimum of 150 L aliquot. The GFs being nonradioactive were prepared in the hood and handled on bench top counters. A microwave digestion apparatus with 120 mL Teflon™ digestion vessels were used with the same quantity of reagents then heated in a batch of 12 digestion vessels for 27 minutes. However, post-boric digestion was not required since Th was not present in the glass-former makeup recipe.

The SHDs were analyzed after remote aliquoting a suspension of known proportion of ground glass to water to provide an aliquot size between 0.10 to 0.20 g, dispensed into a predried, preweighed container that is dried in a thermal oven and weighed to constant weight. The dissolution was similar to WI and WGF samples.

7.2.8 Alkali fusion

WI and WGF samples (aliquot minimum sizes 200 μ L and 150 μ L, respectively) were prepared for fusion by adding 2 mL of 25% w/w of KOH and a sample aliquot to a zirconium crucible in order to analyze for Ti. For GF samples, a minimum aliquot of 100 μ L was used for fusion preparation and was analyzed for Al, Mg, Si, and Ti. The fusion procedure involved drying in the thermal oven at 150°C for 30 minutes and then transferring to a furnace for 15 minutes at 500°C, followed by cooling and leaching with 20 mL of 0.01 M oxalic acid. Then concentrated nitric acid was added to the crucible and the contents transferred to a tared bottle and diluted to 100 g. The SHDs did not require alkali fusion, only dissolution.

7.2.9 Inter-element correction

Inter-element correction was evaluated for all the analytes for potential interferences. Interference correction standards were made for determining the amount of interference with each analyte. The software of the instrument was utilized to determine the interference correction factors of the interferences (perturbing) on the interfered (perturbed) analyte. Interference check standards were made and analyzed to confirm that the interference correction factors were accurate. The interfered and interfering elements results are summarized in Table 2 for the JY-46™ and the JY-50™ measurements.

Table 2. Inter-Element Correction for JY46P™ and JY50P™

JY-46P™ Inter-element Correction IEC13A.COR (File Name) U @ 385.958 nm				JY-50P™ Inter-element Correction IEC13B.COR (File Name) U @ 409.014 nm			
<div> <div>Theo. = Theoretical</div> <div>λ = Wavelength (nanometer [nm])</div> </div>							
Perturbed Element	Theo. λ	Perturbing Element	Theo. λ	Perturbed Element	Theo. λ	Perturbing Element	Theo. λ
Th	283.730	Zr	343.823	Th	283.730	Zr	343.823
Ce	399.924	Zr	343.823	Ce	399.924	Zr	343.823
Nd	401.225	Ce	399.924	Nd	401.225	Ce	399.924
U	385.958	Nd	401.225	U	409.014	Ce	399.924
Pd	342.124	Nd	401.225	U	409.014	Nd	401.225
Rh	343.489	Nd	401.225	Pd	342.124	Nd	401.225
Ru	240.272	Fe	259.940	Rh	343.489	Nd	401.225
U	385.958	Fe	259.940	Ru	240.272	Fe	259.940
Th	283.730	U	385.958	Th	283.730	U	409.014
Pd	342.124	U	385.958	Pd	342.124	U	409.014
U	385.958	Th	283.730	Pd	342.124	Th	283.730
Pd	342.124	Th	283.730	Rh	343.489	Th	283.730
Pd	342.124	Cr	205.552	Pd	342.124	Cr	205.552
Rh	343.489	Th	283.730	Ce	399.924	Nd	401.225
Ce	399.924	Nd	401.225				

Table 2. Inter-Element Correction for JY46P™ and JY50P™ (cont.)

JY-50P™ Inter-element Correction IEC10A.COR (File Name) U @ 385.958 nm				JY-50P™ Inter-element Correction IEC10B.COR (File Name) U @ 409.014 nm			
Perturbed Element	Theo. λ	Perturbing Element	Theo. λ	Perturbed Element	Theo. λ	Perturbing Element	Theo. λ
Th	283.730	U	385.958	Th	283.730	U	409.014
Pd	340.458	Th	283.730	Pd	340.458	Th	283.730
Al	308.215	Th	283.730	Al	308.215	Th	283.730
U	385.958	Th	283.730	U	409.014	Nd	401.225
U	385.958	Nd	401.225	Co	228.616	Ba	230.424
Co	228.616	Ba	230.424	Li	670.784	Ba	230.424
Li	670.784	Ba	230.424	Th	283.730	Zr	349.621
Th	283.730	Zr	349.621	Ru	240.272	Fe	259.940
Ru	240.272	Fe	259.940	Rh	343.489	Th	283.730
Rh	343.489	Th	283.730	Ce	380.153	Nd	401.225
Ce	380.153	Nd	401.225	Nd	401.225	Ce	380.153
Nd	401.225	Ce	380.153				

7.2.10 Spikes

Al, B, Fe, Si, Th, and Zr are added as a spiking solution for the digestion method, while Ti is added as a spiking solution for the fusion method for spike recovery determination. The percent spike recovery ranges from 75 to 125%.

7.2.11 Duplicate samples

The relative percent difference (RPD) of +/-20% is usually obtained for duplicate analysis.

7.2.12 Method detection limit

The empirical method detection limits (MDLs) of the analytes are displayed in Table 3 and were derived from the dissolution and fusion preparation of spiked solutions. These are the most conservative values from the two ICPs (JY/Horiba simultaneous JY-46P™ [installed 1995] and JY-50P™ [installed 1990]). The sample preparation is 100 mg of sample with 5 mL of HF and 5 mL of HNO₃ in a closed digestion vessel and 100 mg of sample with KOH for fusion of the sample. The empirical MDL was defined as the lowest concentration detected above the background within + 20% based on the method of dissolution. An estimated MDL was determined instrumentally by calculating the limits of detection (LOD) which was twice the standard deviation (SD) of integrating a blank (4% HNO₃) analyzed 11 times. Five times the calculated LOD was the estimated MDL. The estimated MDL was used as the starting point for determining the empirical MDL. The empirical MDL was then determined as three times the standard deviation of seven replicates analyzed over three nonconsecutive days.

Table 3. Method Detection**JY-46P™ and JY-50P™ MDLs**

Element	MDL(μg/mL)
Al	1.50
B	1.00
Ba	0.20
Ca	0.20
Ce	0.50
Co	0.50
Cr	0.20
Cu	0.20
Fe	0.50
K	1.00
La	0.50
Li	0.10
Mg	0.80
Mn	0.30
Mo	0.10
Na	3.00
Nd	0.50
Ni	0.20
P	1.00
Pd	0.20
Rh	0.20
Ru	0.20
S	1.00
Si	5.00
Sr	0.50
Th	0.20
Ti	0.25
U @ 385.958nm	0.50
U @ 409.014nm	0.50
Zn	0.50
Zr	0.50

7.2.13 Calibration control

Before the start of a batch analysis on the ICP and after calibration, an initial calibration verification (ICV) standard is performed followed by the sample. The continuing calibration verification (CCV) standard is analyzed at the end of a sample run or after 12 samples, whichever is more frequent. These QC results are plotted on the same QC chart to verify instrument performance.

7.3 Results

7.3.1 Analytes

The 15 key analytes that are monitored in the process control plan are the following for WI and WGF streams: Al, B, Ca, Fe, K, Li, Mg, Mn, Na, P, Si, Th, Ti, U, and Zr. The 13 analytes required for the glass formers are Al, B, Ca, Fe, K, Li, Mg, Mn, Na, P, Si, Ti, and Zr. For the SHDs, the required analytes are the 15 analytes (mentioned above) that form metal oxides as a weight percent (%) equal to or greater than 0.5% and an additional nine analytes that are followed for trending: Ba, Ce, Cr, Nd, Ni, Ru, S, Sr, and Zn. The WI, WGF, and SHDs being radioactive were prepared remotely in shielded hot cells, while the GFs or cold chemicals were prepared in the hood furnace and the bench-top microwave oven. All the prepped samples were analyzed by the JY-50P™ or JY-46P™ system. The measured analyte values were compared to the calculated values of the process recipe. The statistical requirement for comparing the recipe required a 95-percentile confidence level. This in turn defined the number of random sample sequences for the ICP analyses and other parameters of interest.

7.3.2 Batch analysis

Each batch is composed of the WI, GF, and WGF results. Although every batch is analyzed to confirm the process make-up, only ten batches were selected from the start of the campaign in June 1996 through June 1998 for comparing results. Batch #50 was used as an example to follow the results from WI, GF, and WGF. SHD results from ten predetermined randomly selected and process control canister samples are also compared. The various results, including the mean, standard deviation (SD), and percent relative standard deviation (%RSD) on the above selected batches, are summarized in Tables 4 through 12.

7.3.2.1 Waste initial results

The number of statistically valid data points required from each batch for ICP data evaluation of waste initial transfers were nine. The sample preparation for the WI analysis required, in addition to the nine samples, a blank, spike and duplicate analysis, for a total of 12 samples. Samples were prepared by acid dissolution for all analytes except Ti, which required alkali fusion, and Th analysis, which required the additional post-boric digestion.

Table 4 summarizes typical results from a WI batch (#50) showing the nine sample analyses. The mean, SD, and %RSD are also included in Table 4. The %RSD based on replicate analyses ranged from 3 to 14 for all the analytes.

Table 5 shows comparative results for all the analytes for ten selected batches (every fifth batch) from the start of the campaign in June 1996 through June 1998 along with the mean, SD, and %RSD. The U results are only shown starting with batch #30 as they were not measured in detectable levels in earlier batches. The %RSD comparison among the batches clearly shows less than 10% variation for all the analytes. Although, it should be pointed out that the %RSD was abnormally high (13.91) for Ti in Batch #50. Similarly, the mean and SD comparisons were in good agreement and within experimental uncertainties.

Table 4. WI Analysis Batch: Mean, SD, and %RSD for a Representative Batch

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Mean	SD	% RSD
Al	5.56E+03	5.25E+03	5.69E+03	5.57E+03	5.26E+03	5.18E+03	5.39E+03	5.40E+03	5.65E+03	5.44E+03	1.88E+02	3.43
B	1.69E+03	1.60E+03	1.74E+03	1.71E+03	1.59E+03	1.58E+03	1.69E+03	1.61E+03	1.68E+03	1.65E+03	5.94E+01	3.59
Ca	1.32E+03	1.30E+03	1.36E+03	1.51E+03	1.26E+03	1.26E+03	1.31E+03	1.23E+03	1.36E+03	1.32E+03	8.29E+01	6.27
Fe	3.59E+04	3.41E+04	3.82E+04	3.73E+04	3.42E+04	3.35E+04	3.50E+04	3.46E+04	3.65E+04	3.55E+04	1.60E+03	4.50
K	1.83E+03	1.79E+03	1.76E+03	1.74E+03	1.70E+03	1.74E+03	1.78E+03	1.68E+03	1.67E+03	1.74E+03	5.32E+01	3.05
Li	5.21E+02	5.04E+02	5.47E+02	5.23E+02	4.98E+02	4.88E+02	5.13E+02	5.02E+02	5.25E+02	5.13E+02	1.77E+01	3.45
Mg	5.24E+02	5.07E+02	5.43E+02	5.26E+02	5.02E+02	4.97E+02	5.22E+02	5.12E+02	5.52E+02	5.21E+02	1.83E+01	3.52
Mn	2.56E+03	2.45E+03	2.68E+03	2.63E+03	2.46E+03	2.42E+03	2.56E+03	2.50E+03	2.63E+03	2.54E+03	9.15E+01	3.60
Na	1.32E+04	1.27E+04	1.36E+04	1.33E+04	1.25E+04	1.24E+04	1.33E+04	1.28E+04	1.33E+04	1.30E+04	4.20E+02	3.23
P	1.18E+03	1.05E+03	1.15E+03	1.10E+03	1.08E+03	1.04E+03	1.06E+03	1.13E+03	1.13E+03	1.10E+03	4.80E+01	4.35
Si	1.81E+04	1.71E+04	1.86E+04	1.82E+04	1.71E+04	1.67E+04	1.73E+04	1.73E+04	1.81E+04	1.76E+04	6.47E+02	3.67
Th	1.09E+04	1.13E+04	1.11E+04	1.33E+04	1.11E+04	1.15E+04	1.13E+04	1.09E+04	1.12E+04	1.14E+04	7.38E+02	6.48
Ti	4.13E+02	3.44E+02	4.07E+02	3.64E+02	4.12E+02	2.47E+02	3.92E+02	3.76E+02	3.76E+02	3.70E+02	5.17E+01	13.97
U	2.43E+03	2.36E+03	2.53E+03	2.49E+03	2.29E+03	2.30E+03	2.40E+03	2.35E+03	2.44E+03	2.40E+03	8.19E+01	3.41
Zr	1.26E+03	1.21E+03	1.32E+03	1.29E+03	1.21E+03	1.19E+03	1.25E+03	1.22E+03	1.28E+03	1.25E+03	4.35E+01	3.49

Batch 50, 9 Replicates

Table 5. WI Analysis: Mean, SD, and %RSD for Ten Selected Batches

Mean										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	4.96E+03	3.97E+03	4.22E+03	5.34E+03	5.23E+03	6.15E+03	6.77E+03	5.44E+03	5.69E+03	5.87E+03
B	1.58E+03	7.78E+02	1.22E+03	9.87E+02	1.45E+03	1.27E+03	1.09E+03	1.65E+03	1.94E+03	1.44E+03
Ca	1.32E+03	9.96E+02	1.12E+03	9.46E+02	9.92E+02	1.22E+03	1.39E+03	1.32E+03	1.26E+03	1.45E+03
Fe	3.50E+04	2.47E+04	2.98E+04	3.47E+04	2.78E+04	3.17E+04	3.59E+04	3.55E+04	3.43E+04	3.59E+04
K	2.05E+03	1.29E+03	1.83E+03	2.13E+03	2.20E+03	1.95E+03	2.25E+03	1.74E+03	2.26E+03	2.64E+03
Li	5.63E+02	5.72E+02	4.47E+02	4.75E+02	5.51E+02	4.57E+02	5.30E+02	5.13E+02	5.94E+02	5.88E+02
Mg	4.35E+02	2.84E+02	3.17E+02	4.46E+02	5.09E+02	4.11E+02	4.28E+02	5.21E+02	5.15E+02	5.13E+02
Mn	2.60E+03	1.82E+03	2.17E+03	2.49E+03	2.01E+03	2.34E+03	2.60E+03	2.54E+03	2.53E+03	2.44E+03
Na	9.86E+03	9.41E+03	1.07E+04	1.21E+04	1.38E+04	1.33E+04	1.54E+04	1.30E+04	2.00E+04	1.90E+04
P	1.44E+03	1.11E+03	8.73E+02	1.42E+03	1.12E+03	1.03E+03	1.01E+03	1.10E+03	9.65E+02	9.94E+02
Si	1.68E+04	1.57E+04	1.48E+04	1.64E+04	1.51E+04	1.65E+04	1.91E+04	1.76E+04	1.58E+04	1.85E+04
Th	1.66E+04	1.25E+04	1.29E+04	1.13E+04	1.11E+04	1.24E+04	1.26E+04	1.14E+04	1.10E+04	1.12E+04
Ti	3.64E+02	4.02E+02	3.80E+02	3.74E+02	3.62E+02	3.85E+02	3.92E+02	3.70E+02	3.76E+02	2.99E+02
U	N/A	N/A	N/A	N/A	1.81E+03	2.04E+03	2.44E+03	2.40E+03	2.30E+03	2.40E+03
Zr	1.29E+03	1.03E+03	1.08E+03	1.34E+03	1.06E+03	1.13E+03	1.29E+03	1.25E+03	1.19E+03	1.27E+03

Standard Deviation										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	6.00E+01	1.37E+02	1.05E+02	1.25E+02	1.44E+02	4.17E+02	2.15E+02	1.86E+02	3.40E+02	1.25E+02
B	2.01E+01	8.02E+01	3.67E+01	7.64E+01	6.62E+01	6.71E+01	5.31E+01	5.94E+01	5.79E+01	3.73E+01
Ca	3.08E+01	2.88E+01	4.35E+01	1.55E+02	6.32E+01	3.17E+01	4.16E+01	8.29E+01	1.86E+02	9.08E+01
Fe	2.59E+02	6.18E+02	6.12E+02	6.44E+02	1.14E+03	8.86E+02	9.71E+02	1.60E+03	1.50E+03	6.41E+02
K	3.97E+01	1.45E+02	1.04E+02	8.62E+01	3.61E+01	9.60E+01	6.26E+01	5.32E+01	1.31E+02	1.25E+02
Li	4.41E+00	1.28E+01	9.21E+00	2.08E+01	9.04E+00	6.23E+00	1.64E+01	1.77E+01	1.63E+01	1.67E+01
Mg	1.53E+01	1.47E+01	2.69E+01	1.38E+01	5.14E+01	2.66E+01	2.26E+01	1.83E+01	2.73E+01	1.37E+01
Mn	2.06E+01	4.19E+01	2.12E+01	4.97E+01	9.02E+01	6.21E+01	7.50E+01	9.15E+01	1.20E+02	4.66E+01
Na	9.66E+01	2.18E+02	1.69E+02	2.24E+02	2.21E+03	2.82E+02	4.68E+02	4.20E+02	5.96E+02	2.13E+02
P	9.90E+01	9.89E+01	8.19E+01	2.39E+02	4.85E+01	6.87E+01	9.10E+01	4.80E+01	3.96E+01	6.63E+01
Si	2.71E+02	1.87E+03	5.80E+02	6.10E+02	4.33E+02	1.16E+03	5.94E+02	6.47E+02	7.38E+02	4.41E+02
Th	1.48E+02	5.08E+02	2.57E+02	1.48E+03	2.17E+02	4.42E+02	8.11E+02	7.38E+02	6.35E+02	1.81E+02
Ti	1.80E+01	8.32E+01	1.51E+01	2.30E+01	2.09E+01	3.43E+01	2.42E+01	5.17E+01	5.71E+01	1.85E+01
U	N/A	N/A	N/A	N/A	8.26E+01	5.22E+01	6.90E+01	8.19E+01	9.06E+01	5.02E+01
Zr	1.13E+01	2.17E+01	1.41E+01	2.22E+01	4.19E+01	2.93E+01	3.48E+01	4.35E+01	5.14E+01	1.41E+01

% Relative Standard Deviation										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	1.21	3.46	2.49	2.34	2.76	6.77	3.17	3.43	5.98	2.13
B	1.27	10.31	3.00	7.74	4.57	5.30	4.88	3.59	2.99	2.60
Ca	2.34	2.90	3.89	16.36	6.37	2.61	3.00	6.27	14.71	6.25
Fe	0.74	2.51	2.05	1.85	4.12	2.79	2.71	4.50	4.36	1.79
K	1.94	11.29	5.71	4.05	1.64	4.92	2.78	3.05	5.80	4.73
Li	0.78	2.24	2.05	4.37	1.64	1.36	3.09	3.45	2.74	2.84
Mg	3.51	5.17	8.49	3.09	10.10	6.48	5.28	3.52	5.31	2.66
Mn	0.79	2.31	0.98	2.00	4.49	2.65	2.88	3.60	4.72	1.91
Na	0.96	2.32	1.58	1.85	15.97	2.12	3.05	3.23	2.98	1.12
P	6.88	8.93	9.38	16.91	4.34	6.69	9.02	4.35	4.10	6.66
Si	1.61	11.93	3.92	3.71	2.86	7.14	3.11	3.67	4.66	2.39
Th	0.89	4.05	1.99	13.08	1.95	3.55	6.46	6.48	5.75	1.62
Ti	4.93	20.69	3.98	6.14	5.76	8.90	6.17	13.97	15.17	6.18
U	N/A	N/A	N/A	N/A	4.57	2.55	2.83	3.41	3.94	1.93
Zr	0.01	2.10	1.31	1.66	3.93	2.60	2.68	3.49	4.31	1.11

7.3.2.2 Glass-formers results

The number of statistically valid data points required from each batch makeup of glass formers for ICP data evaluation were three. The sample preparation for the nonradioactive GF analysis required, in addition to the three samples, a blank, spike, and duplicate analysis, for a total of 6 samples. Samples were prepared by acid dissolution for all analytes except Ti which required alkali fusion.

Table 6 summarizes typical results of three sample GF analyses from the same batch #50 as the WI. The mean, SD, and %RSD are included in Table 6. The %RSDs based on replicate analyses are less than five for the GF analytes. As mentioned previously, U and Th are not analyzed since these constituents are not part of the recipe for glass formers.

Table 7 displays comparative GF results for all the analytes for ten selected batches (every fifth batch) from the start of the campaign in June 1996 through June 1998 along with the mean (result of three analyses), SD, and %RSD. The %RSD among the batches shows less than 5% variation for greater than 92% of the analytes. Similarly, the mean and SD comparisons indicate excellent agreement for all analytes among the batches.

Table 6. GF Analysis: Mean, SD, and %RSD for a Representative Batch

	<i>Sample 1</i>	<i>Sample 2</i>	<i>Sample 3</i>	<i>Mean</i>	<i>SD</i>	<i>% RSD</i>
Al	1.40E+04	1.39E+04	1.45E+04	1.41E+04	3.21E+02	2.27
B	2.59E+04	2.57E+04	2.62E+04	2.59E+04	2.52E+02	0.97
Ca	1.92E+02	1.87E+02	2.03E+02	1.94E+02	8.19E+00	4.22
Fe	4.81E+02	4.67E+02	5.09E+02	4.86E+02	2.14E+01	4.40
K	2.43E+04	2.38E+04	2.41E+04	2.41E+04	2.52E+02	1.05
Li	1.13E+04	1.12E+04	1.13E+04	1.13E+04	5.77E+01	0.51
Mg	2.78E+03	2.83E+03	2.90E+03	2.84E+03	6.03E+01	2.12
Mn	2.89E+02	2.80E+02	3.05E+02	2.91E+02	1.27E+01	4.35
Na	2.02E+04	1.98E+04	2.01E+04	2.00E+04	2.08E+02	1.04
P	2.00E+03	2.02E+03	2.01E+03	2.01E+03	1.00E+01	0.50
Si	1.11E+05	1.10E+05	1.14E+05	1.12E+05	2.08E+03	1.86
Ti	2.45E+03	2.42E+03	2.50E+03	2.46E+03	4.04E+01	1.65
Zr	4.97E+03	4.90E+03	4.99E+03	4.95E+03	4.73E+01	0.95

Batch 50, 3 Replicates

Table 7. GF Analysis: Mean, SD, and %RSD for Ten Selected Batches

Mean										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	1.11E+04	1.05E+04	1.16E+04	1.04E+04	1.05E+04	1.01E+04	1.02E+04	1.41E+04	1.38E+04	9.58E+03
B	2.03E+04	2.07E+04	2.23E+04	2.52E+04	1.88E+04	2.35E+04	2.40E+04	2.59E+04	2.62E+04	2.25E+04
Ca	2.58E+02	3.27E+02	3.16E+02	2.87E+02	1.36E+02	9.90E+01	1.54E+02	1.94E+02	1.98E+02	1.77E+02
Fe	6.73E+03	1.30E+04	7.44E+03	2.44E+02	3.44E+03	2.64E+03	5.02E+03	4.86E+02	2.28E+03	4.49E+03
K	1.90E+04	1.85E+04	2.11E+04	2.38E+04	2.25E+04	2.12E+04	2.24E+04	2.41E+04	2.39E+04	2.18E+04
Li	8.18E+03	7.72E+03	9.90E+03	1.10E+04	9.02E+03	9.39E+03	1.03E+04	1.13E+04	1.03E+04	9.11E+03
Mg	2.13E+03	2.20E+03	2.55E+03	2.57E+03	2.38E+03	2.69E+03	2.59E+03	2.84E+03	3.38E+03	2.14E+03
Mn	5.28E+02	1.00E+03	6.18E+02	2.08E+02	3.46E+02	2.58E+02	4.58E+02	2.91E+02	2.93E+02	6.51E+02
Na	2.21E+04	1.90E+04	2.02E+04	2.07E+04	1.17E+04	1.59E+04	1.70E+04	2.00E+04	8.80E+03	1.24E+04
P	1.37E+03	1.41E+03	1.83E+03	1.29E+03	1.06E+03	1.53E+03	1.88E+03	2.01E+03	1.76E+03	2.34E+03
Si	7.87E+04	8.65E+04	9.84E+04	1.01E+05	9.87E+04	1.02E+05	1.03E+05	1.12E+05	1.27E+05	9.37E+04
Ti	1.97E+03	1.86E+03	2.09E+03	2.17E+03	2.08E+03	2.03E+03	2.20E+03	2.46E+03	2.57E+03	2.02E+03
Zr	3.79E+03	3.99E+03	4.39E+03	4.58E+03	3.77E+03	4.32E+03	4.56E+03	4.95E+03	5.12E+03	4.32E+03

Standard Deviation										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	8.00E+02	1.00E+02	7.02E+02	2.52E+02	2.52E+02	1.42E+02	2.25E+02	3.21E+02	4.58E+02	2.69E+02
B	7.09E+02	5.77E+01	1.01E+03	3.78E+02	4.93E+02	5.03E+02	7.02E+02	2.52E+02	4.58E+02	3.21E+02
Ca	1.35E+01	4.93E+00	9.29E+00	6.11E+00	1.15E+00	2.50E+00	1.70E+01	8.19E+00	8.50E+00	7.57E+00
Fe	2.43E+02	5.77E+01	1.84E+02	7.77E+00	5.03E+01	5.51E+01	1.21E+02	2.14E+01	2.52E+01	3.21E+01
K	5.69E+02	1.15E+02	2.65E+02	1.15E+02	3.21E+02	1.53E+02	3.51E+02	2.52E+02	4.16E+02	1.53E+02
Li	2.12E+02	6.43E+01	8.50E+01	1.73E+02	1.81E+02	9.07E+01	2.52E+02	5.77E+01	1.53E+02	7.09E+01
Mg	1.95E+02	2.08E+01	1.67E+02	9.07E+01	5.77E+00	3.08E+01	6.68E+01	6.03E+01	5.51E+01	1.27E+02
Mn	2.25E+01	5.77E+00	1.91E+01	5.77E-01	1.42E+01	1.03E+01	1.07E+01	1.27E+01	1.25E+01	3.08E+00
Na	5.51E+02	1.53E+02	3.21E+02	1.73E+02	1.73E+02	2.08E+02	3.51E+02	2.08E+02	2.74E+02	1.53E+02
P	3.46E+01	1.53E+01	5.57E+01	2.31E+01	2.65E+01	1.06E+02	8.33E+01	1.00E+01	4.58E+01	4.04E+01
Si	2.85E+03	1.30E+03	4.03E+03	2.76E+03	5.72E+03	2.19E+03	1.15E+03	2.08E+03	3.51E+03	3.13E+03
Ti	5.51E+01	3.46E+01	1.10E+02	7.00E+01	6.66E+01	1.41E+01	4.58E+01	4.04E+01	3.79E+01	8.66E+01
Zr	1.23E+02	1.53E+01	1.04E+02	2.52E+01	5.13E+01	6.51E+01	1.05E+02	4.73E+01	5.86E+01	2.52E+01

% Relative Standard Deviation										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	7.21	0.95	6.07	2.41	2.40	1.41	2.21	2.27	3.32	2.80
B	3.49	0.28	4.52	1.50	2.50	2.14	2.93	0.97	1.75	1.43
Ca	5.26	1.51	2.94	2.13	0.85	2.52	11.04	4.22	4.35	4.29
Fe	3.61	0.45	2.47	3.19	1.46	2.08	2.40	4.40	1.10	0.72
K	3.00	0.62	1.25	0.49	1.43	0.72	1.57	1.05	1.74	0.70
Li	2.59	0.83	0.86	1.57	1.78	0.97	2.45	0.51	1.48	0.78
Mg	9.14	0.95	6.52	3.53	0.24	1.13	2.57	2.12	1.63	5.96
Mn	4.27	0.58	3.10	0.28	4.10	3.98	2.34	4.35	4.25	0.47
Na	2.50	0.81	1.59	0.84	1.48	1.31	2.06	1.04	3.12	1.23
P	2.53	1.08	3.04	1.79	2.50	6.91	4.42	0.50	2.80	1.72
Si	3.63	1.50	4.09	2.73	5.79	2.15	1.12	1.86	2.76	3.34
Ti	2.80	1.86	5.26	3.23	3.20	0.70	2.08	1.85	1.48	4.29
Zr	3.24	0.38	2.38	0.55	1.36	1.51	2.31	0.95	1.15	0.58

7.3.2.3 Waste plus glass formers

The number of statistically valid data points required from each batch for ICP data evaluation of WGF batch makeup were nine. The WGFs are from the addition of the GFs to WI batch #50. The sample preparation for the WGF (as for the WI) analysis required, in addition to the nine samples, a blank, spike, and duplicate analysis, for a total of 12 samples. Samples were prepared by acid dissolution for all analytes, except Ti which required alkali fusion and Th analysis which required the additional post-boric digestion.

Table 8 summarizes typical results from a WGF batch (#50) showing the nine sample analyses. The mean, SD, and %RSD are also included in Table 8. The result of the low %RSDs ensures that the composition is within the process stream uncertainty limits and therefore confirms a HLW glass product that meets the agreed upon composition requirements for the federal repository.

Table 9 shows comparative results for all the analytes for ten selected batches (every fifth batch) from the start of the campaign in June 1996 through June 1998 along with the mean, SD, and %RSD. The U results are only shown starting with batch #30 as they were not measured in detectable levels in earlier batches. The % RSD comparison among the batches clearly shows less than 10% variation in the majority of analytes. Batches #30 and #35 show high %RSDs which were the result of averaging more than nine samples with separate repeats. While %RSDs are less than 10% variation for batch #60, the majority of the individual analyte %RSDs are increased in a range of 4 to 7 as compared to the other batch runs. However, the mean and SD comparisons were in good agreement and within experimental uncertainties.

Table 8. WGF Analysis Batch: Mean SD, and %RSD for a Representative Batch

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Mean	SD	% RSD
Al	1.32E+04	1.37E+04	1.43E+04	1.35E+04	1.34E+04	1.32E+04	1.34E+04	1.30E+04	1.35E+04	1.35E+04	3.74E+02	2.78
B	1.75E+04	1.75E+04	1.94E+04	1.81E+04	1.82E+04	1.70E+04	1.74E+04	1.74E+04	1.70E+04	1.77E+04	7.53E+02	4.25
Ca	2.03E+03	1.88E+03	1.81E+03	1.79E+03	1.80E+03	1.70E+03	1.82E+03	1.96E+03	1.79E+03	1.84E+03	9.90E+01	5.38
Fe	3.41E+04	3.56E+04	3.76E+04	3.55E+04	3.57E+04	3.37E+04	3.41E+04	3.30E+04	3.44E+04	3.49E+04	1.38E+03	3.97
K	1.66E+04	1.73E+04	1.83E+04	1.70E+04	1.72E+04	1.66E+04	1.67E+04	1.63E+04	1.69E+04	1.70E+04	5.84E+02	3.44
Li	7.48E+03	7.96E+03	8.34E+03	7.79E+03	7.81E+03	7.51E+03	7.59E+03	7.39E+03	7.65E+03	7.72E+03	2.93E+02	3.79
Mg	2.43E+03	2.49E+03	2.60E+03	2.46E+03	2.47E+03	2.38E+03	2.45E+03	2.41E+03	2.47E+03	2.46E+03	6.18E+01	2.51
Mn	2.52E+03	2.65E+03	2.80E+03	2.63E+03	2.65E+03	2.53E+03	2.55E+03	2.49E+03	2.61E+03	2.60E+03	9.47E+01	3.64
Na	2.56E+04	2.65E+04	2.74E+04	2.80E+04	2.58E+04	2.45E+04	2.48E+04	2.45E+04	2.53E+04	2.56E+04	9.62E+02	3.76
P	2.28E+03	2.25E+03	2.45E+03	2.38E+03	2.27E+03	2.26E+03	2.24E+03	2.40E+03	2.38E+03	2.32E+03	7.86E+01	3.38
Si	8.19E+04	8.59E+04	9.02E+04	8.53E+04	8.52E+04	8.26E+04	8.23E+04	7.99E+04	8.39E+04	8.41E+04	2.98E+03	3.54
Th	1.22E+04	1.28E+04	1.25E+04	1.25E+04	1.05E+04	1.25E+04	1.17E+04	1.13E+04	1.11E+04	1.19E+04	7.89E+02	6.63
Ti	1.91E+03	1.92E+03	1.70E+03	1.94E+03	1.90E+03	1.98E+03	1.95E+03	1.79E+03	1.95E+03	1.89E+03	9.03E+01	4.77
U	2.15E+03	2.21E+03	2.42E+03	2.22E+03	2.26E+03	2.14E+03	2.19E+03	2.10E+03	2.22E+03	2.21E+03	9.20E+01	4.16
Zr	4.19E+03	4.26E+03	4.59E+03	4.32E+03	4.37E+03	4.10E+03	4.11E+03	4.18E+03	4.23E+03	4.26E+03	1.52E+02	3.57

Batch 50, 9 Replicates

Table 9. WGF Analysis: Mean, SD, and %RSD for Ten Selected Batches

Mean										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	1.27E+04	1.09E+04	1.25E+04	1.39E+04	1.30E+04	1.26E+04	1.31E+04	1.35E+04	1.35E+04	1.27E+04
B	1.61E+04	1.61E+04	1.58E+04	1.69E+04	1.52E+04	1.53E+04	1.65E+04	1.77E+04	1.75E+04	1.67E+04
Ca	1.11E+03	1.13E+03	1.34E+03	1.65E+03	1.48E+03	1.32E+03	1.48E+03	1.84E+03	1.35E+03	1.49E+03
Fe	2.88E+04	3.19E+04	3.13E+04	3.55E+04	3.50E+04	3.37E+04	3.47E+04	3.49E+04	3.44E+04	3.43E+04
K	1.54E+04	1.44E+04	1.57E+04	1.71E+04	1.84E+04	1.51E+04	1.65E+04	1.70E+04	1.66E+04	1.68E+04
Li	6.43E+03	5.96E+03	7.06E+03	7.47E+03	6.80E+03	6.50E+03	7.02E+03	7.72E+03	6.98E+03	6.59E+03
Mg	2.02E+03	1.97E+03	2.21E+03	2.26E+03	2.11E+03	2.12E+03	2.19E+03	2.46E+03	2.65E+03	2.38E+03
Mn	2.15E+03	2.39E+03	2.38E+03	2.66E+03	2.65E+03	2.47E+03	2.61E+03	2.60E+03	2.47E+03	2.57E+03
Na	2.34E+04	2.06E+04	2.40E+04	2.52E+04	2.39E+04	2.31E+04	2.42E+04	2.56E+04	2.56E+04	2.47E+04
P	1.82E+03	1.73E+03	2.13E+03	2.01E+03	1.95E+03	1.93E+03	1.64E+03	2.32E+03	2.02E+03	2.29E+03
Si	7.04E+04	6.49E+04	7.54E+04	8.84E+04	7.39E+04	7.45E+04	7.66E+04	8.41E+04	8.19E+04	7.92E+04
Th	1.10E+03	1.04E+03	1.19E+03	1.16E+03	1.24E+03	1.31E+03	1.07E+03	1.19E+03	1.09E+03	9.60E+02
Ti	1.68E+03	1.51E+03	1.95E+03	2.01E+03	1.68E+03	1.79E+03	1.89E+03	1.89E+03	1.81E+03	1.65E+03
U	N/A	N/A	N/A	2.46E+03	2.16E+03	2.10E+03	2.28E+03	2.21E+03	2.22E+03	2.32E+03
Zr	3.70E+03	3.55E+03	3.86E+03	4.18E+03	3.85E+03	3.67E+03	4.14E+03	4.26E+03	4.13E+03	3.96E+03

Standard Deviation										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	1.69E+02	3.85E+02	1.17E+02	2.09E+03	9.28E+02	3.86E+02	4.76E+02	3.74E+02	5.46E+02	7.14E+02
B	3.08E+02	9.82E+02	4.96E+02	2.52E+03	1.07E+03	6.28E+02	7.20E+02	7.53E+02	4.77E+02	1.02E+03
Ca	4.24E+01	5.59E+01	9.30E+01	1.65E+02	1.77E+02	8.73E+01	1.53E+02	9.90E+01	8.38E+01	1.35E+02
Fe	5.56E+02	7.79E+02	5.29E+02	3.84E+03	2.07E+03	1.01E+03	1.21E+03	1.38E+03	1.06E+03	1.90E+03
K	2.51E+02	4.77E+02	1.59E+02	1.17E+03	1.25E+03	3.77E+02	4.06E+02	5.84E+02	5.83E+02	1.00E+03
Li	8.59E+01	1.62E+02	8.50E+01	7.23E+02	3.82E+02	1.68E+02	1.36E+02	2.93E+02	2.37E+02	3.95E+02
Mg	4.02E+01	7.36E+01	3.61E+01	1.38E+02	1.97E+02	6.74E+01	1.05E+02	6.18E+01	9.33E+01	1.52E+02
Mn	4.27E+01	7.79E+01	3.36E+01	2.06E+02	1.54E+02	7.35E+01	8.57E+01	9.47E+01	8.15E+01	1.44E+02
Na	5.23E+02	5.70E+02	2.54E+02	1.93E+03	1.86E+03	6.28E+02	5.79E+02	9.62E+02	8.08E+02	1.51E+03
P	6.44E+01	1.17E+02	7.57E+01	3.13E+02	8.28E+01	1.68E+02	1.16E+02	7.86E+01	8.18E+01	2.09E+02
Si	1.19E+03	2.18E+03	1.08E+03	1.35E+04	3.97E+03	2.06E+03	4.00E+03	2.98E+03	2.79E+03	4.64E+03
Th	1.62E+02	6.18E+02	4.29E+02	1.89E+03	3.97E+02	1.64E+02	6.62E+02	7.89E+02	4.76E+02	5.05E+02
Ti	6.71E+01	7.38E+01	2.19E+02	1.18E+02	7.74E+01	9.41E+01	6.70E+01	9.03E+01	5.63E+01	6.61E+01
U	N/A	N/A	N/A	2.47E+02	1.05E+02	5.82E+01	7.90E+01	9.20E+01	7.05E+01	1.19E+02
Zr	5.70E+01	7.83E+01	4.95E+01	4.37E+02	3.00E+02	1.04E+02	1.26E+02	1.52E+02	1.34E+02	2.27E+02

% Relative Standard Deviation										
Batch #	15	20	25	30	35	40	45	50	55	60
Al	1.33	3.55	0.93	15.07	7.15	3.06	3.63	2.78	4.04	5.63
B	1.91	6.10	3.14	14.92	7.03	4.10	4.36	4.25	2.70	6.14
Ca	3.81	4.93	6.92	10.00	11.96	6.62	10.36	5.38	6.22	9.04
Fe	1.93	2.44	1.69	10.80	5.91	2.99	3.48	3.97	3.09	5.52
K	1.63	3.32	1.02	6.80	6.78	2.50	2.46	3.44	3.52	5.94
Li	1.34	2.71	1.20	9.68	5.62	2.55	1.93	3.79	3.40	6.00
Mg	1.99	3.74	1.63	6.10	9.33	3.18	4.78	2.51	3.52	6.40
Mn	1.99	3.28	1.41	7.73	5.81	2.98	3.28	3.64	3.30	5.82
Na	2.23	2.76	1.06	7.63	7.77	2.72	2.40	3.76	3.15	6.09
P	3.55	6.78	3.56	15.60	4.25	8.72	7.11	3.38	4.05	9.10
Si	1.68	3.37	1.43	15.33	5.38	2.77	5.22	3.54	3.41	5.86
Th	1.47	5.94	3.60	16.25	3.19	1.25	8.08	6.63	4.36	5.28
Ti	3.93	4.89	11.19	5.87	4.60	5.26	3.55	4.77	3.11	4.00
U	N/A	N/A	N/A	10.04	4.83	2.77	3.46	4.16	3.17	5.13
Zr	1.54	2.21	1.28	10.47	7.81	2.84	3.04	3.57	3.25	5.72

7.3.2.4 Shards

The ICP analysis of the HLW glass shards initially started at the beginning of the vitrification processing campaign with three samples increasing to six and finally to nine samples for statistical verification. This increase to nine SHD samples was to improve the statistics by not relying on analyzing archival samples in the future. The remote preparations of the SHD samples are quite involved, requiring grinding, suspension, and aliquoting the suspension into a tared vessel, drying the sample, weighing, dissolution, and dilution for ICP analysis. As an example, Table 10 presents the SHD analysis of nine replicates from Canister WV-279 displaying the mean, SD, and %RSD. The %RSD is less than six for all analytes.

The mean, SD, and %RSD of the ten selected canisters exhibited good precision for the analysis of the glass shards despite the involved remote sample preparation (see Table 11). The %RSD is less than seven for the majority of analytes except Th which has a higher %RSD in Canisters WV-191, WV-263, and WV-265.

As previously mentioned, an additional nine analytes (Ba, Ce, Cr, Nd, Ni, P, Ru, S, Sr, and Zn) were also monitored to provide trends for trace analysis. Among the nine additional analytes that were monitored was Ru to determine whether the noble metals (Ru as the major noble metal constituent) may be decreasing due to precipitation as a metal in the melter. Canister WV-289 results are exhibited as an example with the additional analytes (see Table 12). However, for the trace analytes, the concentration of the raw ICP readout was very close or less than the MDL and therefore the results on many of the analytes could not be utilized for trends. The less than concentration reflects that the ICP concentration was less than the empirical MDL.

Table 10. SHD Analysis: Mean, SD, and %RSD for a Representative Canister

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Mean	SD	% RSD
Al	3.12E+04	3.30E+04	3.08E+04	3.25E+04	3.15E+04	3.16E+04	3.09E+04	3.34E+04	2.98E+04	3.16E+04	1.15E+03	3.63
B	3.56E+04	3.81E+04	3.52E+04	3.70E+04	3.66E+04	3.69E+04	3.60E+04	3.92E+04	3.46E+04	3.66E+04	1.44E+03	3.94
Ca	3.39E+03	3.64E+03	3.53E+03	3.55E+03	3.42E+03	3.45E+03	3.37E+03	3.69E+03	3.20E+03	3.47E+03	1.50E+02	4.32
Fe	7.76E+04	8.33E+04	7.67E+04	8.12E+04	8.00E+04	8.10E+04	7.88E+04	8.61E+04	7.58E+04	8.01E+04	3.28E+03	4.10
K	3.67E+04	3.92E+04	3.63E+04	3.82E+04	3.78E+04	3.83E+04	3.76E+04	4.07E+04	3.65E+04	3.79E+04	1.41E+03	3.71
Li	1.60E+04	1.70E+04	1.58E+04	1.65E+04	1.82E+04	1.63E+04	1.60E+04	1.71E+04	1.52E+04	1.62E+04	5.89E+02	3.63
Mg	5.35E+03	5.70E+03	5.42E+03	5.82E+03	5.65E+03	5.70E+03	5.55E+03	6.11E+03	5.36E+03	5.63E+03	2.45E+02	4.34
Mn	5.78E+03	6.20E+03	5.73E+03	6.08E+03	6.00E+03	6.07E+03	5.91E+03	6.46E+03	5.70E+03	5.99E+03	2.45E+02	4.10
Na	5.96E+04	5.99E+04	5.53E+04	5.82E+04	5.86E+04	5.74E+04	5.63E+04	6.06E+04	5.38E+04	5.75E+04	2.27E+03	3.94
P	6.12E+03	6.41E+03	5.52E+03	6.16E+03	6.25E+03	6.27E+03	6.17E+03	6.65E+03	6.86E+03	6.27E+03	3.74E+02	5.97
Si	1.79E+05	1.92E+05	1.77E+05	1.86E+05	1.83E+05	1.86E+05	1.82E+05	1.98E+05	1.76E+05	1.84E+05	7.16E+03	3.88
Th	2.79E+04	2.81E+04	2.65E+04	2.71E+04	2.82E+04	2.86E+04	2.77E+04	2.82E+04	2.85E+04	2.79E+04	6.80E+02	2.44
Ti	4.53E+03	4.87E+03	4.48E+03	4.72E+03	4.67E+03	4.71E+03	4.59E+03	5.01E+03	4.39E+03	4.66E+03	1.94E+02	4.15
U	4.83E+03	5.00E+03	4.68E+03	4.88E+03	4.99E+03	4.87E+03	4.85E+03	5.20E+03	4.87E+03	4.89E+03	1.70E+02	3.49
Zr	9.10E+03	9.80E+03	8.98E+03	9.48E+03	9.35E+03	9.43E+03	9.17E+03	1.00E+04	8.80E+03	9.35E+03	3.84E+02	4.11

Canister WV-279, 9 Replicates

Table 11. SHD Analysis: Mean, SD, and %RSD for Ten Selected Canisters.

Mean

Can #	191	241 *	257	263	265	274	277	279	303	305
Al	3.01E+04	3.33E+04	3.14E+04	3.02E+04	3.04E+04	3.14E+04	3.08E+04	3.16E+04	3.69E+04	3.30E+04
B	3.77E+04	4.08E+04	3.74E+04	3.79E+04	3.68E+04	4.12E+04	3.69E+04	3.60E+04	4.02E+04	3.97E+04
Ce	3.36E+03	3.55E+03	3.37E+03	3.17E+03	3.20E+03	3.65E+03	3.44E+03	3.47E+03	3.54E+03	3.81E+03
Fe	8.58E+04	9.30E+04	8.12E+04	8.24E+04	7.83E+04	8.95E+04	8.79E+04	8.01E+04	8.92E+04	8.50E+04
K	3.70E+04	4.02E+04	4.03E+04	3.97E+04	3.81E+04	3.94E+04	3.72E+04	3.79E+04	4.00E+04	4.17E+04
Li	1.60E+04	1.73E+04	1.67E+04	1.66E+04	1.60E+04	1.81E+04	1.68E+04	1.62E+04	1.66E+04	1.68E+04
Mg	5.47E+03	5.59E+03	5.66E+03	5.33E+03	5.53E+03	5.93E+03	5.65E+03	5.63E+03	6.37E+03	5.97E+03
Mn	6.63E+03	6.93E+03	6.04E+03	6.11E+03	5.88E+03	6.63E+03	6.51E+03	5.99E+03	6.71E+03	6.38E+03
Na	5.96E+04	6.03E+04	5.66E+04	5.55E+04	5.44E+04	6.12E+04	5.68E+04	5.75E+04	6.16E+04	6.09E+04
P	4.93E+03	4.83E+03	5.04E+03	5.43E+03	4.96E+03	6.06E+03	6.17E+03	6.27E+03	5.32E+03	5.10E+03
Si	1.86E+05	2.01E+05	1.90E+05	1.82E+05	1.88E+05	2.07E+05	2.01E+05	1.84E+05	2.05E+05	1.95E+05
Th	2.78E+04	2.92E+04	2.50E+04	2.04E+04	2.35E+04	3.03E+04	2.68E+04	2.79E+04	2.62E+04	2.61E+04
Ti	4.52E+03	4.93E+03	4.88E+03	4.94E+03	4.69E+03	5.09E+03	4.91E+03	4.66E+03	5.01E+03	4.93E+03
U	5.23E+03	5.33E+03	4.79E+03	4.82E+03	4.65E+03	5.50E+03	5.41E+03	4.89E+03	5.31E+03	5.30E+03
Zr	9.84E+03	1.03E+04	9.45E+03	9.71E+03	9.28E+03	1.02E+04	9.79E+03	9.35E+03	9.84E+03	1.01E+04

Standard Deviation

Can #	191	241 *	257	263	265	274	277	279	303	305
Al	6.32E+02	1.04E+03	7.60E+02	5.22E+02	4.65E+02	5.60E+02	7.29E+02	1.15E+03	1.45E+03	7.86E+02
B	1.69E+03	1.61E+03	8.94E+02	7.09E+02	6.16E+02	9.29E+02	1.06E+03	1.44E+03	1.03E+03	1.07E+03
Ce	7.55E+01	1.85E+02	1.00E+02	8.99E+01	5.43E+01	1.18E+02	9.02E+01	1.50E+02	1.53E+02	3.42E+02
Fe	2.05E+03	3.30E+03	2.02E+03	1.39E+03	1.21E+03	1.87E+03	2.16E+03	3.28E+03	2.59E+03	2.29E+03
K	6.80E+02	1.12E+03	1.14E+03	6.84E+02	5.31E+02	8.03E+02	9.92E+02	1.41E+03	8.10E+02	6.67E+02
Li	3.02E+02	6.70E+02	4.78E+02	2.28E+02	2.09E+02	2.99E+02	4.16E+02	5.89E+02	4.95E+02	2.46E+02
Mg	1.65E+02	3.09E+02	1.44E+02	1.72E+02	9.31E+01	1.20E+02	1.21E+02	2.45E+02	3.45E+02	2.11E+02
Mn	1.67E+02	2.41E+02	1.47E+02	1.04E+02	8.60E+01	1.48E+02	1.56E+02	2.45E+02	2.31E+02	1.92E+02
Na	1.16E+03	2.21E+03	1.40E+03	8.65E+02	7.67E+02	1.04E+03	1.62E+03	2.27E+03	1.68E+03	9.69E+02
P	3.07E+02	2.85E+02	3.21E+02	2.04E+02	2.01E+02	1.35E+02	1.85E+02	3.74E+02	2.33E+02	1.12E+02
Si	5.70E+03	7.28E+03	4.28E+03	3.94E+03	2.69E+03	4.66E+03	4.92E+03	7.16E+03	5.12E+03	4.95E+03
Th	2.95E+03	1.04E+03	1.63E+03	2.17E+03	3.18E+03	8.91E+02	1.10E+03	8.80E+02	1.71E+03	9.02E+02
Ti	1.06E+02	1.93E+02	1.06E+02	8.55E+01	7.48E+01	1.12E+02	1.21E+02	1.94E+02	1.17E+02	1.22E+02
U	1.48E+02	2.14E+02	1.11E+02	8.23E+01	4.72E+01	1.14E+02	1.31E+02	1.70E+02	1.03E+02	1.23E+02
Zr	2.40E+02	3.93E+02	2.29E+02	1.55E+02	1.56E+02	1.68E+02	2.37E+02	3.84E+02	2.08E+02	2.23E+02

% Relative Standard Deviation

Can #	191	241 *	257	263	265	274	277	279	303	305
Al	2.10	3.13	2.42	1.73	1.53	1.78	2.37	3.63	3.94	2.35
B	4.48	3.94	2.39	1.87	1.67	2.25	2.63	3.94	2.57	2.69
Ce	2.25	5.21	2.98	2.83	1.70	3.23	2.62	4.32	4.33	8.97
Fe	2.39	3.55	2.49	1.69	1.54	2.09	2.46	4.10	2.90	2.69
K	1.84	2.79	2.82	1.72	1.39	2.04	2.67	3.71	1.53	1.60
Li	1.89	3.88	2.86	1.36	1.31	1.65	2.47	3.63	2.98	1.46
Mg	3.05	5.53	2.55	3.23	1.68	2.02	2.14	4.34	5.41	3.54
Mn	2.52	3.48	2.44	1.71	1.46	2.24	2.44	4.10	3.44	3.01
Na	1.94	3.67	2.57	1.56	1.45	1.70	2.85	3.94	2.73	1.59
P	6.23	5.79	6.37	3.76	4.05	2.23	3.00	5.97	4.38	2.19
Si	3.06	3.61	2.24	2.05	1.43	2.26	2.44	3.88	2.50	2.54
Th	8.45	3.55	6.52	10.63	13.53	2.94	4.08	2.44	6.53	3.45
Ti	2.34	3.92	2.26	1.73	1.69	2.20	2.47	4.15	2.33	2.47
U	2.83	4.02	2.32	1.71	1.02	2.07	2.42	3.49	1.93	2.31
Zr	2.44	3.83	2.42	1.60	1.68	1.82	2.42	4.11	2.12	2.21

- Indicates a canister randomly selected for shard samples.

- Indicates shards sampled for process control analysis.

Table 12. SHD Analysis: Mean, SD, and %RSD for Major and Minor Analytes for a Representative Canister

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6	Sample 7	Sample 8	Sample 9	Mean	SD	% RSD
Al	3.21E+04	3.16E+04	3.09E+04	3.24E+04	3.12E+04	3.26E+04	3.44E+04	3.11E+04	3.16E+04	3.20E+04	1.08E+03	3.36
B	4.22E+04	4.22E+04	4.18E+04	4.26E+04	4.15E+04	4.20E+04	4.20E+04	4.13E+04	4.18E+04	4.19E+04	3.91E+02	0.93
Ba	6.88E+02	6.85E+02	7.11E+02	7.15E+02	6.70E+02	6.98E+02	6.76E+02	6.84E+02	6.94E+02	6.91E+02	1.50E+01	2.17
Ca	3.54E+03	3.51E+03	3.48E+03	3.64E+03	3.53E+03	3.63E+03	3.80E+03	3.43E+03	3.49E+03	3.56E+03	1.14E+02	3.21
Ce	4.18E+03	4.19E+03	4.21E+03	4.19E+03	4.02E+03	4.19E+03	4.00E+03	4.03E+03	4.10E+03	4.12E+03	8.60E+01	2.09
Cy	6.58E+02	6.63E+02	6.51E+02	6.69E+02	6.47E+02	6.54E+02	6.82E+02	6.49E+02	6.69E+02	6.60E+02	1.15E+01	1.75
Fe	8.55E+04	8.54E+04	8.41E+04	8.61E+04	8.38E+04	8.53E+04	8.54E+04	8.33E+04	8.44E+04	8.48E+04	9.39E+02	1.11
K	4.15E+04	4.18E+04	4.11E+04	4.23E+04	4.13E+04	4.19E+04	4.23E+04	4.08E+04	4.13E+04	4.16E+04	5.23E+02	1.26
Li	1.82E+04	1.80E+04	1.78E+04	1.85E+04	1.78E+04	1.83E+04	1.90E+04	1.76E+04	1.80E+04	1.81E+04	4.27E+02	2.36
Mg	5.89E+03	5.81E+03	5.70E+03	5.96E+03	5.76E+03	6.09E+03	6.20E+03	5.75E+03	5.79E+03	5.88E+03	1.89E+02	2.88
Mn	6.48E+03	6.46E+03	6.35E+03	6.52E+03	6.37E+03	6.50E+03	6.52E+03	6.33E+03	6.40E+03	6.44E+03	7.50E+01	1.17
Na	6.16E+04	6.09E+04	6.08E+04	6.30E+04	6.10E+04	6.21E+04	6.50E+04	6.02E+04	6.16E+04	6.18E+04	1.45E+03	2.35
Nd	1.78E+03	1.76E+03	1.80E+03	1.77E+03	1.69E+03	1.76E+03	1.67E+03	1.73E+03	1.74E+03	1.74E+03	4.22E+01	2.42
Ni	2.17E+03	2.18E+03	2.13E+03	2.20E+03	2.20E+03	2.20E+03	2.19E+03	2.21E+03	2.22E+03	2.19E+03	2.67E+01	1.22
P	5.56E+03	5.88E+03	5.53E+03	5.76E+03	5.89E+03	5.63E+03	5.58E+03	5.52E+03	5.71E+03	5.87E+03	1.43E+02	2.52
Ru	<2.67E+02	<2.01E+02	<2.44E+02	<2.27E+02	<2.51E+02	<2.33E+02	2.21E+02	<2.31E+02	<2.18E+02	2.21E+02	N/A	N/A
S	<1.23E+03	<1.25E+03	<1.24E+03	<1.16E+03	<1.09E+03	<1.03E+03	<1.20E+03	<1.20E+03	<1.23E+03	N/A	N/A	N/A
Si	2.08E+05	2.07E+05	2.04E+05	2.09E+05	2.07E+05	N/A	2.07E+05	2.04E+05	2.06E+05	2.07E+05	1.77E+03	0.86
Sr	<6.16E+02	<6.27E+02	<6.18E+02	<5.78E+02	<5.43E+02	<5.15E+02	<6.00E+02	<6.01E+02	<6.17E+02	N/A	N/A	N/A
Th	2.72E+04	2.58E+04	2.79E+04	2.71E+04	2.67E+04	2.68E+04	2.59E+04	2.69E+04	2.73E+04	2.68E+04	6.64E+02	2.47
Ti	5.15E+03	5.17E+03	5.10E+03	5.21E+03	5.07E+03	5.14E+03	5.14E+03	5.03E+03	5.10E+03	5.12E+03	5.43E+01	1.06
U	5.50E+03	5.43E+03	5.48E+03	5.56E+03	5.43E+03	5.50E+03	6.72E+03	5.36E+03	5.51E+03	5.62E+03	4.18E+02	7.44
Zn	<6.16E+02	<6.27E+02	6.22E+02	<5.78E+02	<5.43E+02	<5.15E+02	7.14E+02	<6.01E+02	<6.17E+02	6.66E+02	6.51E+01	9.74
Zr	1.01E+04	1.01E+04	1.00E+04	1.02E+04	9.88E+03	1.01E+04	1.01E+04	9.84E+03	9.99E+03	1.00E+04	1.17E+02	1.17

Bold Italics - Indicate trace analytes.

Shaded - Indicates actual values were less than values shown. Calculations performed omitting shown values.

Canister WV-289, 9 Replicates

7.4 Discussion

The ICP results for the WI, GF, and WGF were based upon a target glass composition with upper and lower bounds of uncertainty based on a 95% confidence of the model used for the HLW composition. The %RSD utilization allowed for an evaluation of whether the analysis of a particular sample or samples had to be reanalyzed for internal consistency. The Vitrification Process Engineering group evaluated the ICP results and other desired parameters for batch acceptance and make-up in order to meet the target composition requirements of the canister HLW glass. A&PC evaluated the ICP results as an internal check. Using batch #50 WGF as an example, Table 13 displays the mean of the nine replicates, SD, %RSD, oxide factor, target oxide, + uncertainty limits for the analytes with percent (%) metal oxide > 0.5%, and the measured oxide with upper and lower target boundaries. The target boundaries were met with the exception of one analyte. The Ca analysis was slightly above the upper boundary, but was accounted for in the shard analysis due to the model confidence interval.

Table 13. WGF Batch #50 with %Oxide Target, Upper/Lower Boundaries, and Measured %Oxide

	Mean	SD	% RSD	Oxide Ratio	Target Oxide %	- %	+ %	LL	% Oxide	UL
Al	1.35E+04	3.74E+02	2.78	1.89	6.00	9.50	9.50	5.43	5.8947	6.57
B	1.77E+04	7.53E+02	4.25	3.22	12.89	15.00	15.00	10.96	13.2164	14.82
Ca	1.84E+03	9.90E+01	5.38	1.40	0.48	25.00	15.00	0.36	0.5956	0.66
Fe	3.49E+04	1.38E+03	3.97	1.43	12.02	15.00	15.00	10.22	11.5438	13.82
K	1.70E+04	5.84E+02	3.44	1.20	5.00	12.50	12.50	4.38	4.7216	5.63
Li	7.72E+03	2.93E+02	3.79	2.15	3.71	12.50	12.50	3.25	3.8463	4.17
Mg	2.46E+03	6.18E+01	2.51	1.66	0.89	15.00	15.00	0.76	0.9466	1.02
Mn	2.60E+03	9.47E+01	3.64	1.29	0.82	15.00	15.00	0.70	0.7778	0.94
Na	2.58E+04	9.62E+02	3.76	1.35	8.00	12.50	12.50	7.00	8.0041	9.00
P	2.32E+03	7.66E+01	3.38	2.29	1.20	15.00	15.00	1.02	1.2322	1.38
Si	8.41E+04	2.98E+03	3.54	2.14	40.98	5.50	5.50	38.73	41.8986	43.23
Th	1.19E+04	7.86E+02	6.63	1.14	3.56	25.00	25.00	2.67	3.1419	4.45
Ti	1.89E+03	9.03E+01	4.77	1.67	0.80	15.00	15.00	0.68	0.7323	0.92
U	2.21E+03	9.20E+01	4.16	1.20	0.63	25.00	15.00	0.47	0.6148	0.72
Zr	4.26E+03	1.52E+02	3.57	1.35	1.32	15.00	15.00	1.12	1.3323	1.52
TOTAL					98.30	98.3000				

Batch 50

In a similar table (see Table 14), the shard analysis of Canister WV-279 shows the target composition with upper and lower bounds in terms of percent oxide analysis and the determined % oxide. Comparison of the upper and lower bounds for the SHD composition with batch #50 shows that the glass will be within the compositional required bounds for the percent metal oxides by weight.

Table 14. Canister WV-279 SHD with %Oxide Target, Upper/Lower Boundary Limits, and Measured %Oxide

	Mean	SD	% RSD	Oxide Ratio	Target Oxide %	- %	+ %	LL	% Oxide	UL
Al	3.16E+04	1.16E+03	3.63	1.89	6.00	9.50	9.50	5.43	6.2555	6.57
B	3.66E+04	1.44E+03	3.94	3.22	12.89	15.00	15.00	10.96	12.3312	14.82
Ca	3.47E+03	1.50E+02	4.32	1.40	0.48	25.00	15.00	0.36	0.5088	0.66
Fe	8.01E+04	3.28E+03	4.10	1.43	12.02	15.00	15.00	10.22	11.9856	13.82
K	3.79E+04	1.41E+03	3.71	1.20	5.00	12.50	12.50	4.38	4.7844	5.63
Li	1.62E+04	5.89E+02	3.63	2.15	3.71	12.50	12.50	3.25	3.6541	4.17
Mg	5.63E+03	2.45E+02	4.34	1.66	0.89	15.00	15.00	0.76	0.9783	1.02
Mn	5.99E+03	2.45E+02	4.10	1.29	0.82	15.00	15.00	0.70	0.8093	0.94
Na	5.75E+04	2.27E+03	3.94	1.35	8.00	12.50	12.50	7.00	8.1902	9.00
P	6.27E+03	3.74E+02	5.97	2.29	1.20	15.00	15.00	1.02	1.5027	1.38
Si	1.84E+05	7.16E+03	3.88	2.14	40.98	5.50	5.50	38.73	41.2989	43.23
Th	2.79E+04	6.80E+02	2.44	1.14	3.56	25.00	25.00	2.67	3.3280	4.45
Ti	4.68E+03	1.94E+02	4.15	1.67	0.80	15.00	15.00	0.68	0.8154	0.92
U	4.89E+03	1.70E+02	3.49	1.20	0.63	25.00	15.00	0.47	0.6138	0.72
Zr	8.35E+03	3.84E+02	4.61	1.35	1.32	15.00	15.00	1.12	1.3206	1.52
TOTAL					98.30	98.3000				

Canister WV-279

The ten selected canister SHD analysis results are displayed in terms of the determined percent oxide analyte results along with the glass target percent oxide composition and its upper and lower bounds (see Table 15). These results demonstrated that the sample preparation and ICP analyses have verified the composition of the canisters. The low %RSDs of the WI, GF, and WGF analytical data contributes to the canister composition being within the glass target boundaries. This conclusion can be demonstrated in Table 15 that documents the analyte percent oxides of the ten selected canisters to be within the upper and lower target oxide limits.

Table 15. Ten Selected Canister SHD Analyses Demonstrating %Oxide is within Upper and Lower Limits

Can	% Oxide Glass Shards										Limit		Target %Oxide
	191	241	257	263	265	274	277	279	303	305	Lower	Upper	
Al	5.8719	6.0632	6.1267	5.8969	6.0850	5.6473	5.7492	6.2595	6.6788	6.1874	5.43	6.57	6.00
B	12.5354	12.6301	12.4214	12.6268	12.5526	12.6230	12.6807	12.3312	12.4106	12.6911	10.96	14.82	12.89
Ca	0.4859	0.4782	0.4872	0.4592	0.4740	0.4856	0.4758	0.5088	0.4746	0.5292	0.36	0.55	0.48
Fe	12.6750	12.7992	11.9906	12.1899	11.8540	12.1715	12.4097	11.9856	12.2183	12.0706	10.22	13.82	12.02
K	4.5917	4.6466	4.9925	4.9332	4.8431	4.5026	4.4059	4.7644	4.5957	4.9719	4.38	5.63	5.00
Li	3.5508	3.5751	3.7042	3.7001	3.6394	3.7063	3.5755	3.6541	3.4291	3.5964	3.25	4.17	3.71
Mg	0.9383	0.8931	0.9694	0.9149	0.9717	0.9362	0.9260	0.9783	1.0134	0.9849	0.76	1.02	0.89
Mn	0.8840	0.8600	0.8041	0.8158	0.8024	0.8137	0.8295	0.8093	0.8292	0.8176	0.70	0.94	0.82
Na	8.3109	7.8317	7.8836	7.7506	7.7798	7.8535	7.5704	8.1302	7.9658	8.1659	7.00	9.00	8.00
P	1.1672	1.0858	1.1915	1.2876	1.2020	1.3210	1.3960	1.5027	1.1684	1.1603	1.02	1.38	1.20
Si	41.2129	41.4890	42.0740	42.5120	42.5176	42.2061	42.5135	41.2999	41.9694	41.3695	38.73	43.23	40.98
Th	3.2749	3.2074	2.9370	2.4049	2.8335	3.2846	3.0138	3.3260	2.8621	2.9560	2.67	4.45	3.56
Ti	0.7799	0.7924	0.8069	0.8528	0.8290	0.8086	0.8090	0.8154	0.8017	0.8170	0.68	0.92	0.80
U	0.6484	0.6156	0.5940	0.5984	0.5903	0.6282	0.6404	0.6138	0.6106	0.6321	0.47	0.72	0.63
Zr	1.3728	1.3328	1.3169	1.3567	1.3257	1.3119	1.3047	1.3209	1.2723	1.3500	1.12	1.52	1.32

- Indicates a canister randomly selected for shard samples.

- Indicates shards sampled for process control analysis.

8.0 CONCLUSION

The successful startup and operation of the second generation JY-46P ICP™ glovebox assembly in the spring of 1996 allowed supporting the startup of radioactive operations of the Vitrification Process Control Plan (PCP) in June 1996 with two independent operational ICP glovebox systems for compositional analyses. The two ICP glovebox assemblies have been supporting the Vitrification PCP with analysis of these various process streams:

- Submerged Bed Scrubber (SBS) addition to the heel in the CFMT (Concentrator Feed Makeup Tank)
- Waste Initial (WI) addition to the CFMT
- Glass Formers (GF) = Cold Chemicals
- Waste plus Glass Formers (WGF)
- Glass Shards (SHDs) retrieved from the canisters.

The ICP analyses have determined the compositional makeup of the batch process streams and then confirmed that the glass product constituents were within the upper and lower limits of the %oxide target. This ICP analysis support has been on a continuous basis without impacting the melter process. The use of a service contract and maintenance of spare parts has further ensured continuous use of the ICP glovebox systems. This support will continue until the end of the vitrification campaign with ~300 canisters being produced.

9.0 ACRONYMS AND ABBREVIATIONS

AAS	Atomic Absorption Spectrophotometer
AC	Analytical Cell
Ag	Silver
Al	Aluminum
A&PC	Analytical and Process Chemistry
As	Arsenic
B	Boron
Ba	Barium
C	Celsius (degrees)
Ca	Calcium
CCV	Continuing Calibration Verification
Cd	Cadmium
Ce	Cerium
CFM	Cubic feet per minute
CFMT	Concentrated Feed Make-Up Tank
Co	Cobalt
conc.	Concentrated
Cr	Chromium
CRL	Central Research Labs
Cu	Copper
DOE	United States Department of Energy
EPA	United States Environmental Protection Agency
FACTS	Functional and Checkout Testing of Systems
Fe	Iron
GF	Glass Former
HEPA	High Efficiency Particulate Air
HF	Hydrofluoric Acid
Hg	Mercury
HLW	High-Level Waste
HNO ₃	Nitric Acid
H ₂ O ₂	Hydrogen Peroxide
hr	Hour(s)
ICP	Inductively Coupled Plasma Spectrometer
ICP/AES	Inductively Coupled Plasma/Atomic Emission Spectroscopy
ICP/MS	Inductively Coupled Plasma/Mass Spectrometer
ICV	Initial Calibration Verification
ID	Internal Dimension
IEC	Inter-element correction

ISA	Instruments SA, Inc.
g	Gram(s)
GF	Glass Formers
JY	Jobin Yvon
JY-32	Jobin Yvon brand Spectrometer Model 32
JY-38	Jobin Yvon brand Monochromator Model 38
JY-46P	Jobin Yvon brand ICP Model 46P
JY-50P	Jobin Yvon brand ICP Model 50P
JY-70	Jobin Yvon brand ICP Model 70
K	Potassium
K	Thousand(s)
KOH	Potassium Hydroxide
kW	Kilowatt(s)
L	Liter(s)
La	Lanthanum
Li	Lithium
LL	Lower Limit
LOD	Limit of Detection
LPM	Liters per minute
M	Molar
Mass Spec	Mass Spectrometry
MDL	Method Detection Limits
MFHT	Melter Feed Hold Tank
Mg	Magnesium
MHZ	Megahertz
min	Minute(s)
mL	Milliliter(s)
mm	Millimeter(s)
Mn	Manganese
Mo	Molybdenum
M	Megohms
mR	Millirem
Na	Sodium
Nd	Neodymium
Ni	Nickel
NFS	Nuclear Fuel Services, Inc.
NIST	National Institute for Standards and Technology
P	Phosphorus
Pb	Lead
Pd	Palladium

Pr	Praseodymium
PCP	Process Control Plan
PMT	Photomultiplier Tubes
PVC	Polyvinyl Chloride
QC	Quality Control
RCT	Radiological Control Technicians
RE	Radiological Engineering
RF	Radio Frequency
Rh	Rhodium
RPD	Relative Percent Difference
RSD	Relative Standard Deviation
Ru	Ruthenium
RWP	Radiological Work Permit
S	Sulfur
SBS	Submerged Bed Scrubber
Sc	Scandium
SC	Sampling Cell
SD	Standard Deviation
Se	Selenium
SGN	Societe Generale Pour Les Techniques Nouvelles
SHD	Glass Shard
Si	Silicon
Sm	Samarium
Sn	Tin
Sr	Strontium
SSC	Sample Storage Cell
Std.	Standards
STS	Supernatant Treatment System
TCLP	Toxicity Characteristic Leaching Procedure
Te	Tellurium
Th	Thorium
Ti	Titanium
TM	Trade Mark
U	Uranium
UL	Upper Limit
Vit	Vitrification
WGF	Waste plus Glass Formers
WI	Waste Initial
WV-	West Valley Vitrification Canister Number (e.g., WV-279)
WVDP	West Valley Demonstration Project

WVNS	West Valley Nuclear Services Company
w/w	Weight/weight measurement (e.g., micrograms/grams)
Y	Yttrium
Zn	Zinc
Zr	Zirconium
°	Degrees
L	Microliter(s)
%	Percent
‘	Foot (feet)
“	Inch(es)

10.0 BIBLIOGRAPHY

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DOE/NE/44139-89

WEST VALLEY DEMONSTRATION PROJECT

DOE

**Inductively Coupled Plasma-Atomic Emission
Spectroscopy Glovebox Assembly System at the
West Valley Demonstration Project**