LA-UR-12-23790
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Title:	Characterization of Representative Materials in Support of Safe, Long Term Storage of Surplus Plutonium in DOE-STD-3013 Containers
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Intended for:	Report

Characterization of Representative Materials in Support of Safe, Long Term Storage of Surplus Plutonium in DOE-STD-3013 Containers

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Abstract

The Surveillance and Monitoring Program is a joint Los Alamos National Laboratory / Savannah River Site effort funded by the Department of Energy-Environmental Management to provide the technical basis for the safe, long-term storage (up to 50 years) of over 6 metric tons of plutonium in over 5,000 DOE-STD-3013 containers at various facilities around the DOE complex [1]. The majority of this material is nuclear weapons program surplus plutonium, and much of it is destined for conversion to mixed oxide fuel for use in US nuclear power plants. The form of the plutonium ranges from relatively pure metal and oxide to very impure oxide. The performance of the 3013 containers is dependent on moisture content and on the levels, types, and chemical forms of the impurities. The oxide materials that present the greatest challenge to storage are those that contain chloride salts. Other common impurities include compounds of calcium, magnesium, iron, and nickel. Over the past 15 years, the program has collected a large body of experimental data on 54 plutonium oxide samples, 53 of which represent the broader population of materials in storage. This paper summarizes the moisture analysis, particle size, surface area, density, wattage, actinide isotopic composition, trace element, and shelf-life surveillance data, including origin and process history information. Limited characterization data on fourteen non-representative samples are also presented.

Introduction

The Materials Identification and Surveillance (MIS) Program (a sub element of the overall Surveillance and Monitoring Program) at Los Alamos National Laboratory (LANL) is charged with characterizing and evaluating nuclear materials in long term storage per the DOE-STD-3013 Standard [1]. The MIS program also provides technical support to the processing sites to assist in the resolution of problems that develop during stabilization and packaging activities. In addition, Section 6.6 of the 3013 Standard states “As a part of site Quality Assurance Plans, the sites are responsible for assuring that materials being packaged to this Standard are represented by the items accumulated in the Materials Identification and Surveillance Program.” The MIS Program is guided by a working group consisting of Department of Energy (DOE) officials and DOE contractors including LANL, Lawrence Livermore National Laboratory (LLNL), Hanford Site, and Savannah River Site (SRS). The MIS characterization and evaluation activity began in earnest in 1997 with receipt of the first items from the inventory at LANL, Rocky Flats Environmental Technology Site (RFETS) and Hanford. These MIS samples are full-size representations of items planned for long-term storage and were selected by individual site representatives for characterization and analysis in the program. This paper is an update to a 1999 characterization status report [2] and an expansion on a 2010 characterization summary report [3], and provides a comprehensive set of data gathered by the stabilization and characterization component of the MIS Program. This characterization data is an essential element of the technical basis for the safe, long-term storage of over 6 metric tons of plutonium in over 5,000 containers stored across the DOE complex.

Material Representation

The MIS Program has a collection of 53 unique samples currently used for representation of materials packaged in 3013 containers. This inventory of represented materials is referred to the MIS inventory. Characterization data on these samples as well as one non representative MIS sample, MISSTD-1, are discussed in detail. Limited characterization data on 14 non-representative samples is also included. The samples in the MIS program were provided by RFETS, Hanford, and LANL to represent their inventories of materials. SRS and LLNL identified these materials as representing their site inventories through technical justification documents [4-10]. The containers packaged by SRS were linked to representative samples in the MIS inventory that have a similar processing history, and the containers packaged by LLNL were linked to representative samples based on the impurities detected by prompt gamma analysis [4].

Historical information for the representative samples was provided if they were available. In cases, where the historical information was incomplete, new characterization data sorted the material into the appropriate category. For the purpose of this report, each of the samples was placed into a process category and subcategory, based on the process in which it was produced. The categories, which included aqueous processing, metal oxidation, miscellaneous, mixed actinide and molten salt operation, are discussed in detail below. The subcategory was either the product output from the operation or a byproduct from an effluent stream that contained too much plutonium to be discarded as waste. Classification of materials as product or byproduct was obtained from originating site process history documentation. A summary of the general process categories represented by the MIS inventory are presented in Table 1 along with the ranges of plutonium and uranium assay. A complete listing of the MIS inventory is given in Appendix 1. Example photographs are presented in Figure 1 and show the variety of materials that are stored in 3013 containers and the bulk effects of processing (calcination and milling).

Table 1. Summary of the MIS Inventory of Samples That Represent the Broader Inventory of Plutonium-Bearing Materials in Long Term Storage

Process Category	Process Subcategory	Source Site	Number of Items	Min Pu%	Max Pu%	Min U%	Max U%
Aqueous Processing	Byproduct	Hanford	4	29.0	65.6	0.0	0.0
		RFETS	4	7.7	67.9	0.0	0.0
	Product	Hanford	2	85.2	87.5	0.0	0.0
		LANL	7	76.6	87.8	0.0	0.0
		RFETS	2	84.2	84.5	0.0	0.0
Metal Oxidation	Byproduct	Hanford	1	86.3	86.3	0.0	0.0
		RFETS	1	69.8	69.8	0.0	0.0
	Product	RFETS	5	77.7	87.0	0.0	0.0
		LANL	1	87.0	87.0	0.0	0.0
Miscellaneous	Miscellaneous	Hanford	3	32.7	83.4	0.0	0.0
		LANL	2	66.8	82.9	0.0	0.0
Mixed Actinide Operations	Byproduct	Hanford	1	13.4	13.4	65.1	65.1
		LANL	1	36.1	36.1	14.7	14.7
		RFETS	5	13.8	85.9	0.5	69.2
	Miscellaneous	LANL	2	6.0	84.6	2.0	79.9
	Product	LANL	2	6.2	17.5	70.0	78.3
		RFETS	1	85.1	85.1	0.1	0.1
Molten Salt Operations	Byproduct	Hanford	3	39.6	70.9	0.0	0.0
		LANL	2	71.9	74.2	0.0	0.0
		RFETS	6	33.7	77.7	0.0	0.0

Metal Oxidation Product, RFETS, 011608 (“as-received”) 	Aqueous Processing Product Quality Oxide, Hanford, BLO-39-11-14-004 (“as-received”) 
Molten Salt Operations Byproduct Oxide, Hanford, ARF-102-85-295 (“as-received”) 	Molten Salt Operations Byproduct Oxide, Hanford, ARF-102-85-295 (After Calcination) 
Aqueous processing Byproduct Oxide, RFETS, 07242201A (“as-received”) 	Aqueous processing Byproduct Oxide, RFETS, 07242201A (After Rod-milling) 

Figure 1. Photographs of materials received from Hanford and RFETS for characterization.

Aqueous Processing

The materials in this process category include product oxides produced in the oxalate precipitation, peroxide precipitation, and magnesium hydroxide precipitation processes. Also included are the oxides generated as byproducts from these operations such as dissolution residues (heels that could not be dissolved from processing foundry and scrap oxide in the oxide dissolution process) and low-purity oxides from the magnesium hydroxide precipitation process.

Aqueous processing at Hanford was used to produce product quality oxide through oxalate precipitation or to recover plutonium and uranium from impure solutions through magnesium hydroxide precipitation and oxalate precipitation [10]. The product oxides were made to fast flux test facility (FFTF) feedstock specifications, or RFETS metal production operations in the oxalate (IV) precipitation process. The processing was done in either remote mechanical line A (RMA) in the plutonium uranium extraction (PUREX) N-Cell or in the plutonium finishing plant (PFP). In the case of the MIS samples, BLO-39-11-14-004 "Button Line Oxide" was produced in RMA Line in November 1979, and PBO-47-09-012-023 "PUREX Blend Oxide" was produced in the PUREX N-Cell in September 1987. Precipitates were generally calcined to convert the oxalate to plutonium oxide in a 2-stage calciner, which had the first stage between 350 and 450°C and the second stage between 475 and 575°C. MIS samples 66-00-11-355 and 66-01-01-439 were produced in the magnesium hydroxide precipitation process in November 2000 and January 2001, respectively, with the former being considered lean in Pu and the latter considered rich in Pu. Together these materials represent the range of materials produced by this process.

Rocky Flats used the hydrogen peroxide precipitation process to produce product quality oxide for metal production. In the RFETS aqueous processing, materials were selected and sent for nitric acid dissolution, and the plutonium was precipitated as plutonium (IV) peroxide, which was calcined at 450°C to produce oxide [11, 12]. MIS samples 07161856 and 1000089 were produced in this manner. Undissolved solids remaining after multiple passes through the dissolution process were removed and stored as dissolution residues. MIS samples of these byproduct materials include 07032282A, 07242165A, and 07242201A. Additionally, MIS sample ARF-102-85-355 (a RFETS material that was sent to Hanford for recovery) may have originated in this process, based on its composition.

Plutonium and uranium were recovered from impure solutions at RFETS through the magnesium hydroxide precipitation process. Likewise to the Hanford process, this process produced various grades of oxide. MIS sample 39-01153A is a very lean plutonium content oxide containing only 7.7% Pu. The lean materials produced in this process (less than 30% Pu) were eventually sent to WIPP in lieu of storage in a 3013 container.

LANL produces product quality oxides from both aqueous chloride and aqueous nitrate processing operations. The Experimental Chloride Extraction Line (EXCEL), which has been operational since March of 1993, is used to recover, purify, stabilize, and produce plutonium oxide suitable for long-term storage or future weapons application. In the EXCEL line, hydrochloric acid is used to dissolve and leach various feed materials such as metal turnings, impure metals and pyrochemical residues. The feed materials are primarily residues from pyrochemical processing. The bulk of these residues are chloride salts containing significant amounts of plutonium. Solvent extraction is the primary process used to separate the plutonium from the impurities. Significant impurity decontamination is also achieved through oxalate precipitation for select elements. For select feeds, aqueous chloride operations have operated without the solvent extraction and generated product oxides with significantly lower impurity levels than the input feed. After purification the plutonium is precipitated as plutonium (III) oxalate. The plutonium oxalate cake is calcined to produce high purity Pu(IV) oxide product. Calcination conditions have varied

but typically the cake was calcined from 4-8 hours at temperatures ranging from 600 – 650°C. Typically EXCEL products were then sent for blending and further calcinations to 950°C. MIS samples CXLPROD012102 ((87.8% Pu) and CXLPROD091901 (87.6% Pu) were produced in the EXCEL line. CXLNM1 is a pure plutonium oxide standard produced for the MIS program from a mixture of six items produced **in** the EXCEL line. This material intended for use in neutron moderation experiments. An archive sample was calcined at 950°C and received full characterization. For EXCEL line product CXLOX091802, the dissolution solution went directly to oxalate precipitation and was not purified in solvent extraction, resulting in an oxide with only 76.6% Pu. The input item was impure and originated from chemistry sample returns.

Aqueous nitrate operations at LANL are used to recover and purify plutonium from a wide variety of impure scrap materials that do not contain chloride. The materials are dissolved in a refluxing solution of nitric acid containing fluoride ions. Undissolved residues are the primary byproduct from aqueous nitrate operations. Anion exchange is used to purify the solution. Typically the plutonium is precipitated as plutonium (III) oxalate then calcined to convert the oxalate to plutonium (IV) oxide product. When aqueous nitrate operations were first started in 1978 oxalate cakes were typically calcined from 450°C – 500°C for approximately six hours. Since 1987, oxalate cakes were typically calcined at 600°C for four to six hours. The sample PEOR3258 was produced in LANL aqueous nitrate operations prior to 1992. Typically, the process feed at that time consisted of lean residue material with low Pu content such as ash, sand, slag, and crucibles. Two MIS samples are blends of several items produced in aqueous nitrate processing. MISSTD-1 is a pure plutonium oxide standard produced for the MIS program from a mixture of five items produced in the lean residue anion exchange process from Pu nitrate solutions prior to 1992. Lean residue operations typically ran. This material was found to have a high surface area in its “as-received” state and readily absorbs moisture at low relative humidity (less than 15%). MISSTD-1 was never calcined to the 3013 standard requirements, so this item is not considered representative. MIS sample PEOF1 is a pure plutonium oxide standard produced for the MIS program from a mixture of four items produced the aqueous nitrate operations. This material was calcined at 975°C for four hours.

Metal Oxidation

The materials in this process category include product and byproduct oxides generated by the burning or oxidation of plutonium metal. The quality of the oxide depends on the source of the material. In general, product quality oxides were generated by burning pure metal (buttons, ingots, turnings) to oxide. Product quality oxide was also produced during operations that melt and blend metal. Byproduct quality oxides were generated from the burning of casting skulls, metal screenings, or from sweepings. Metal brushings may fall into either category depending on the purity of the metal.

The MIS samples from RFETS metal oxidation processes include 011608, TS707001, 011589A, and TS707013. The first two samples are high purity oxides. The latter two contained 1.6 and 8.1 wt% chlorine, respectively, when received into the MIS program and may have been generated by burning casting skulls. MIS sample 07221730 is a high purity oxide that originated in metal and chip burning operations, and MT1490 is a high purity oxide with neptunium that originated in research and development operations. Additionally, MIS sample ARF-102-85-114-1 (a RFETS material that was sent to Hanford for recovery) may have originated in one of metal oxidation processes, based on its composition.

One MIS sample originated at LANL and was produced by direct metal oxidation in the Advanced Recovery and Integrated Extraction System (ARIES) process (UPOPLOT0003). This material was calcined above 950°C prior to being received in the MIS program.

Mixed Actinide Operations

The materials in this process category include product and byproduct plutonium/uranium mixed oxides from a variety of processes at RFETS, Hanford, and LANL. At RFETS the processes that generated mixed plutonium/uranium oxides included hydride oxidation (5501407 and 5501579), special assembly projects (669194), oxide dissolution (62750), hydroxide precipitation (053038), and other operations associated with research and development (CAN92). The hydride oxidation process was used to separate uranium and plutonium metal by reacting with hydrogen gas to form hydrides [12]. Oxides were formed for use in other processes by reacting the hydrides with oxygen. Two MIS samples (5501407 and 5501579) were generated in this process.

LANL included MIS samples from fuel production (SCP711-46 and SCP711-56) in the MIS inventory as well as a mixture of pure plutonium oxide and depleted uranium (CXL1685). The latter was originally intended for neutron moderation experiments, but an archive sample was accepted into the MIS program as a representative sample and characterized. In addition, a mixed oxide with an unknown origin (PuUOXBC05) was included early on in the program.

Hanford also generated mixed oxides from a variety of processes, but most of the packaged materials that originated in these processes are represented by the RFETS and LANL samples. One MIS sample (PSU-85-06-05) originated at Hanford in pyrolytic processing and contains mixed oxide recovered from polycubes, a compacted mixture of high purity plutonium/depleted uranium oxide and polystyrene [10].

Molten Salt Operations

The materials in this process category include byproduct Pu oxides that originated in “pyrochemical processes”, a generic term used by RFETS to describe a variety of activities used to process plutonium at high temperature. Most of these processes used immiscible molten-salt molten-metal phases, and the oxides generated are high in chloride salt impurities [11]. Historically, the byproducts from these processes accumulated at RFETS, and a portion of this inventory was transferred to Hanford, LANL and Savannah River for recovery in the mid-1980s as part of the Interim Plan for Recovery of Plutonium Program (IPRP). Most of that material was never recovered, however, and it remained at these sites until it was eventually packaged in 3013 containers for long-term storage.

Several of the MIS Samples from RFETS originated in the electrorefining process (C00695, CLLANL025, C00024A), and pyrochemical R&D operations (520610020). The oxide screenings items (07242141A and C06032A) were generated during the packaging process for the IPRP program. The processing history for MIS sample C00024A indicates that this item was unable to be packaged for offsite shipment during the IPRP program because it had failed LOI.

The Hanford materials in this process category (ARF-102-85-223, ARF-102-85-295, and ARF-102-85-365) originated in the RFETS “pyrochemical processes”. These materials, high in chloride salt impurities, were calcined at 450°C and packaged for offsite shipment to Hanford.

The LANL MIS samples in this process category originated in LANL processes. MIS sample PMAXBS was created in the MIS program by mixing burnt anode heels and material from the electrorefining process, similar to how RFETS prepared many of the pyrochemical materials. MIS sample ATL27960 originated in the advanced testing line for actinide separation, and this sample was characterized early in the program but is no longer available.

Miscellaneous Oxides

The remaining materials do not fall into any particular category. These materials originated in a variety of processes and include sources and standards, oxide from research and development operations, and other scrap oxides from a variety of processes.

This category includes three MIS samples that originated at Hanford (64-85-12-1858, 41-85-08-1379, and PPSL-365). MIS samples 64-85-12-1858 and 41-85-08-1379 were generated in 1985 and represent the large population of scrap oxides generated at PFP with 30 to 80 wt% Pu (along with 63-88-06-121 in the aqueous processing byproduct category). MIS sample 64-85-12-1858 originated in C-Line as a byproduct of metal production, and 41-85-08-1379 is analytical laboratory scrap material, which is likely a mixture of material from a variety of processes. MIS sample PPSL-365 is a high purity oxide that was produced by direct denitration and was intended to represent the population of PFP scrap oxides with greater than 80 wt% plutonium.

This category also includes two MIS samples that originated at LANL (PuF4-1 and 04272-CC-220). MIS sample PuF4-1 is a plutonium fluoride source that was produced by plutonium fluoride precipitation. This material was intended to represent other materials in the complex that contain fluoride when packaged, and other materials identified by prompt gamma analysis to be high in fluoride. MIS sample 04272-CC-220 is a mixture of items that contain plutonium oxide with thorium. These items were combined during 3013 packaging and were identified as unique materials. As a result, an archive sample, which bears the name of the convenience container, was transferred to the MIS program for further study in the surveillance program. To date this material has not received full characterization; only prompt gamma analysis and LOI have been performed.

Sampling

The typical processing path followed by MIS samples received at LANL is shown in Figure 2. The samples were first selected by the packaging sites, based on a materials representation plan approved by the MIS Working Group. Samples were stabilized prior to shipping but specific stabilization conditions are unknown for most samples. The receiving site performed the required nuclear materials accountability receipt verifications once an item was-received, which included gamma-ray spectrometry and calorimetry, and in some cases radiography. The items were then transferred to the unpackaging glovebox where they were logged in, assigned tracking codes, visually inspected for damage, weighed, photographed, and opened. Once opened, the contents of each container were inspected and photographed. Early in the program, items were sent for can puncture and gas sampling [2] before the inner package was opened. Based on resource constraints, later items were not gas sampled. Approximately nine different samples were pulled from each parent item including the fines and chunks after sieving through a 40 mesh screen and V-blending for one hour. The balance of the material was placed into a ceramic calcination boat for subsequent calcination at 600-950°C.

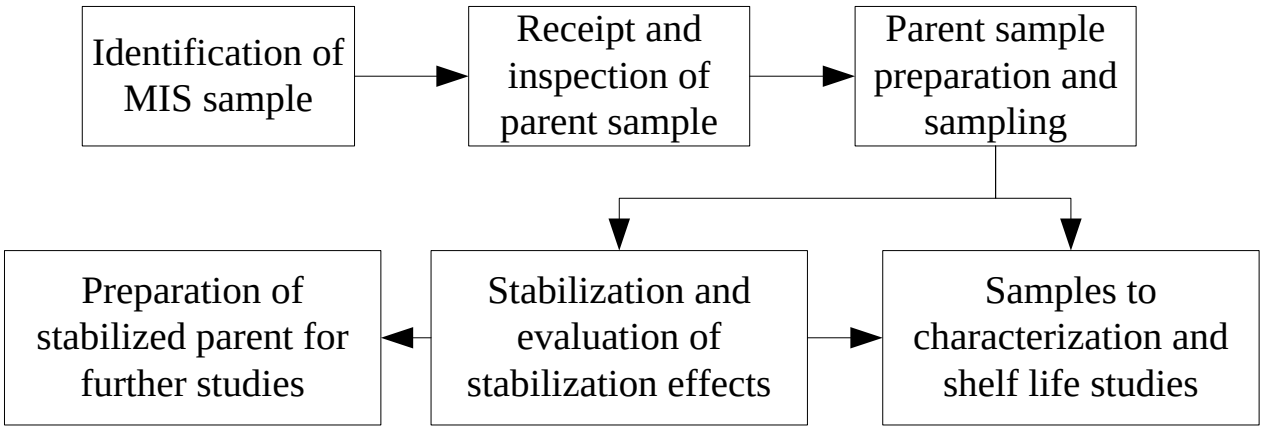

Figure 2. Sample preparation and processing path for MIS samples.

Weight Loss from Calcination

Each MIS item was calcined at specified temperatures to develop the technical basis in the 3013 Standard stabilization requirements. A portion of all the materials was stabilized to 950°C for a minimum of 2 hours. To examine the effects of calcination temperature upon stabilization, some parent items were split and a portion was calcined at a lower temperature (600°C for 12 hours or 800°C for 1 hour) prior to the 950°C calcination. In addition, some materials were sequentially calcined, first at 600°C, then at 950°C. The weight lost upon calcination of the samples varies significantly depending on the level of moisture and other impurities. The average percent weight loss after calcination at 950°C is illustrated in Figure 3 and summarized in Appendix 2, Table A2-1. As expected, the byproduct oxides lose significantly more weight than the product quality oxides. Also, the byproduct oxides from aqueous processing and molten salt operations tend to lose significantly more weight than the byproducts from metal oxidation and mixed actinide operations. The highest weight loss for any sample (53 wt %) was observed in 39-01153A, an aqueous byproduct that contained less than 8% plutonium. The high weight losses seen at higher temperatures for the molten salt residues are mostly attributed to loss of NaCl and KCl salts. Detailed data on the percent weight loss due to calcination by item is presented in Appendix 2, Table A2-2 and is shown in Figure 4. Appendix 2, Table A2-3 includes data for the nonrepresentative samples

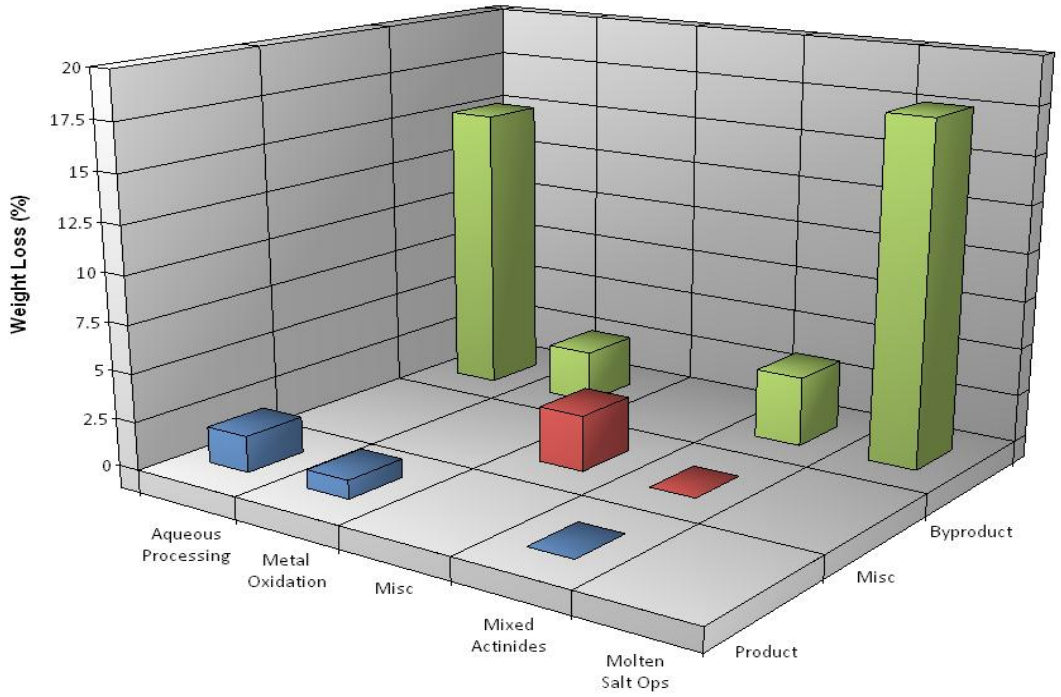

Figure 3. Average material weight loss after calcination at 950°C.

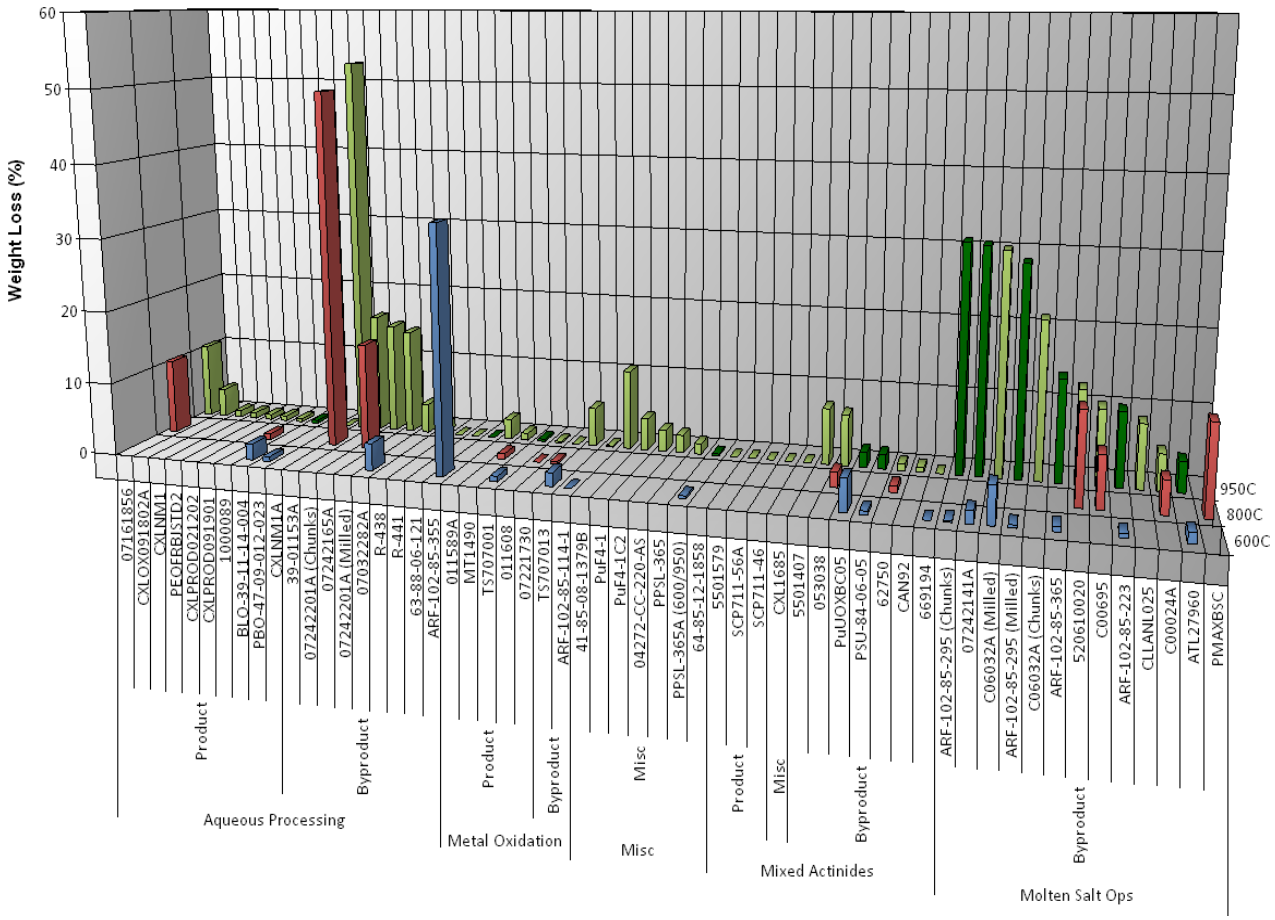

Figure 4. Material weight loss after calcination at 950°C.
Note: For the MIS samples calcined at 950°C, the light-green bars indicate that "as-received" material was calcined at 950°C, and dark-green bars indicate that material was calcined at 600°C before to calcination at 950°C.

Characterization

The parent materials were sampled for the following analyses:

- Moisture Analysis by Loss on Ignition (LOI) and/or thermal gravimetric analysis (TGA)
- Particle Size Distribution
- Morphology (Scanning Electron Microscope (SEM) images)
- Specific Surface Area
- Density (Pycnometer (Crystal)[1] Density, Bulk Density and Tap Density)
- Wattage and Actinide Composition (Calorimetry and Gamma-Ray Spectrometry)
- Trace Element Impurity Analysis (Prompt Gamma Analysis, Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), ICP-MS, Ion Chromatography (IC), Ion Selective Electrode)
- Shelf Life Surveillance

Moisture Analyses by LOI and TGA

Criteria for stabilizing plutonium-bearing materials require verification that moisture content is less than 0.5 wt % at the time of packaging [1]. Approved methods for determining the moisture content on the materials being packaged include LOI and TGA measurements. In the LOI technique, 5 to 10-g samples are placed in a pre-weighed crucible, weighed, and heated in air to nominally 1000°C for 2 hours [13]. The samples are then cooled and reweighed. The difference in the weight before and after heating is the loss on ignition. Note that some samples were cooled in air while others were cooled in a mixed Ar / air atmosphere.

The LOI technique was performed on MIS samples both in the “as-received” (AR) condition, and after calcination to 600°C, 800°C and 950°C. The LOI data for the MIS samples is shown in Figure 5 and is tabulated in Appendix 3. In general, the byproduct materials have higher LOI values, due to the presence of impurities, such as chloride salts, that volatilize at high temperature. MIS samples ARF-102-85-355 and 39-01153A have exceptionally high LOI values in the “as-received” condition. This is consistent with the total weight loss observed in Figures 3 and 4. The high LOI values are due to carbon compound(s) in MIS sample ARF-102-85-355, and to the overall high impurity content in 39-01153A. Table A3-2 compares the average LOI results by process category and subcategory before and after the material was calcined to 950°C. With the exception of the materials from molten salt operations, the data show that the LOI for most of the MIS samples was reduced after the material was calcined to 950°C. The mixed oxide items, and one miscellaneous item, gained weight when heated. When heated in air, mixed oxide items will gain weight due to an increase in the oxygen content with heating and the formation of more oxidized uranium species.

The average LOI percent weight change arranged by process category for samples calcined to 950°C is summarized in Appendix 3, Table A3-1. Molten salt byproducts exhibited the highest average weight percent loss, 5.3%, due primarily to the loss of chloride salts. For most categories, the average percent weight loss by LOI exceeded the 0.5 wt% criteria. LOI measurements, which measure the mass loss upon heating, do not distinguish between mass loss due to moisture and mass loss due to other constituents that volatilize below nominally 1000°C. Therefore, the LOI measurements indicate apparent moisture contents higher than the actual moisture content, especially for impure materials that tend to have a high concentration of salts.

[1] Refered to as particle density in the DOE-STD-3013-2012.

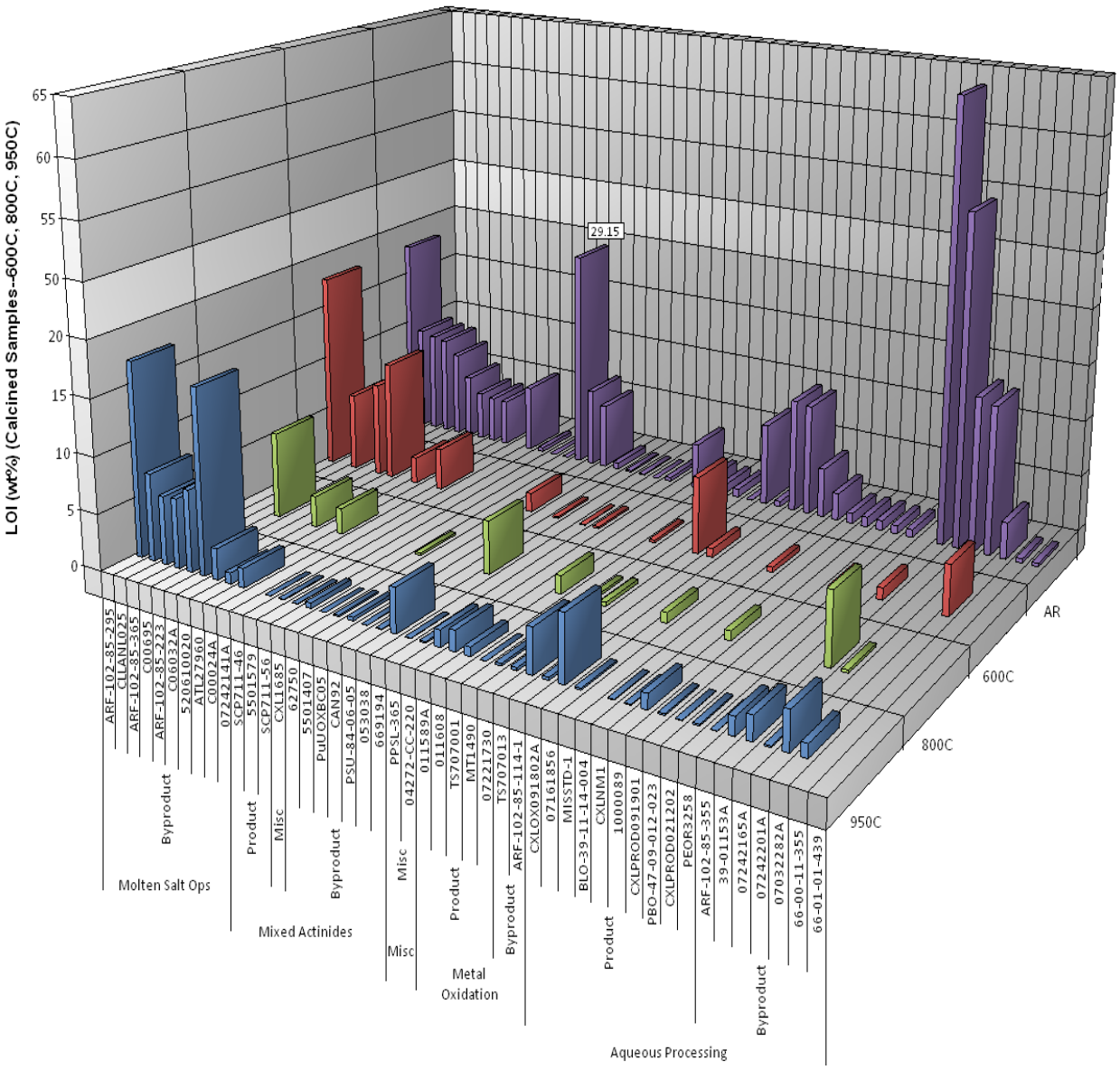

Figure 5. LOI results for MIS samples at various conditions arranged by process category and subcategory.

TGA is an instrument and technique that measures the weight change of a material as it is heated over a known temperature profile. The heating rate and end point temperature are set by the user, and can be varied. In this manner, thermally-induced chemical reactions, phase changes, or volatile mass losses can be monitored directly [14]. At LANL, 3 to 5 gram samples are heated at a fixed rate of 10°C per minute to 1100°C. This technique gives a mass loss to 1000°C that is equivalent to LOI. Like LOI, this method does not provide a direct residual moisture measurement, but, rather, a conservative moisture estimate. However, the TGA instrument also records the weight change as a function of temperature. Sorbed volatile species on the material, such as H_2O and CO_2, are known to desorb over specific temperature ranges, and one can attribute the weight loss within a given temperature range to the thermal desorption of physically adsorbed species on the sample surfaces. In the case of adsorbed H_2O (moisture), a reasonable estimate can be obtained from the weight loss from ambient temperature to 200°C due to the loss of molecularly adsorbed water [15]. Additional moisture from the associative recombination of hydroxyls would be expected to desorb over the temperature range 200-500°C. Structural waters may desorb at still higher temperatures. Other species such as NO_2, CO_2, and SO_2 may also desorb in this temperature range. To distinguish between multiple gaseous species that desorb over a similar temperature range, the TGA may be coupled with a Fourier transform infrared (FTIR) detection system or a mass spectrometer (MS). These instruments are used to directly identify and measure the species being desorbed from the material, allowing for a direct measurement of moisture. Calibration curves for different volatile species may be constructed using compounds that contain known amounts of water. Thus, quantitative volatile analysis is possible using a coupled TGA-MS or TGA-FTIR system.

TGA measurements were performed on many of the MIS samples. Early in the program measurements were performed on a set of samples using a Rheometrics model PL-STA 2000 TGA/DSC, which provides thermal gravimetric analysis and differential scanning calorimetry (DSC) over a given temperature regime. Results were obtained in the range of 0 to 200°C and in the range of 0 to 1000°C [2]. These results are given in Table A3-3. For many samples, a five-year time lapse occurred between stabilization and the TGA measurement. Later, TGA measurements were performed using a Netzsch STA 409 PC Luxx interfaced to a Bruker Vector 22 FTIR and a Pfeiffer ThermoStar mass spectrometer (MS) [16]. The FTIR was used to analyze a small subset of the samples and the data are published in reference 10. The MS was performed on most of the samples to determine and quantify the specific gases desorbed as a function of temperature. For the MIS materials, the data for the most common volatile species H_2O, CO_2, SO_2 and NO_2 were quantified. Calibrations were performed only for H_2O, therefore the other gases are listed as semi quantitative. TGA-MS results are given in Table A3-4.

Figure 6 compares the results obtained from the different approaches and instruments over time, comprising the LOI measurements, the moisture estimated from the mass loss to 200°C, and the moisture measured directly from TGA-MS. The LOI results exceed the 0.5 wt% criteria set for materials packaged in 3013 containers[2]. As expected, the TGA mass losses to 200°C were significantly less than the LOI results, in most cases. TGA results were higher than the LOI results for several samples containing less than 0.2 wt% moisture. This discrepancy may result from the heterogeneity of the material sampled, or from differences in sample handling after the material was stabilized. For many of these samples, the LOI was taken shortly after stabilization while the TGA measurement was performed after years of exposure of the sample to air. (See Figure A3-1). In most cases, however, the moisture estimates from the TGA mass loss to 200°C match the TGA-MS measurement fairly well (Figures 6 and 7). The moisture estimates from the mass loss to 200°C tend to be slightly lower than the MS measurement because they do not account

[2] When the LOI for items destined for 3013 container exceeded the 0.5 wt% criteria, TGA measurements were completed to confirm the criteria was met, and the material was restabilized if necessary.

for the hydroxyls, which may be significant in some materials, particularly in the pure oxides (MISSTD-1, 5501407, 07161856, and PEOF1). For most of the materials studied, TGA mass loss to 200°C gave a reasonable moisture estimate.

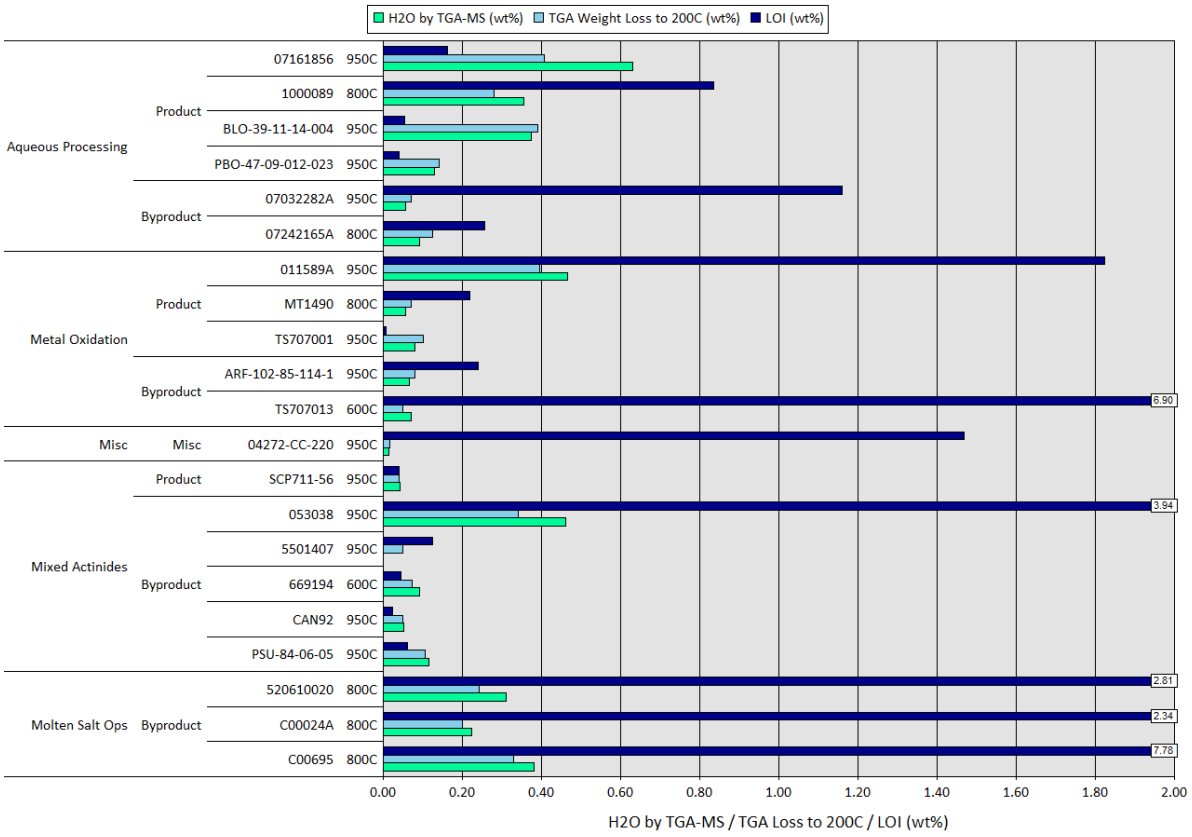

Figure 6. Comparison of the LOI total mass loss, the TGA mass loss up to 200°C and the MS moisture measurement (calcination conditions as specified).

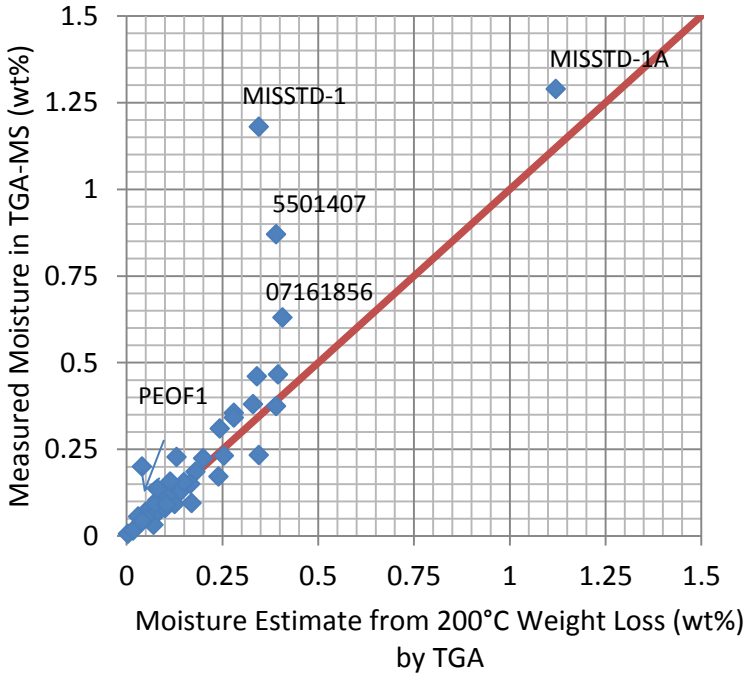

Figure 7. Comparison of MS moisture measurements and moisture estimates from the weight loss to 200°C. Note: Data for various calcination conditions.

Figure 8 shows the TGA-MS results for the most common volatile species detected in MIS samples. As described earlier, most molecularly absorbed water desorbs between 20 and 200°C, and more tightly held hydroxyls desorb between 200 and 500°C. MIS samples MISSTD-1 and 5501407 have the highest moisture by TGA-MS. The latter was a 50/50 mixture of "as-received" material and material calcined at 950°C. Nitrogen dioxide was observed in most of the MIS samples. This species is believed to originate from nitrates or from surface-sorbed nitrogen-oxygen compounds that are produced by the radiolysis of nitrogen and oxygen in the air. Desorption of NO_2 was observed between 200 and 800°C in the MIS materials. The NO_2 measurements were highest for MISSTD-1 and 07161856, both pure oxide samples that had significant exposure to air between calcination and the TGA measurement. Desorption of SO_2 was observed between 500 and 1000°C in the MIS materials. Two MIS samples (5501407 and 07242201A) showed significant SO_2 desorption, which is due to the sulfates present in the materials. The sulfur concentrations in these materials are approximately 2.0 and 0.8 wt% respectively. Carbon dioxide was observed in the MS results for most of the MIS samples. This species is produced from carbon or carbon compounds in the material, or by the surface adsorption of ambient CO_2. Desorption of CO_2 was observed between 200 and 800°C in the MIS materials. The CO_2 measurements were highest for ARF-102-85-355 and MISSTD-1. These two samples have greater than 0.1 wt% carbon, but CO_2 was not observed in other samples with higher carbon concentrations.

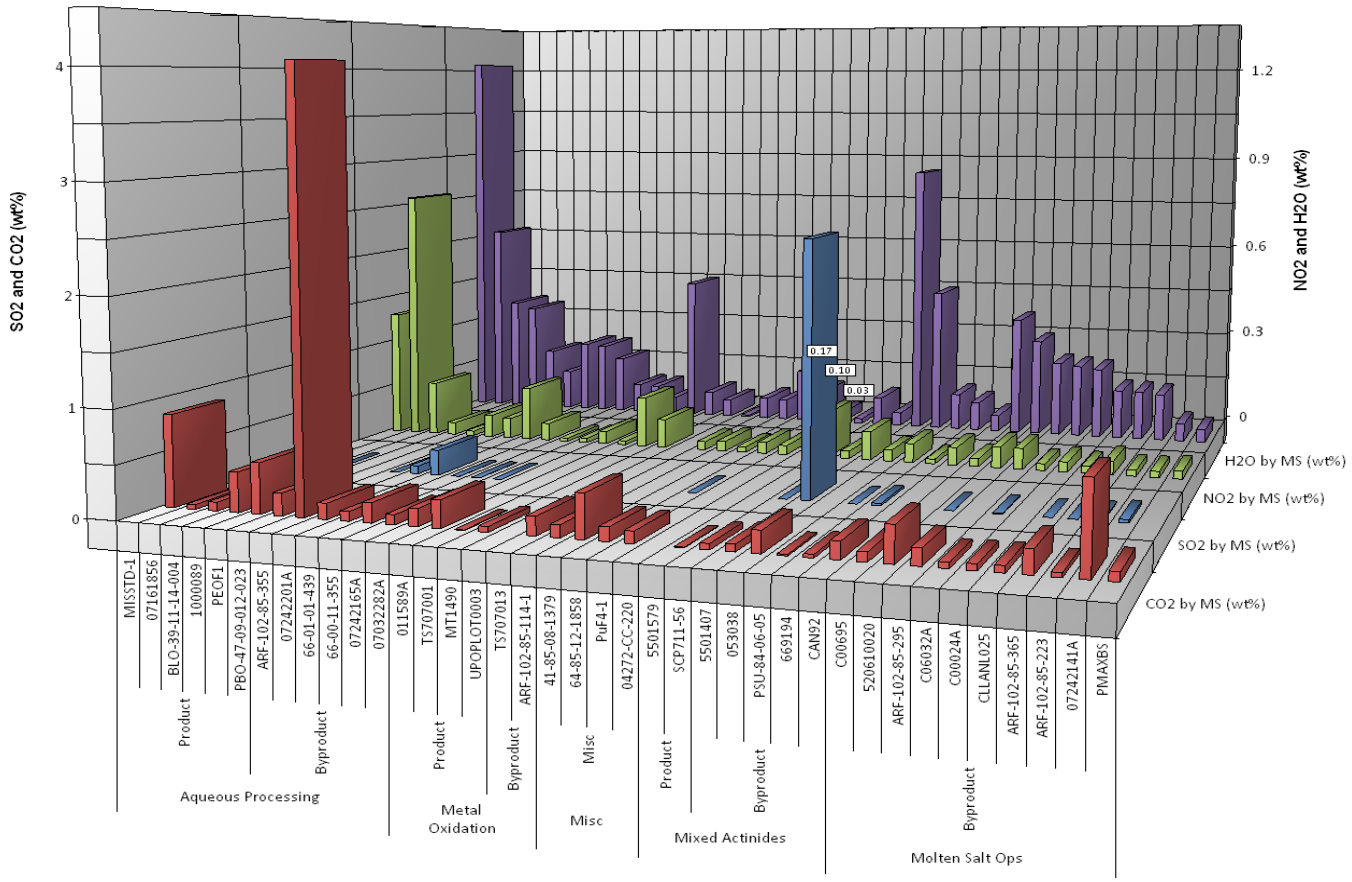

Figure 8. TGA-MS results for the volatile species on MIS samples sorted by process group, subgroup and decreasing moisture.
Note: Missing bars indicate that quantitative data is not available for a given species

Particle Size

Particle size distribution (PSD) measurements were made on several pure and impure MIS samples before and after stabilization. Measurements were made using the laser diffraction (LD) technique with a Horiba LA-920 particle analyzer. The technique obtains particle size measurements on >50,000 particles per analysis via the deconvolution of optical diffraction patterns produced by passing light through a suspension of the sample in a medium having a known refractive index. Typically, 6 to 8 individual analyses are taken and averaged to yield the data presented here. The technique requires a small amount (~100 mg) of riffled sample to be suspended in a flowing electrolyte solution (ISOTON 3). Samples were agitated in an on-board ultrasonic bath prior to analysis. These data replace the previously-published data that was found to have a nonsystematic bias for which corrections cannot be made [17, 18]. A subset of the representative materials based on the number of containers in storage was selected for re-measurement with the new LD system..

Appendix 4 provides the tabulated mean particle diameters measured for the MIS samples, and the plots of the particle size distributions. Averages by the process categories are also reported. As shown in Figure 9, and in Appendix 4, the PSD varied considerably. Various distributions including unimodal, bimodal and trimodal were also observed. The reasons for the variation between the PSDs are unknown, but the contributing factors to the multiple peaks may include the following:

- The oxide particles form agglomerates of various sizes [19].
- Oxide particles are produced by different chemical and physical mechanisms during, or following, oxidation (e.g., comminution versus spallation).
- The plutonium oxide particles are embedded in salts.
- The particles are not spherical and multiple dimensions of the particle are being observed. (The algorithm used by the particle size analyzer assumes spherical particles.)
- Mixtures of material with wide variations in processing history and impurities within a particular MIS sample.

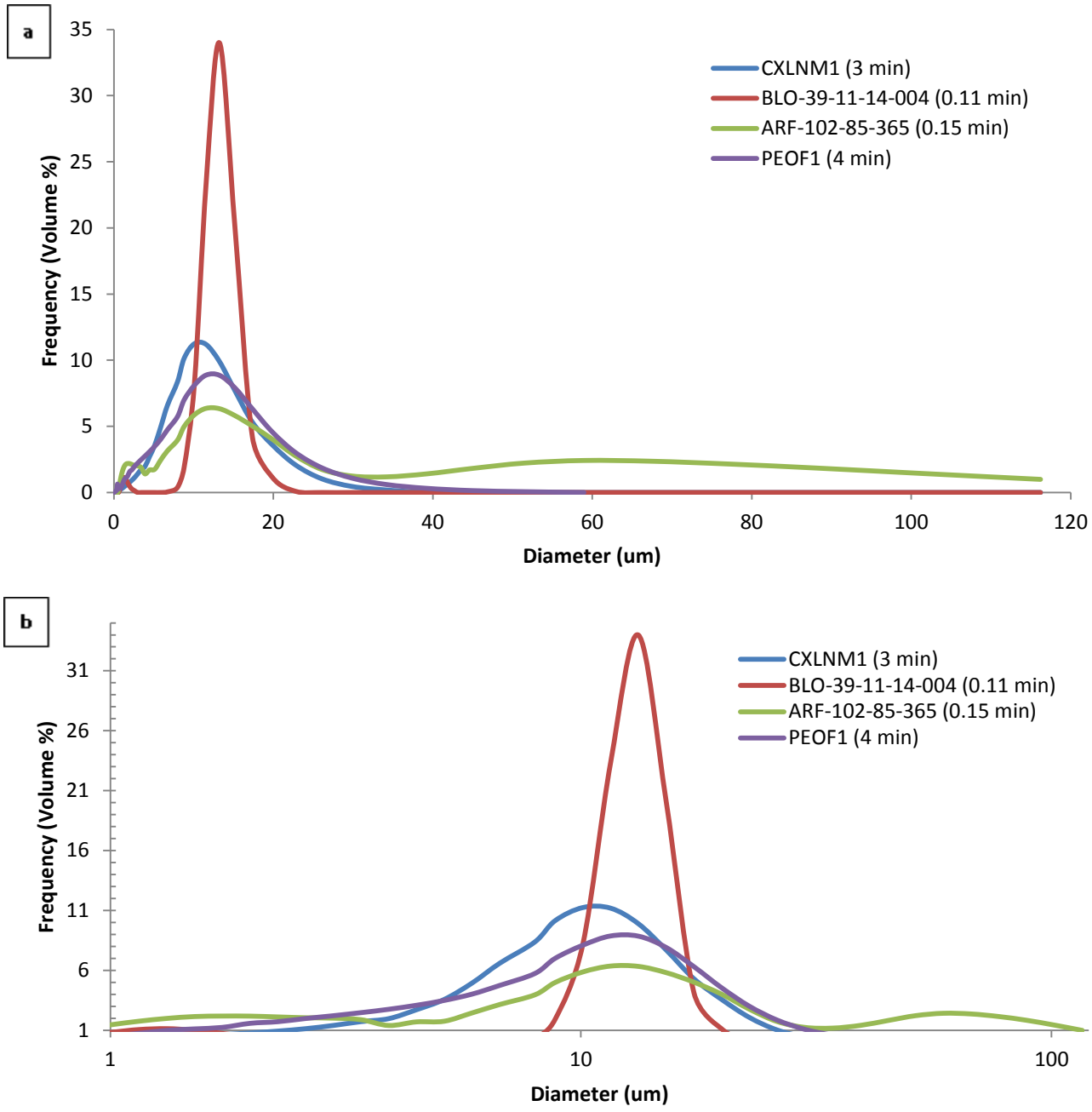

Figure 9. Particle size distributions of frequency (volume %) versus diameter (um) showing one (CXLNM1 and PEOF1) two (BLO-39-11-14-004) and three (ARF-102-85-365) modes. (a) Linear scale (b) Logarithmic Scale

The PSDs can be used to estimate the respirable fraction (RF) that could be released during accident scenarios [20]. One way of estimating an RF value is to use the volume percent less than 3 microns. Figure 10 compares this estimate of RF with the mean particle diameter for calcined MIS samples[3]. As shown,

[3]The respirable fraction (RF) is the mass fraction of airborne radionuclides as particles that can be transported through air and inhaled into the human respiratory system, and is commonly assumed to include particles with an aerodynamic equivalent diameter (AED) of 10 microns or less. The AED refers to the diameter of a sphere with a density of 1 g/cm^3 that has the same terminal settling velocity as that of the particle. The average PuO_2 particle density measured in this study ranged from 6-11 g/cm^{3}with one item less that 3.3 g/cm^3. Therefore, the 10 microns 1 g/cm^3 AED for PuO_2 can be expected to be significantly less than 10 microns and was approximated to be 3 microns.

the mean particle diameter ranged from 4 to 50 microns, and the volume percent under 3 microns ranged from 6 to 58 % of the volume of the sample. The largest volume percent under 3 microns was observed in MIS sample 66-00-11-355, a lean Pu item produced from magnesium hydroxide precipitate. This material contains approximately 31 wt% magnesium in the form of magnesium oxide.

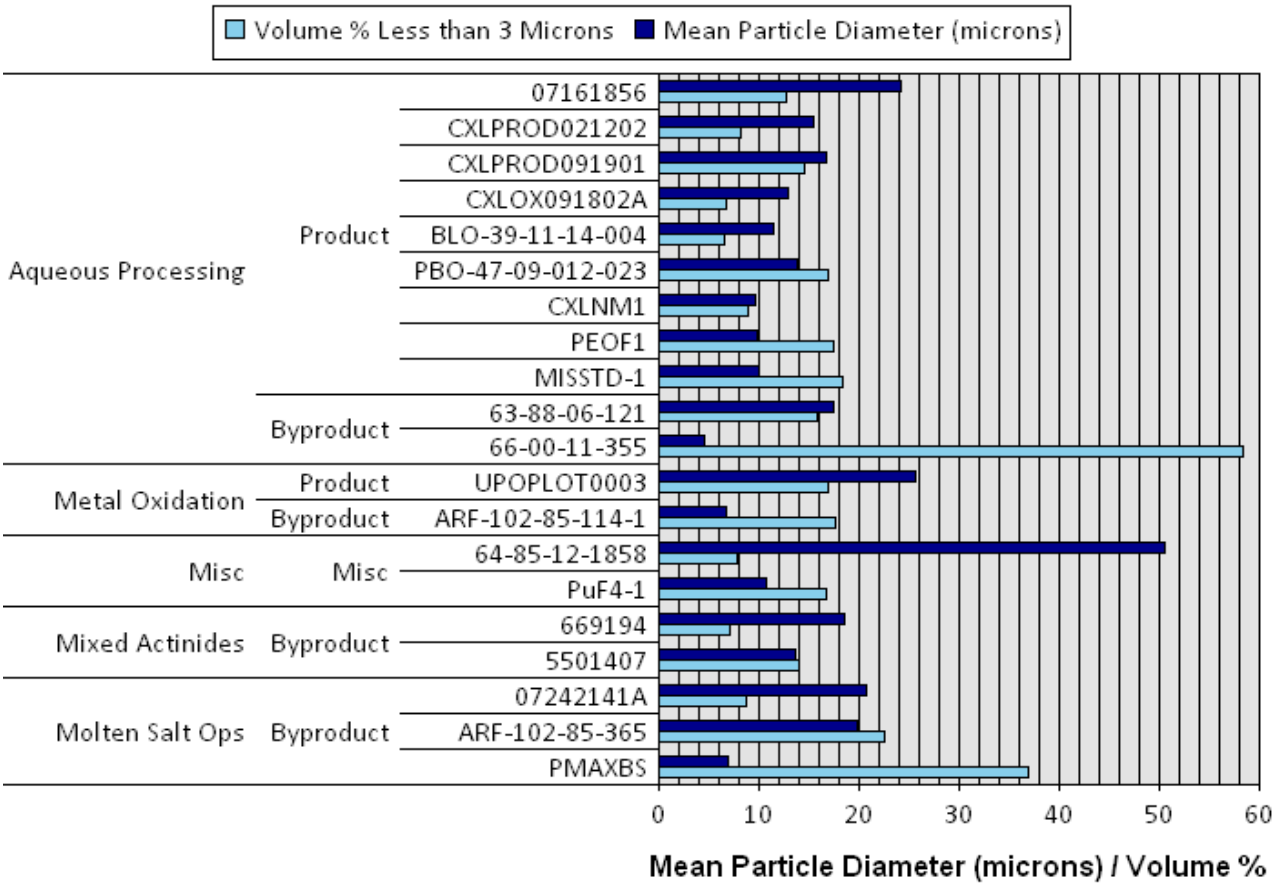

Figure 10. Average particle size after calcination at 950°C with minimum time suspended in solution.
Note: MIS samples MISSTD-1 and PMAXBS were not calcined at 950°C; therefore, the mean particle diameters are given for MISSTD-1 in the "as-received" and for PMAXBS in the 800°C condition.

When measuring particle size by the laser diffraction technique, care must be taken to select appropriate suspension and sonication times for the samples. The suspension time is the amount of time the powder is in contact with the electrolyte solution, and the sonication time is the amount of time the particles are agitated prior to analysis. These parameters can cause considerable variation in the PSDs obtained. For example, the mean particle size of PuO_2 particulates decreases systematically with longer suspension times (Figs. 11, 12, Table A4.2). In pure materials, this phenomenon is due both to the continual break-up of agglomerates, and to the tendency of larger particles to settle out more readily, during analysis. In impure materials, the decrease in mean particle size may also be due to the dissolution of soluble material (usually chloride salts). As a result, short suspension times (30 seconds or less) and a fixed analytical regimen were used to obtain representative particle size data. Data collected with minimal suspension and sonication time most closely represents the original dry sample.

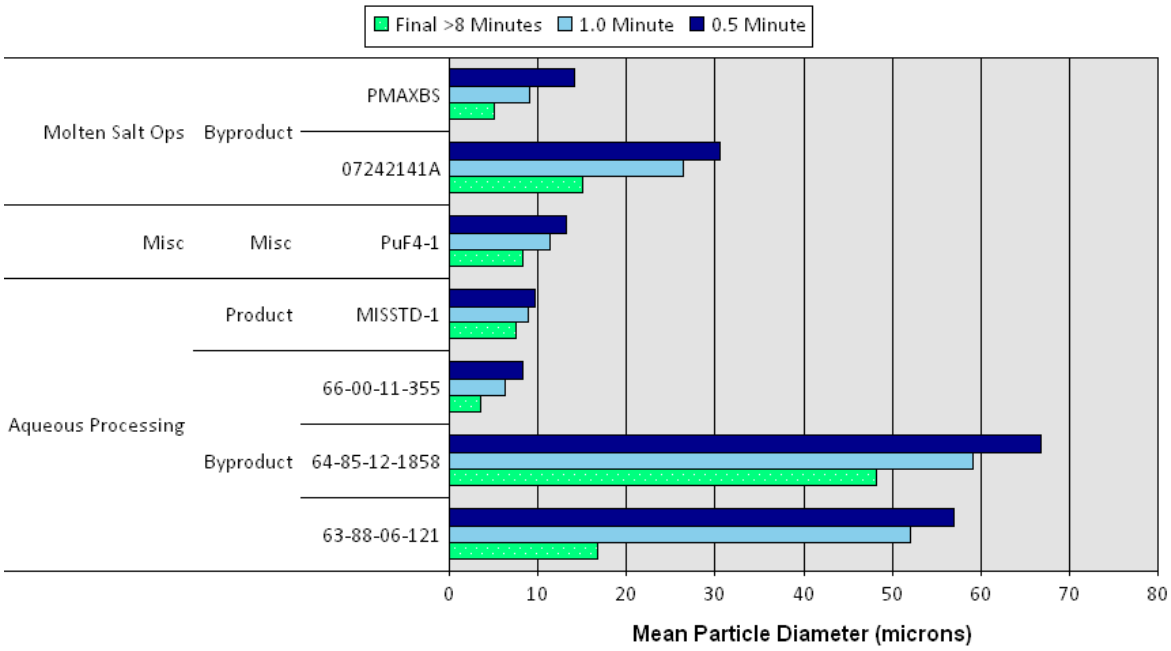

Figure 11. Mean particle diameter as a function of time the sample was suspended in solution.

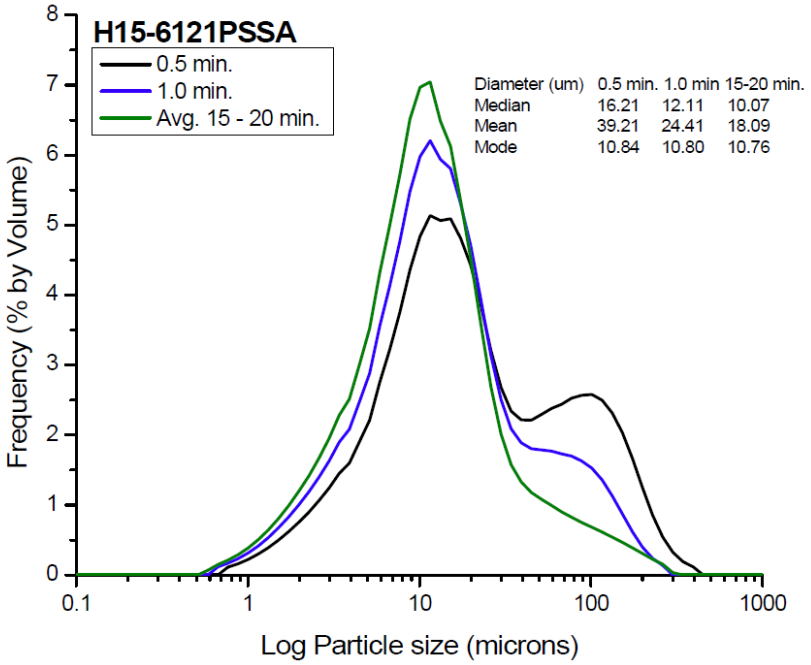

Figure 12. Particle size distribution (PSD) of MIS sample 63-88-06-121 as a function of increasing suspension time: 0.5, 1.0 and average of 15 to 20 minutes.

Particle size data obtained for eight product oxides is shown in Figure 13. Seven product oxides were generated from oxalate precipitation. Sample 7161856, which was generated from a peroxide precipitation, exhibited the most pronounced tri-modal distribution.

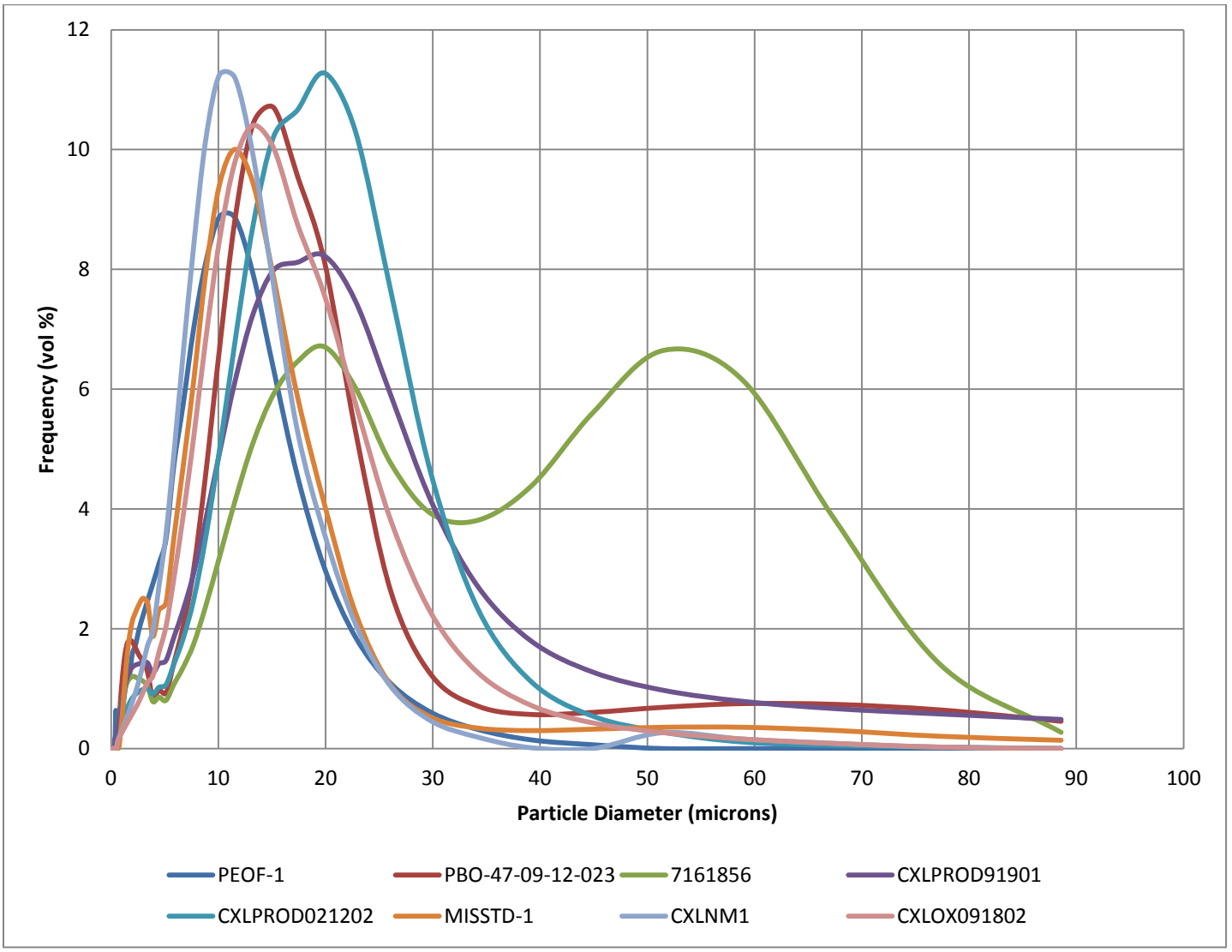

Figure 13 . Particle size distribution for MIS aqueous product oxide samples.

SEM images and Morphology

A series of SEM images were obtained for several of the MIS samples including PEOF1 (Figures 14-17), 053038 (Figures 18-20), 560210020 (Figure 21), C00024A (Figures 22-23), ARF-102-85-355 (Figures 24-25), and PPSL-365 (Figure 26). These images are not necessarily representative of the material as a whole because they illustrate a small portion of potentially heterogeneous samples.

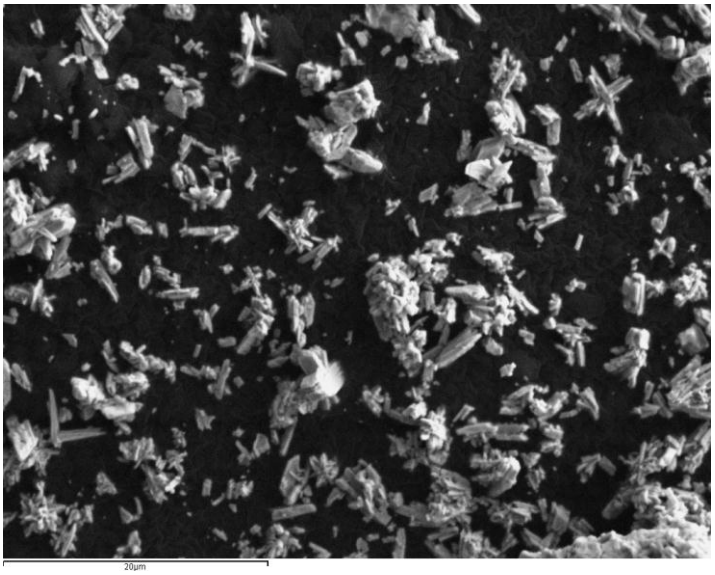

Figure 14. SEM image of MIS Sample PEOF1 used for analysis.

For MIS sample PEOF1, there was a bimodal distribution of particle sizes, consistent with the PSDs reported in Appendix 4. A morphological analysis was completed on several of the images to determine major axis length, mean diameter, and aspect ratio [21]. Several distinct kinds of particles were observed.

The majority of the particles in PEOF1 had an acicular morphology (Figure 15). These particles were almost always less than 1 µm in their short dimension, and often clustered with a 4-fold symmetry, suggesting a cubic or tetragonal crystal structure. For the smaller elongated particles, the average major axis length and mean diameter were 0.6 µm and 0.4 µm, respectively. The average aspect ratio was 1.8.

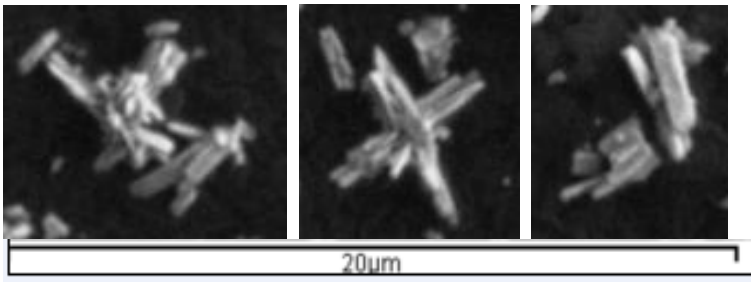

Figure 15. Small singular acicular particles that are often overlapped or conjoined.

Larger agglomerated particles, roughly equiaxed in overall shape, were also observed in PEOF1 (Figure 16). These were agglomerates of smaller flake-shaped particles, which had morphology distinct from the acicular particles shown in Figure 15. Finally, a smaller number of large equiaxed agglomerates of roughly hexagonal particles were observed (Figure 17). The 25 largest grains were selected for measurement, and the average mean diameter was 2.6 µm for this subset. When the 18 largest particles were selected, the average mean diameter was 11.5 µm; (With only 25 or 18 particles in the population, the statistics are quantitatively very uncertain). The mean value obtained from laser diffraction was 9.8 µm. (See Figure A4-17 and A4-18.)

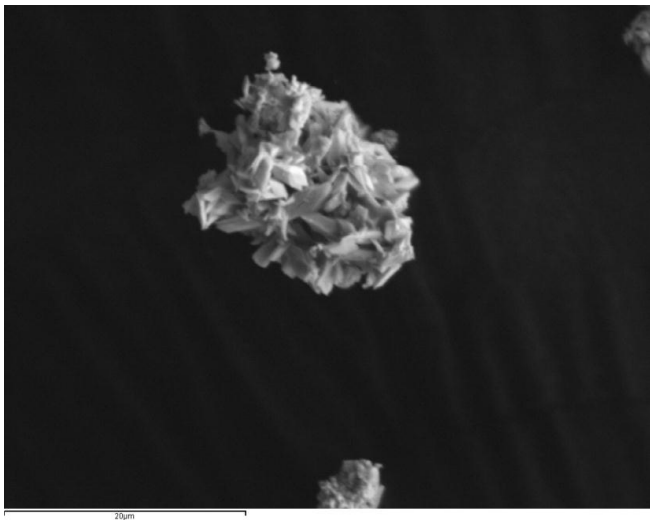

Figure 16. Large agglomerates of flake-shaped particles.

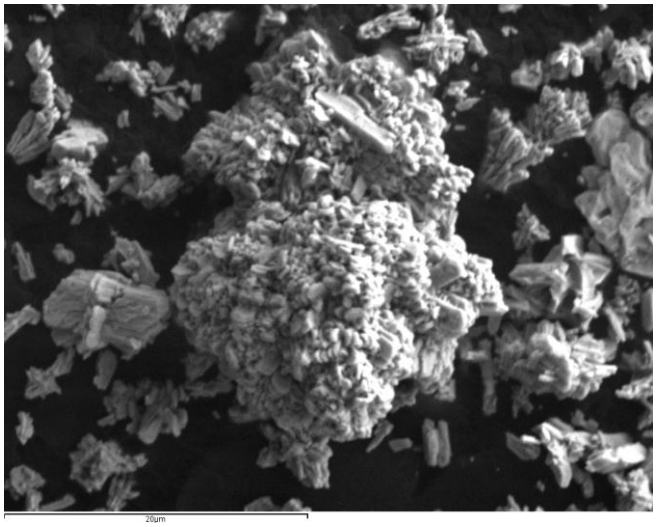

Figure 17. Typical large agglomerate of approximately hexagonal flakes.

SEM images were also obtained for five impure MIS samples to observe particle sizes, identify interesting features, and to determine the elemental composition of various features in the materials. Photos of these features are shown in Figures 18-26. The size and morphology of the observed particles varied widely in the materials, which made this method unsuitable for determining the overall particle size. Materials containing chloride salt impurities contained large agglomerates of salt mixed with oxide and other metal elements.

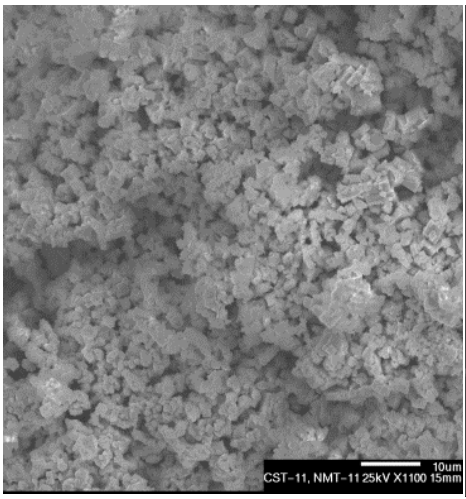

Figure 18. Pu oxide powder.

(MIS Sample 053038; AR condition)

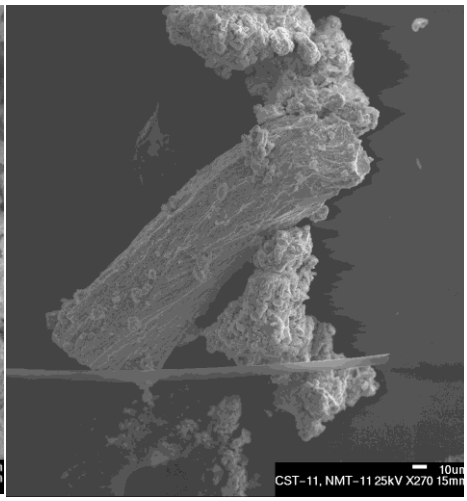

Figure 19 Pu oxide needle with KCl and NaCl.

(MIS Sample 053038; 800°C condition)

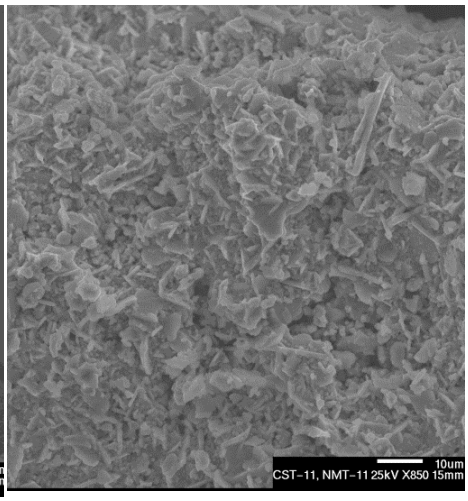

Figure 20. Agglomerate of U oxide and $CaCl_2$.

(MIS Sample 053038; 950°C condition)

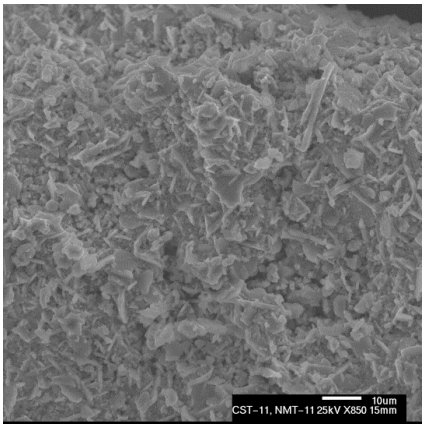

Figure 21. Pu oxide salt agglomerate containing (Mg/K/Cl).

(MIS Sample 5206110020; 950°C condition)

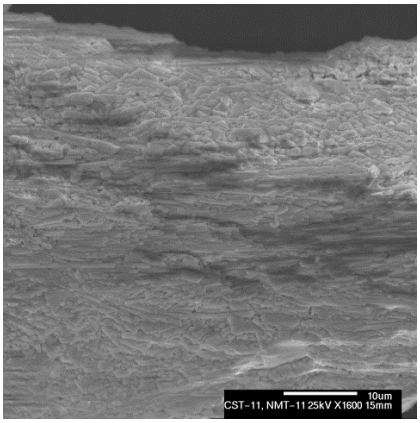

Figure 22. Large Pu oxide needle made up of smaller needles.

(MIS Sample C00024A; 950°C condition)

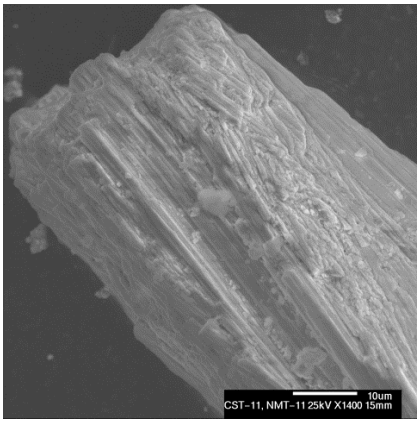

Figure 23. Bundle of Pu oxide needles.

(MIS Sample C00024A; 600°C condition)

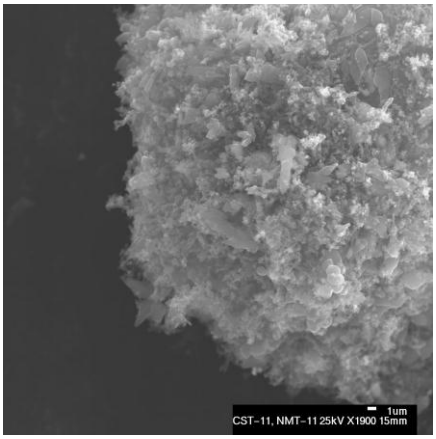

Figure 24. Agglomerate of thin Pu oxide needles and salt.

(MIS Sample ARF-102-85-355; AR°C condition)

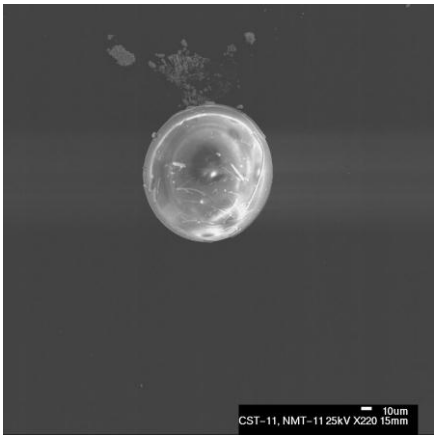

Figure 25. Al/Si bead.

(MIS Sample ARF-102-85-355; 950°C condition)

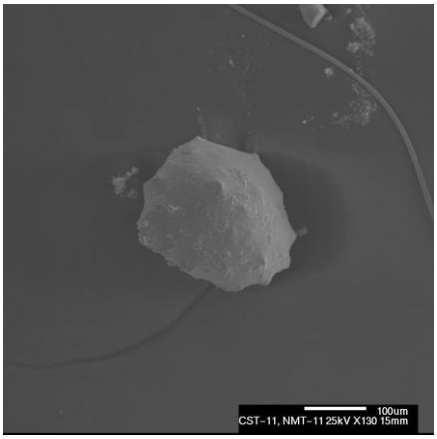

Figure 26. Large Pu oxide grain (>100 µm).

(MIS Sample PPSL-365; 950°C condition)

Surface Area

Surface area influences both chemical reactivity and moisture absorption behavior. Materials with a high surface area are a packaging concern because they have the greatest potential to absorb moisture and, hence, have greater moisture contents. Calcination at 950°C has been shown to reduce the specific surface area (SSA), thereby decreasing the potential for moisture absorption. The decrease in SSA with increasing temperature is due, in part, to the annealing of small fissures within larger particles. Specific surface area measurements were obtained on the MIS samples before and after high temperature stabilization. These results are tabulated in Appendix 5. The measurements were obtained using either the Quantachrome NOVA 3000 instrument, or the Horiba SA-9603-MP instrument, both of which use a gas sorption (BET) technique. Gas sorption is widely recognized as the preferred method because surface area is proportional to surface roughness, and gas sorption allows full contact with the entire surface. Samples are first heated, typically to 150°C to 200°C, under a vacuum or in a stream of dry UHP N2/He gas to ensure that the surfaces are free of water and other contaminants. The temperature is then lowered using liquid nitrogen (LN_2), and mixed He / N_2 gas is injected into the sample holder and allowed to equilibrate and adsorb onto the surface. The presence of He in the gas mixture allows a single layer of

nitrogen molecules to be adsorbed over the entire surface. The temperature is then raised, and the amount of N_2 gas released is measured. The N_2 peak area is proportional to the surface area of the sample.

Figure 27 shows the mean SSA for each process category and sub category in the "as-received" condition and after calcination at 950°C. MIS samples originating in aqueous processing exhibited the largest surface area both before and after calcination at 950°C. MIS sample MISSTD-1, a product quality oxide from oxalate precipitation had SSA results ranging from 19 to 33 m^2/g (the most recent measurement was 21.5 m^2/g). Other high SSA samples include ARF-102-85-355, which was high in carbon, and 39-01153A, a very low purity oxide from magnesium hydroxide precipitation. These items also exhibited the highest percent weight loss in LOI measurements. In both cases, the SSA was reduced to <10 m^2/g after calcination at 950°C. On average, surface area was reduced between 55% to 90% with calcination to 950°C. For example, MIS sample CXLNM1 is a product quality oxide produced by oxalate precipitation in the chloride line. The SSA measured for the "as-received" material, which was likely calcined at 600°C prior to receipt in the MIS program, was 25.6 m^2/g. The SSA was reduced to 2.7 m^2/g following calcination at 950°C. Surface area data is summarized in Appendix 5.

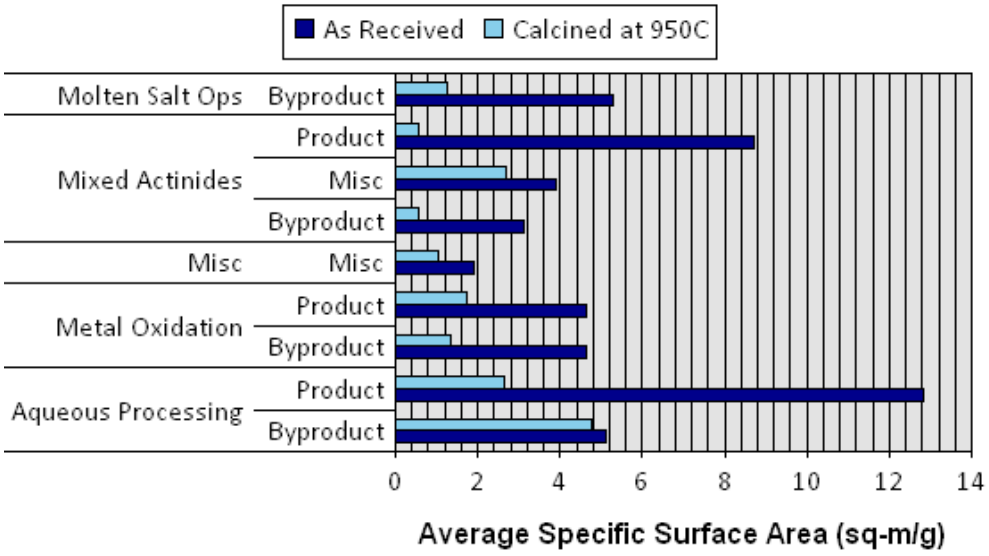

Figure 27. Average surface area measurements for materials before (AR) and after calcination (950°C) arranged by process categories.
Note 1: MIS sample MISSTD-1 (Aqueous Processing, Product) has surface area measurements ranging from 18.7 to 32.8 m^2/g in the AR material.
Note 2: Surface area measurements greater than 30 were not included in the averages shown in the plot.

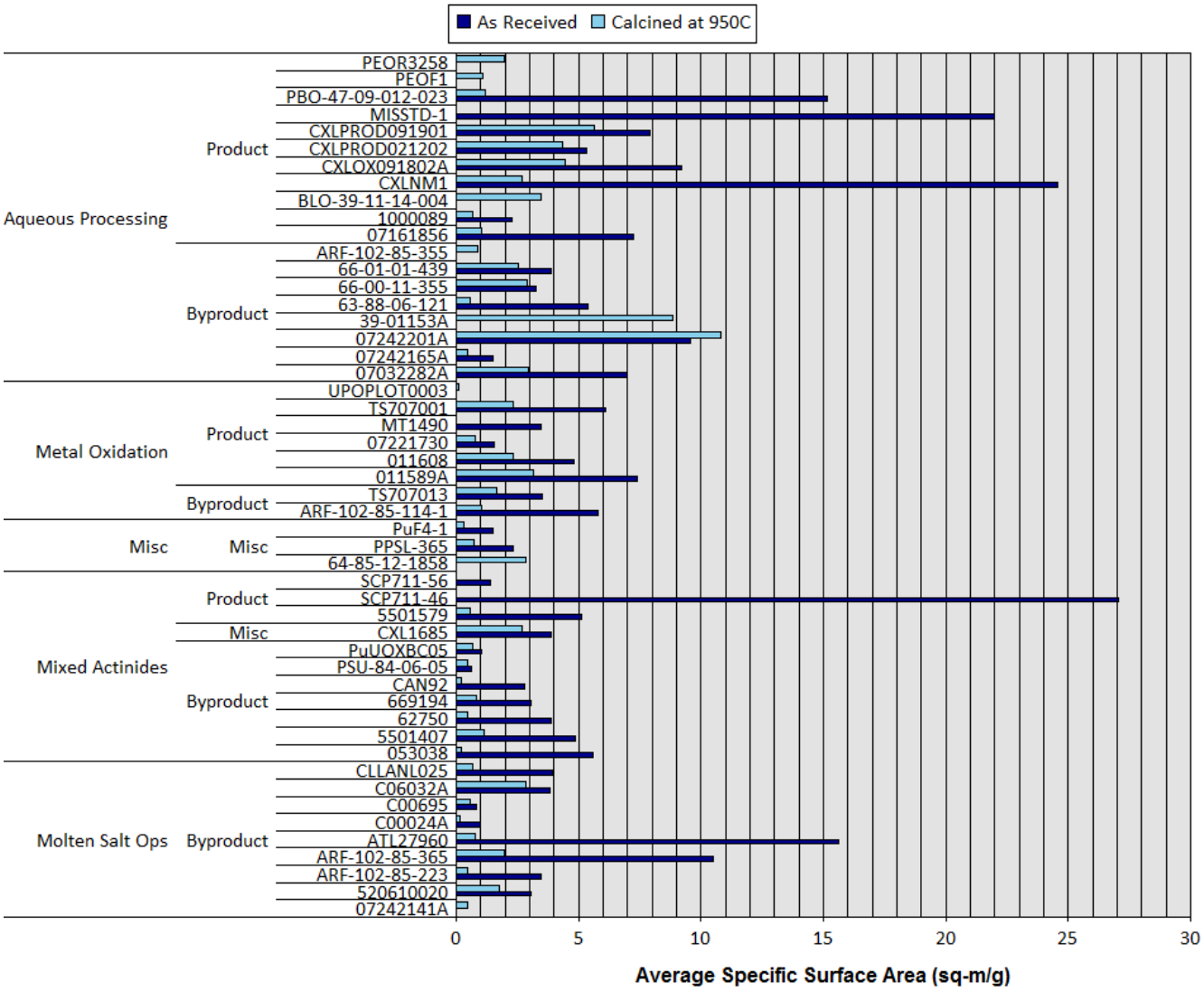

Figure 28. Surface area measurements for materials before (AR) and after calcination (950°C) arranged by process categories and MIS sample. (See notes for Figure 27.)

Density

Bulk, tap, and particle (or true) density measurements were obtained for the MIS samples before (AR) and after calcination (950°C). Bulk, tap and particle data, as well as percent compaction and packing fraction data, are presented in Appendix 6. The average densities for the process categories and subcategories are shown in Figure 29. Bulk densities before and after calcination are shown in Figure 30.

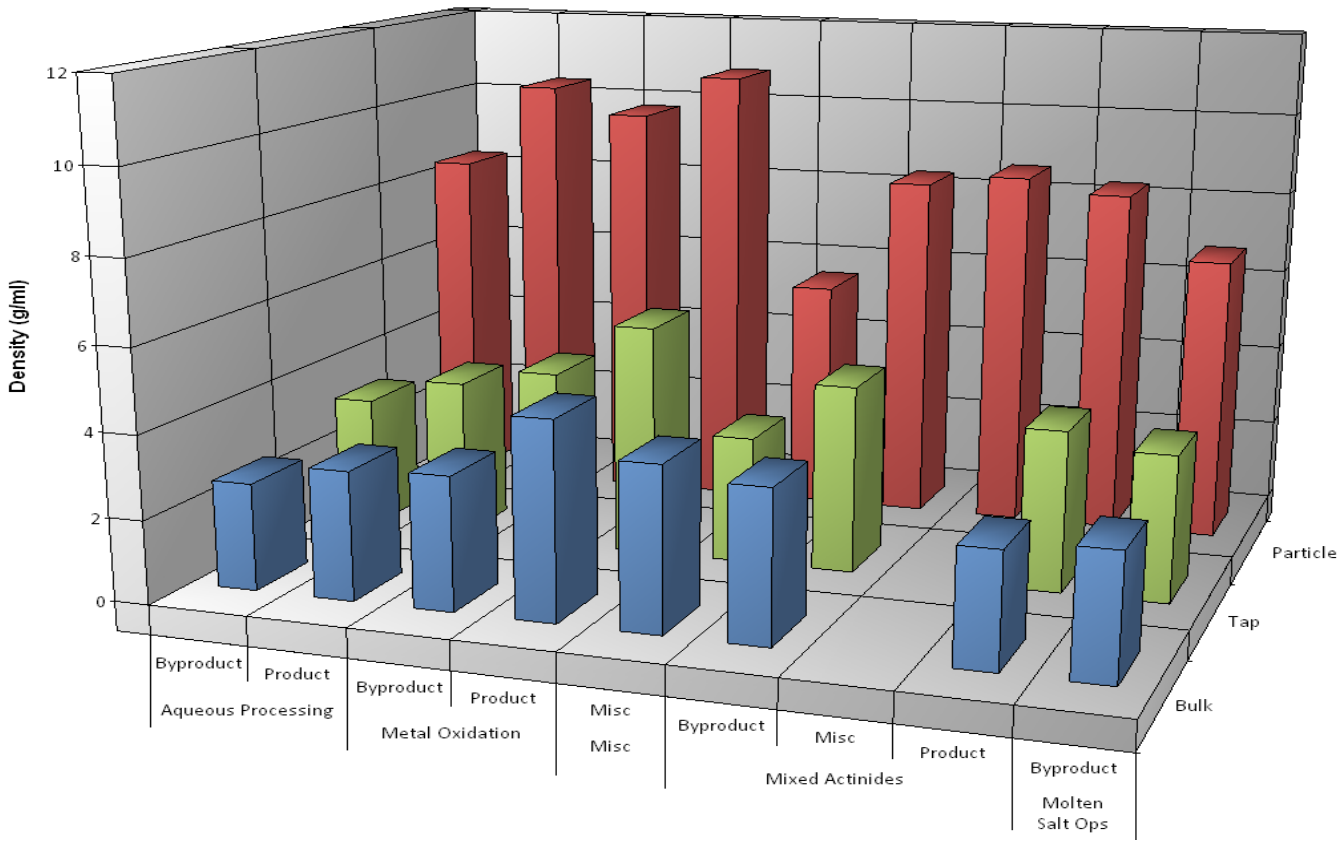

Figure 29. Average density measurements for various materials calcined at 950°C.

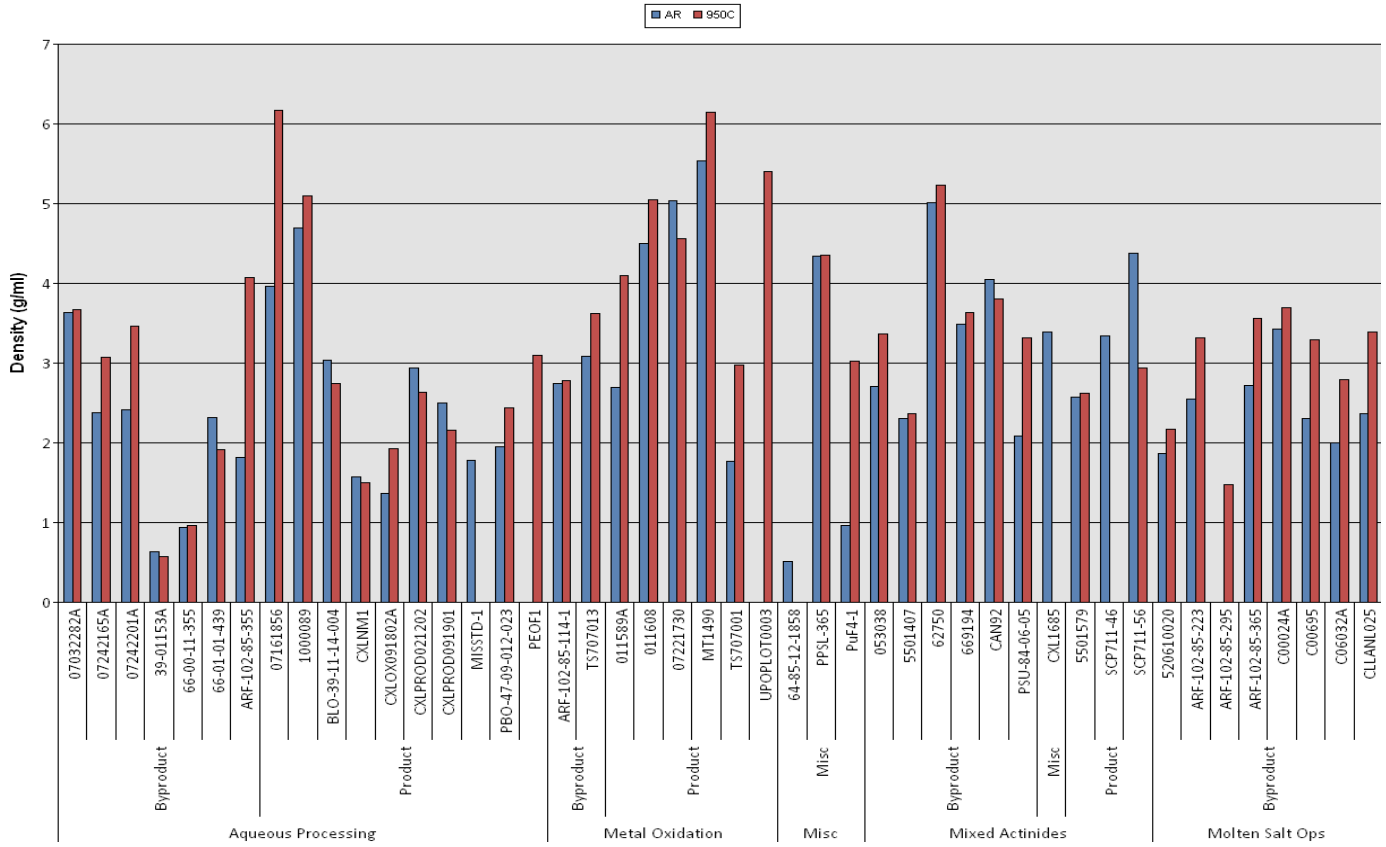

Figure 30. Bulk density measurements before and after calcination.

Each technique provides different information about the material. Bulk density measurements provide the volume occupied by a given mass of material. These measurements are useful for estimating the appropriate number of containers necessary for packaging material. Bulk density measurements are made by pouring material into a pre-weighed graduated cylinder. The volume of the material and the weight of the full cylinder are then recorded. Bulk density for the calcined representative samples ranged from 0.6 g/ml for the low purity aqueous byproduct sample 39-01153A to 6.2 g/ml for product oxide 0761856 and MT1490.

Tap density measurements show how the material can be compacted. Measurements are made by placing the pre-weighed graduated cylinder on an Autotap instrument and tapping the cylinder in an up and down motion for a predetermined number of "taps". The volume of material after completion of this process is then used to determine the tap density. Tap densities ranged from 0.7 g/ml for 39-01153A to 7.5 g/ml for MT1490. Percent compaction (1 – Bulk density/Tap Density) for individual items varied from 8% to 37% with averages ranging from 15% for metal oxidation products to 28% for mixed actinide products.

The pycnometer density gives the density of the individual crystals that comprise the bulk material. Pycnometer density is determined using helium displacement pycnometry using a Quantachrome Stereopyc instrument, or a Micromeritics AccuPyc 1300, together with a standard laboratory analytical balance. Particle densities ranged from 3.3 g/ml to 12.0 g/ml. Pycnometer density is used to calculate the volume occupied by the contained material (the ratio of the mass to the pycnometer density) and the free gas volume, the volume occupied by the gas [1]. For a given mass of material, as pycnometer density increases, free gas volume increases. Free gas volume is an important parameter used to calculate the pressure inside a packaged 3013 container. Pycnometer density is also used to calculate packing fraction which is the ratio of the bulk density to the pycnometer density and the void space, 1 minus the packing fraction. For the MIS samples, the average packing fractions for calcined material ranged from 0.3 to 0.4. MIS sample 39-01153A (less than 8% Pu) exhibited the lowest packing fraction, 0.05. The aqueous product sample 07161856 exhibited the maximum individual value of 0.62. As shown in Figures 31 and 32, the packing fraction increases as a function of bulk density. The line that passes through the data points is a statistical regression that can be used to estimate pycnometer density when bulk density is known. For all representative samples the regression is

$$F_p = 0.0843\ \text{l/kg}(\ d_{bulk}) + 0.1011$$

where *Fp* is the packing fraction and d_{bulk} is the bulk density. A better regression is obtained when only high purity product oxide (percent plutonium greater than 80%) data is considered[4]. The regression for product oxide is

$$F_p = 0.1005\ \text{l/kg}(\ d_{bulk}) + 0.0288.$$

[4]The product oxide packing fraction vs. bulk density plot includes data from the following MIS samples: PBO-47-09-012-023, 07161856, 1000089, BLO-39-11-14-004, CXLNM1, CXLPROD021202, CXLPROD091901, MT1490, TS707001, 011608, 07221730, and 5501579.

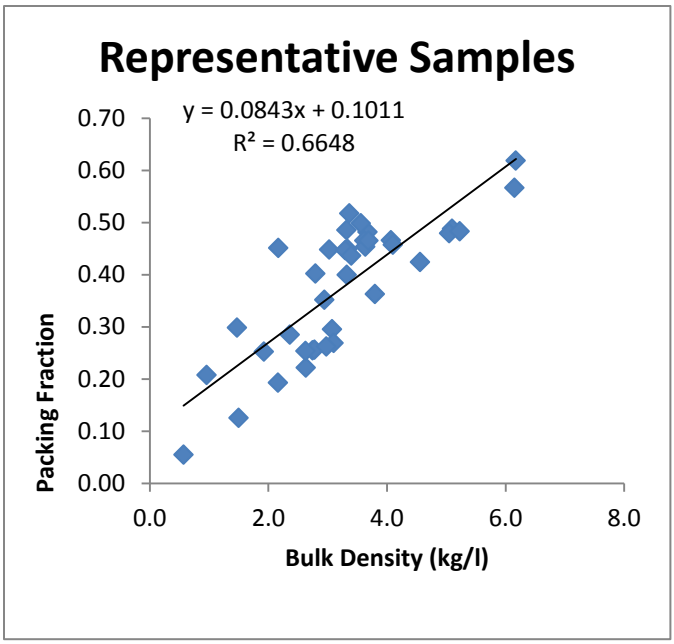

Figure 31. Packing fraction vs. bulk density for MIS samples calcined at 950°C.

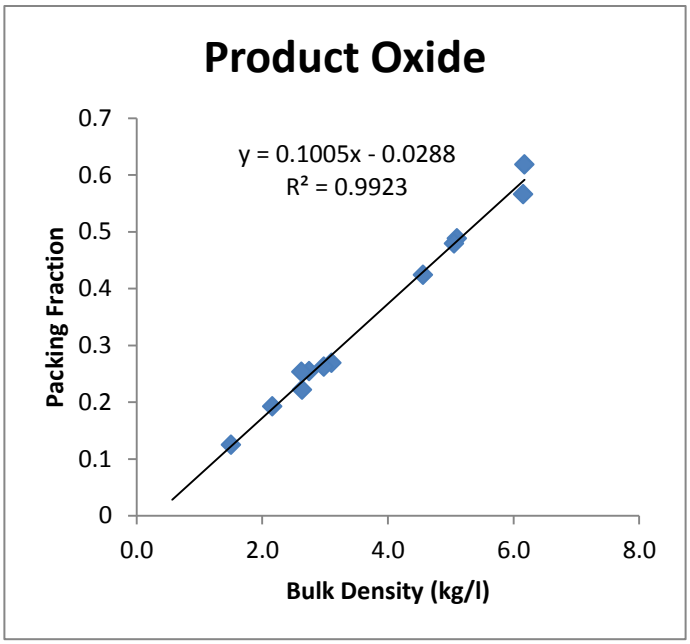

Figure 32. Packing fraction vs. bulk density for product oxide (with percent Pu greater than 80%) MIS samples.

Calorimetry and Actinide Composition

Calorimetry and gamma-ray spectrometry were used to determine the specific wattage, mass of plutonium and other actinides, and the plutonium isotopic composition for each MIS Item[5]. These parameters are used to determine purity of the plutonium (and actinide) and to show whether the plutonium is weapons grade, fuels grade, or reactor grade[6]. Gamma-ray spectra up to 1 MeV were obtained for each sample with a nominal 20% efficiency coaxial HPGe detector. The gamma ray spectra and the wattage were loaded into the FRAM Gamma Ray Isotopic Analysis software (v 4.2) that calculates the isotopic composition and mass of plutonium and other actinides. The other spectrum was obtained in the high-energy region (up to 5 MeV) of the gamma-ray spectrum for prompt gamma-ray analyses. The wattage and the mass of plutonium were determined by calorimetry for each item. The summarized data is given in Appendix 7, including a comparison of AR data with data collected after calcination to 950°C. A summary of the calorimetry and actinide composition results for the MIS samples is shown in Figure 33.

[5] Calorimetry and gamma-ray spectrometry measurements were required for each plutonium-bearing item upon receipt at LANL for materials accountability and safeguards purposes. Any differences in the amounts of nuclear material measured prior to and after the shipment to LANL were reconciled before any processing began.

[6] The DOE defines weapons grade plutonium as plutonium containing less than 7% Pu-240. Fuels grade contains 7-19% Pu-240, and reactor grade plutonium contains greater than 19% Pu-240.

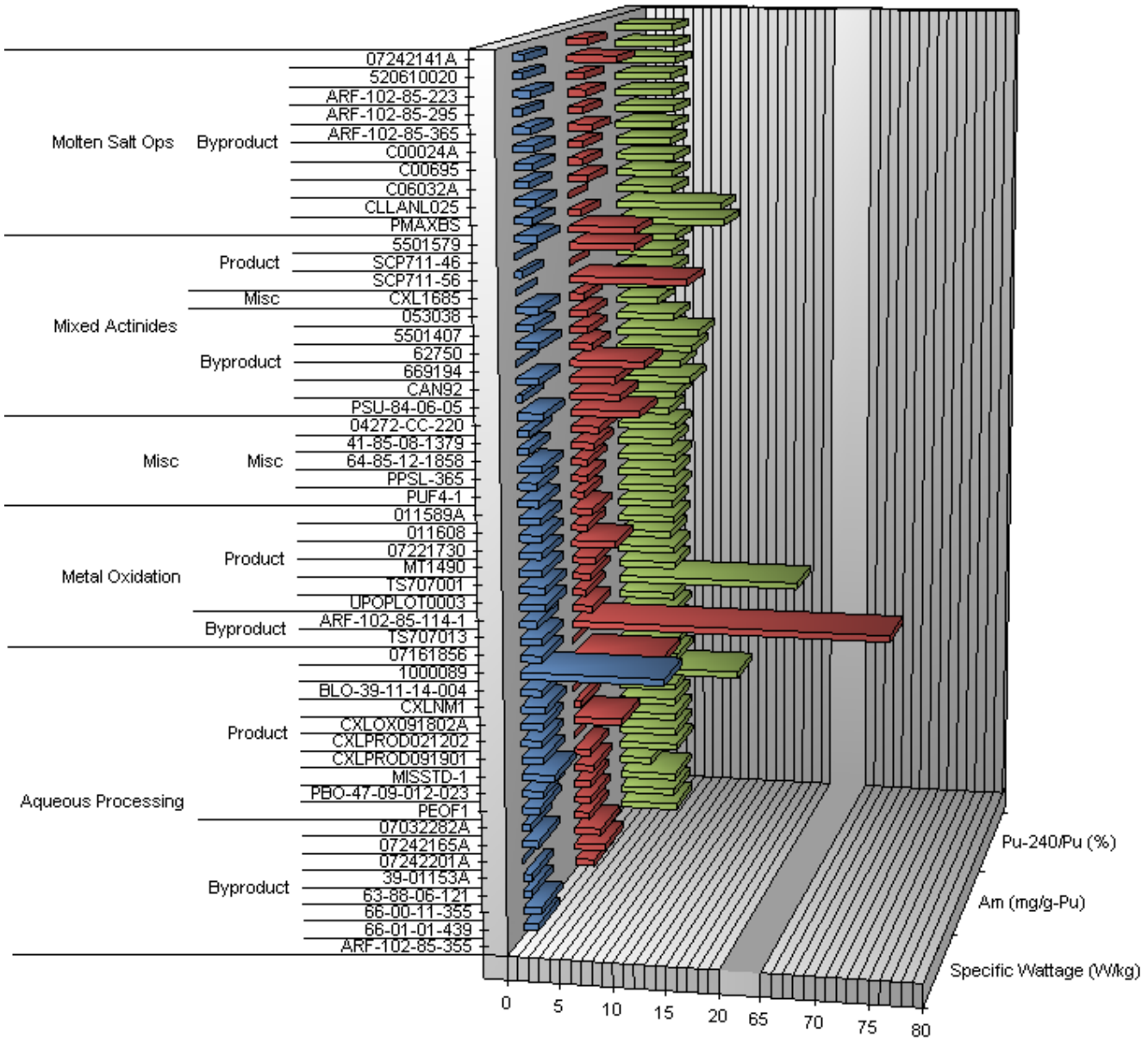

Figure 33. Average specific wattage (W/kg), americium concentration (mg/g-Pu), and Pu-240 isotopic composition (percent of total Pu) for various MIS samples in the AR condition.

Most of the MIS materials received from the sites includes weapon grade plutonium oxide with nominally 6.0% Pu-240. The MIS inventory includes one fuel grade oxide (PBO-47-09-012-023) with nominally 12% Pu-240 and one reactor grade oxide (BLO-39-11-14-004) with nominally 22% Pu-240 that were received from Hanford. The inventory also contains two fuel grade oxides samples from LANL (SCP711-56 and SCP711-46) with 10-11% Pu-240. The MIS inventory also contains mixed Pu/U oxides, Pu oxides with neptunium, and Pu oxides with thorium.

Trace Element Impurity Analyses

Prompt Gamma Analysis

Prompt gamma (PG) analysis is a nondestructive, nuclear, elemental analysis technique that uses charged particle reactions to activate and interrogate a sample. In this technique, certain light elements present in the plutonium oxide sample matrix are identified through the characteristic gamma rays that are emitted during or following nuclear activation with alpha-particles emitted from the plutonium. Using this technique, we can detect 12 light elements and obtain semi-quantitative estimates of concentration for eight of these elements. The elements sensitive to PG analysis are given in Table 2, along with the lower limit of detection for a 60-minute count ($LLD_{60\ min}$) in parts per million (ppm) [22].

Table 2. Prompt Gamma Detection Capability (LLD60 min=lower limit of detection for a 60-minute count)

Element	$LLD_{60\ min}$ (ppm)	Semiquantitative Determination
Li	300	No
Be	100	Yes
B	500	No
O	130,000	No
F	2,000	Yes
Na	200	Yes
Mg	600	Yes
Al	2,000	Yes
Si	Not available	No
P	8,000	Yes
Cl	6,400	Yes
K	20,000	Yes

In the PG analysis technique, gamma-ray spectra are obtained for the container with nominally 20% efficiency coaxial high purity germanium detectors in the range 0 to 5 MeV. The gamma-ray spectra are analyzed with the LANL-developed Prompt Gamma Analysis Software v. 4.7 that uses calibration curves to estimate the impurity concentration based on the peak areas corresponding with these elements normalized to the plutonium and americium activity. The calibration curves used in this method were developed using analytical chemistry and PG measurements on MIS materials. The calibration curves were then used to estimate the concentrations of the impurities in the packaged materials, most of which do not have analytical chemistry measurements. Certain impurities such as chloride salts are of concern for long-term storage, as they may increase the risk of container degradation over time, and many of these elements can be detected and their concentration can be determined on a semi-quantitative basis using PG analysis. The average values by process categories of some impurities measured by PG, for twenty four samples, are illustrated in Figure 34. Results for individual MIS samples are illustrated in Figure 35. The complete set of results is tabulated in Appendix 8.

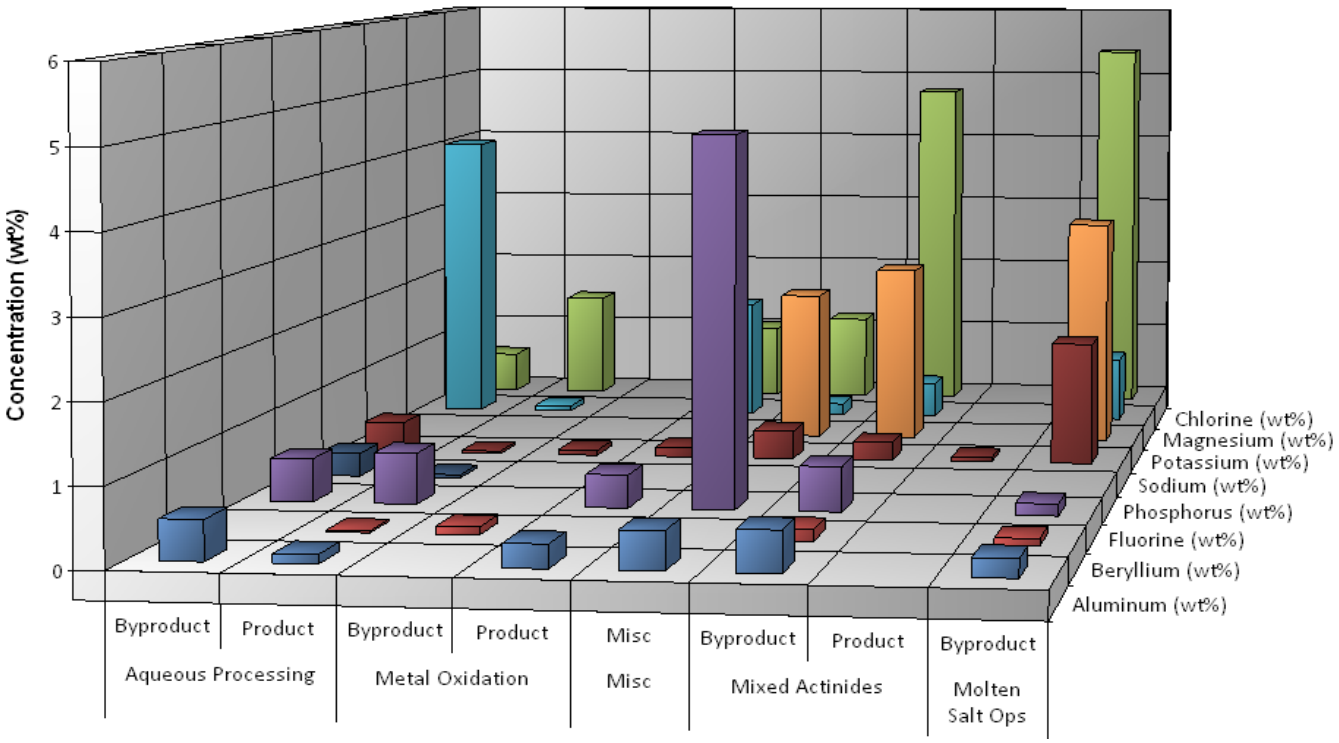

Figure 34. Average estimated impurity levels measured by prompt gamma spectrometry.

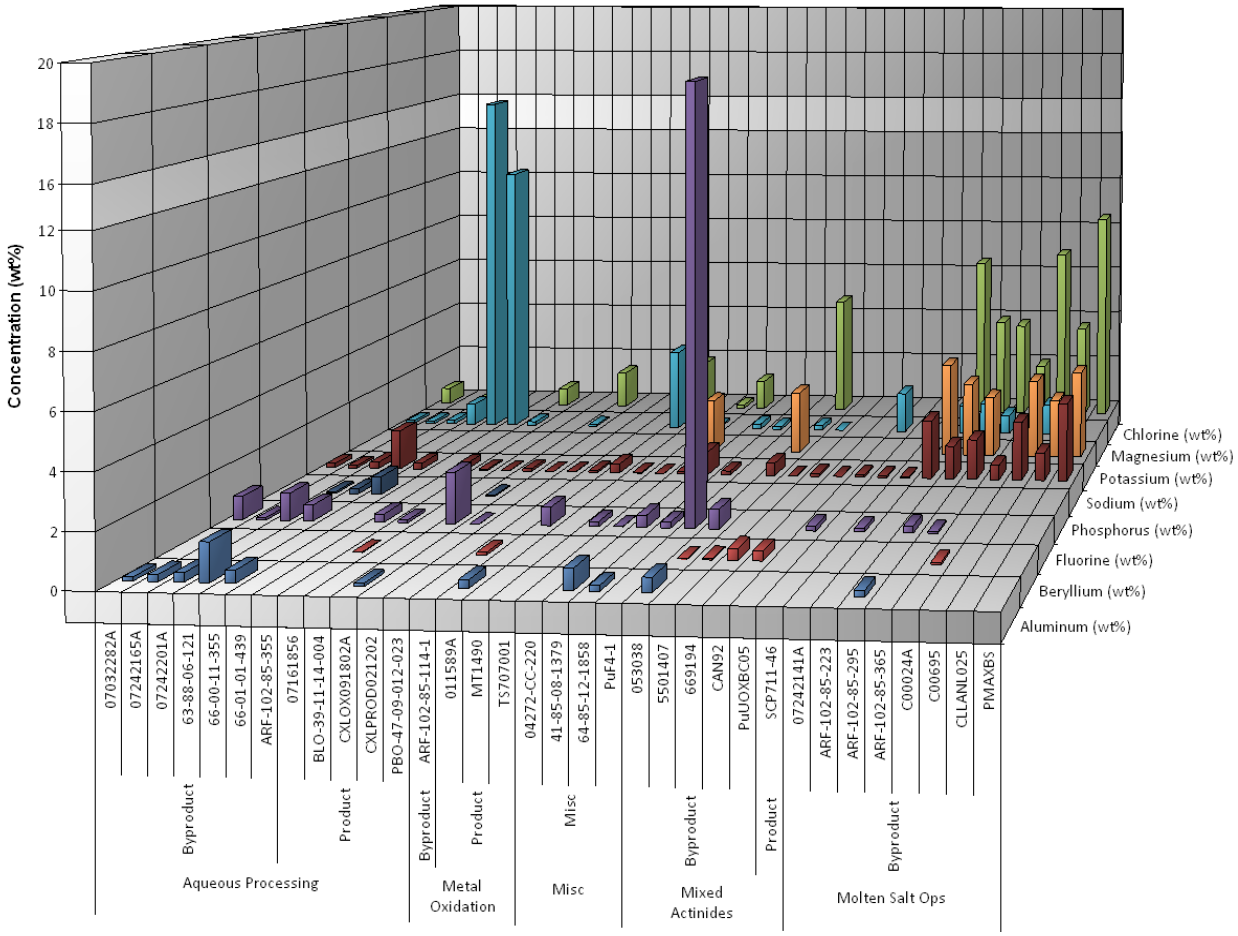

Figure 35. Estimated impurity levels measured by prompt gamma spectrometry.

Analytical Chemistry

The elemental impurities in the MIS samples concentrations were determined using a variety of analytical techniques including Inductively Coupled Plasmas-Atomic Emission Spectrometry (ICP-AES) for light element cations (up to the first row transition elements on the periodic table) and quadrupole ICP-Mass Spectrometry (ICP-MS) for heavier element cations. Ion chromatography was used for chloride and fluoride determination. Other wet chemistry techniques were used to quantify the carbon and nitrogen contents in some samples [2].

The average impurity concentrations by process category and subcategories after calcination at 950°C for select alkali, alkaline-earth metals and chlorine are shown in Figure 36. Impurities concentrations for the individual MIS samples are shown in Figure 37. The results for individual items are tabulated Appendix 9 for all conditions. Impurity concentrations for select nonmetal and metal elements in materials after calcination to 950°C are shown in Figures 38 and 39. The results for the MIS samples vary widely depending on the process in which the material originated. However, chloride salt impurities are a strong indicator that the material (or a portion thereof) originated in pyrochemical processing.

Many of the MIS samples are heterogeneous, consisting of large chunks, fine powders, and even mixtures of both. As a result it was necessary to divide several items in to portions containing the chunks and the powder, and these portions were sampled separately. Differences between these portions can be compared. The changes in impurity concentration with calcination, for samples with an initial impurity concentration of at least 0.3 wt%, are presented in Appendix 9, Table A9-4. The data show considerable variation in the magnitude and direction of the change for most categories and elements. In many cases, however, the effect of calcination may be masked by the chemical differences between samples of the same item due to the difficulty of obtaining representative samples of these heterogeneous materials.

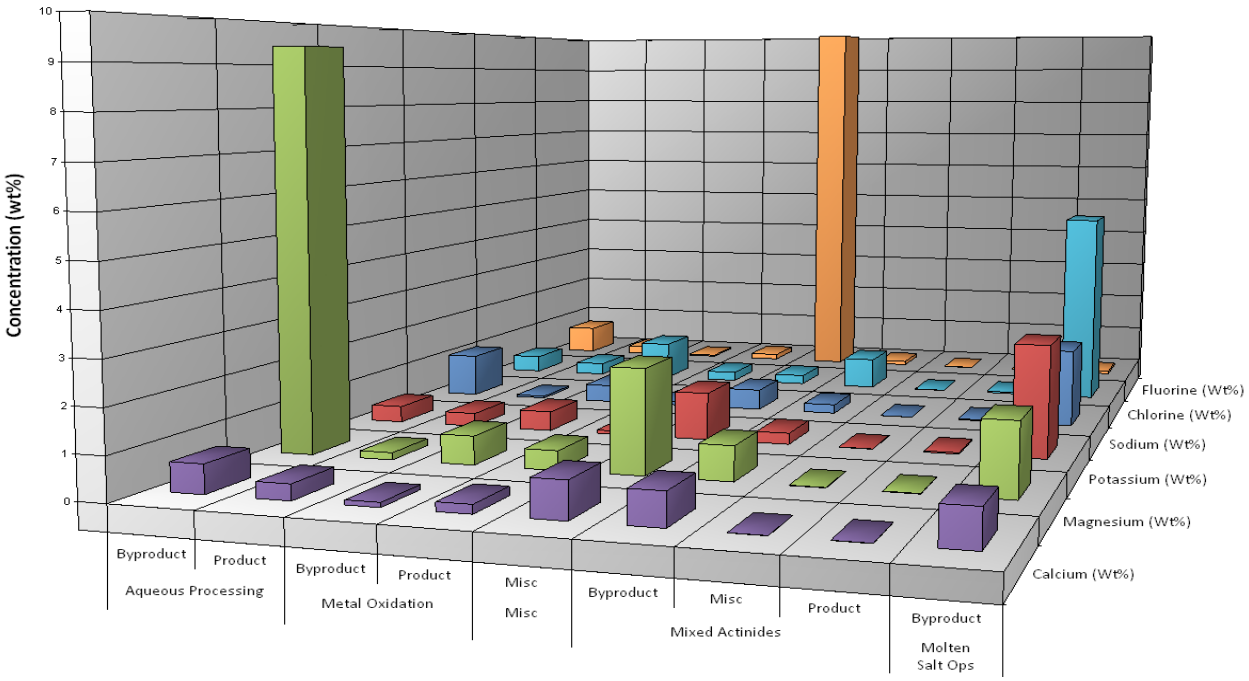

Figure 36. Average impurity concentrations of select alkali, alkaline-earth metals and chlorine impurities after calcination at 950°C.

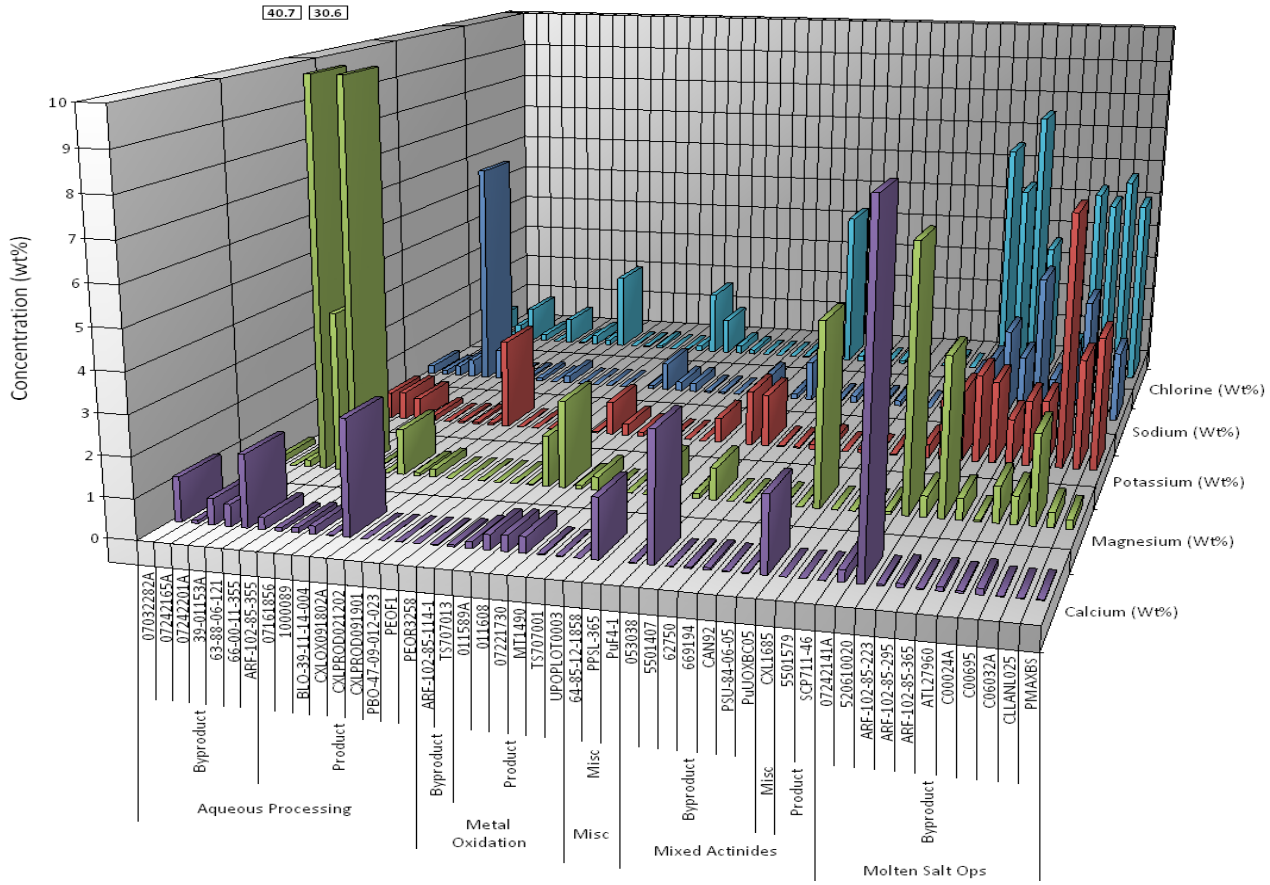

Figure 37. Concentrations of select alkali, alkaline-earth metals and chlorine impurities after calcination at 950°C.
Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

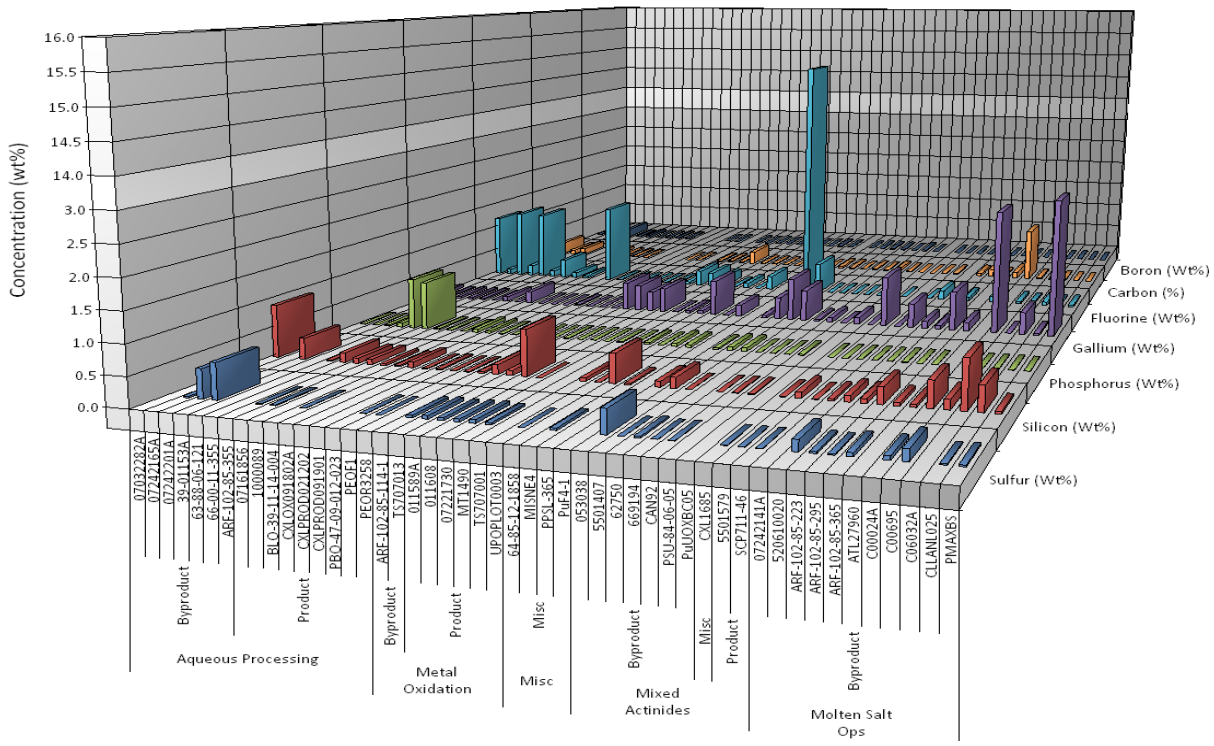

Figure 38. Concentrations of select nonmetal impurities after calcination at 950°C.
Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

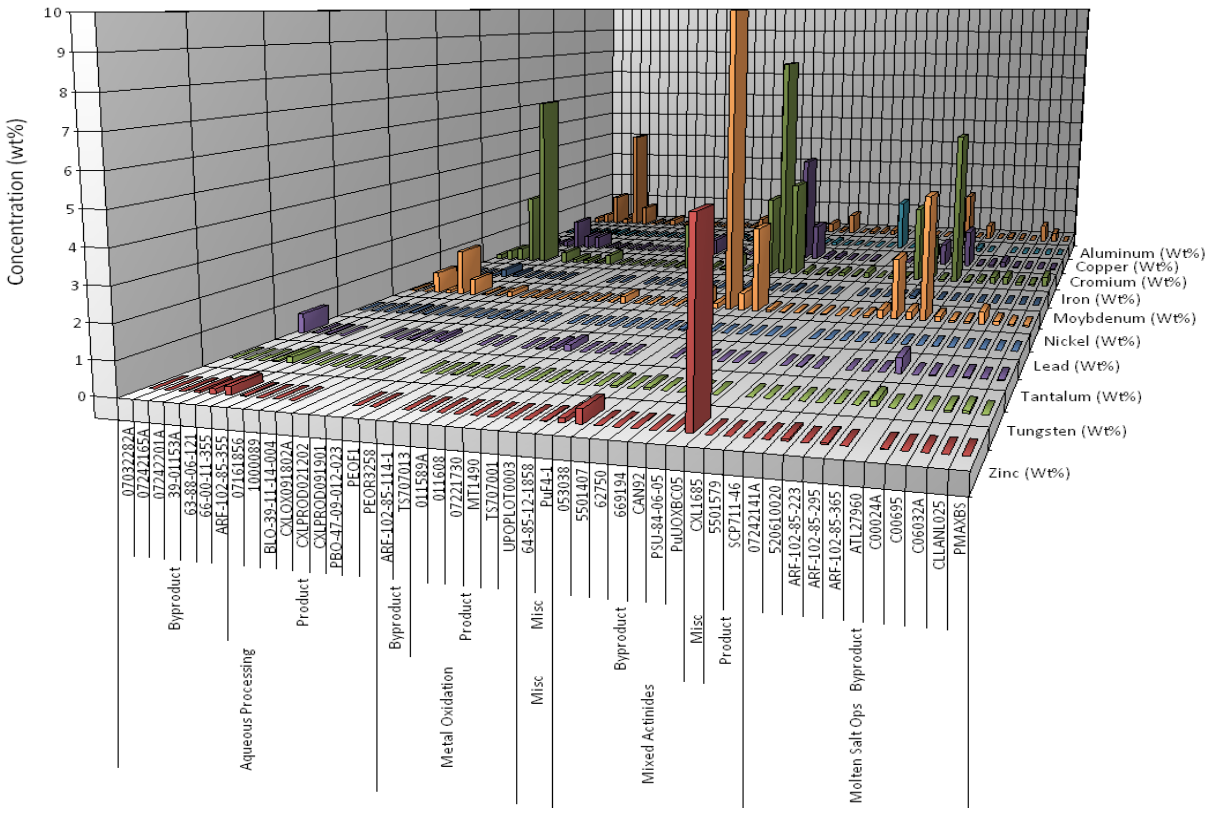

Figure 39. Concentrations of select metal impurities after calcination at 950°C.
Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

The chloride salt concentrations, as well as the moisture content, in these materials are of particular interest because they are associated with gas generation and corrosion of the storage containers. Some chloride salts, particularly magnesium and calcium chlorides, are hygroscopic and can absorb moisture from the glovebox atmosphere under packaging and storage conditions allowed by the 3013 standard [1]. During storage, gases, in particular hydrogen, are generated resulting in pressure buildup in the container. The gas generation behavior of the various materials has been studied extensively in the shelf-life studies, and it was found that it fits into one of three categories: high purity oxides (>85 percent Pu) with very low hydrogen generation rates; chloride containing materials (> 1% percent chloride), which have a much higher hydrogen gas generation rate; and other oxides, which generate hydrogen at rates in between these extremes. Generation of both hydrogen and oxygen occurs when hygroscopic alkaline earth chlorides), such as $MgCl_2$ and $CaCl_2$, are present under certain moisture conditions [23]. The chloride containing materials, particularly those with the hygroscopic chloride species, also have a higher risk for corrosion that other packaged materials. Under the allowed storage conditions, absorbed moisture on the hygroscopic chloride species can deliquesce (form liquid phases) in cooler regions of the container, typically at the container wall, creating conditions that favor pitting or stress corrosion cracking [24].

One way to estimate the amount of hygroscopic chloride species is to assume that the both sodium and potassium are present only as chlorides and the remaining “free chloride” is in the form of a hygroscopic chloride salt. The calculated “free chloride” concentrations for items calcined at 950°C and having greater than 0.25 wt% total chlorine are given in Appendix 10 and summarized in Figure 40. These results show a slight trend in that many of the hydrogen generating impure oxides have “free Cl”. However, calculated “free Cl” is small with respect to the total Cl, and the results from this method have a large uncertainty because the heterogeneity of the materials sampled and the uncertainty in the measurements. This was evident in that the calculated “free Cl” was negative. These values are presented here as zeros.

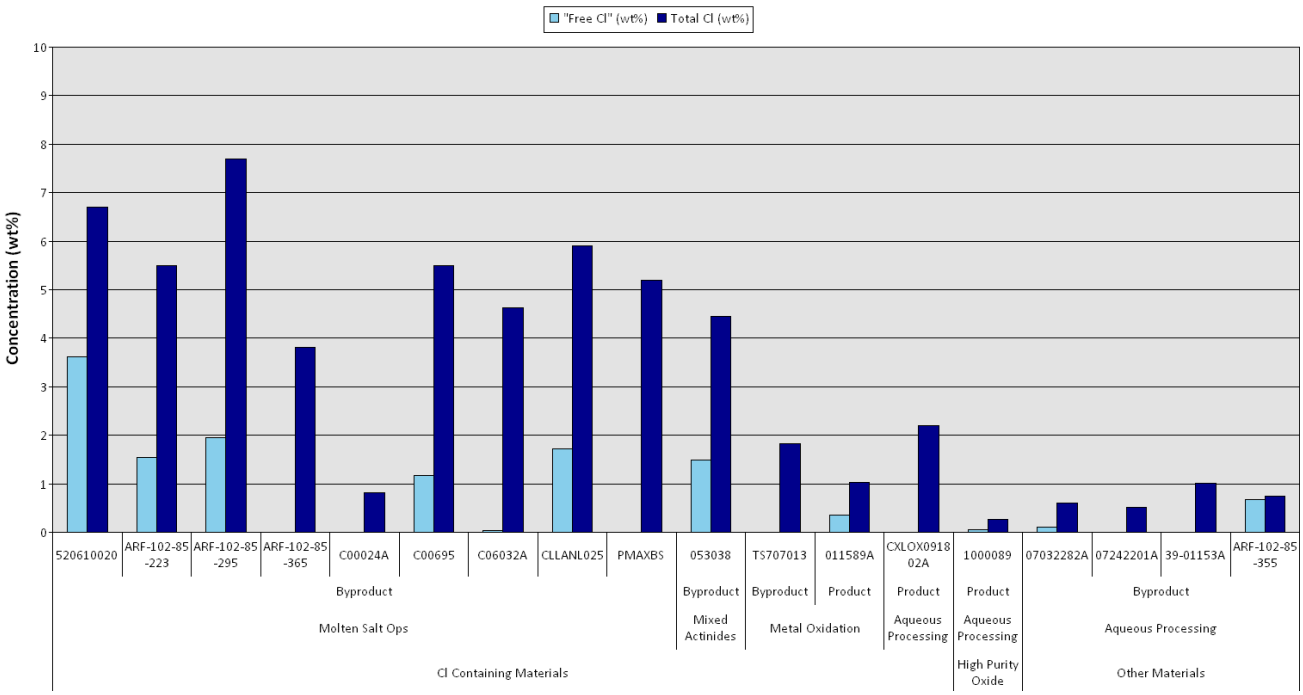

Figure 40. Calculated “free chloride” vs. total chloride in materials calcined at 950°C.
Note 1: Data include only MIS samples with chlorine concentrations greater than 0.25 wt%.
Note 2: MIS sample PMAXBS was calcined at 800°C.

The amount of hygroscopic chloride species can also be derived from measurements of the soluble species present in the materials. Assuming all the soluble cations, particularly magnesium and calcium, are present as chloride salts, one has a conservative method to estimate the hygroscopic chloride. This method is conservative because other soluble or slightly soluble magnesium and calcium compounds may be present in addition to the chloride forms. In addition, these results also indicate the amounts of soluble chloride species that survive calcination. This is important for understanding the packaged materials for which only estimates of total magnesium and total chloride are available from PG analysis.

The soluble species were measured by the ion selective electrode and ICP-AES techniques. Samples were placed in deionized water for 12 hours to allow the soluble components to dissolve into the filtrate. The solutions were analyzed with ICP-AES to determine soluble calcium and magnesium concentration. ICP-AES also was used to determine the chromium, iron, nickel, manganese and molybdenum in the filtrate, which are other impurities that could be present as chloride salts. Ion selective electrodes were used to determine chloride, sodium, potassium, and fluoride concentrations in the filtrate. These results are summarized in Appendix 10. These results also have uncertainties due to the heterogeneity of the materials and measurement error. Results confirm that on average all sodium, potassium, and chloride were present in soluble species. In contrast, roughly 50% of the calcium and 7% of the magnesium and fluoride were present in soluble species after calcination.

Figure 41 compares magnesium concentrations measured by ICP-AES on the filtrate with those obtained on a completely dissolved sample. The results show that the chloride containing materials that have a significant soluble magnesium component also generate hydrogen in shelf-life studies. [23]

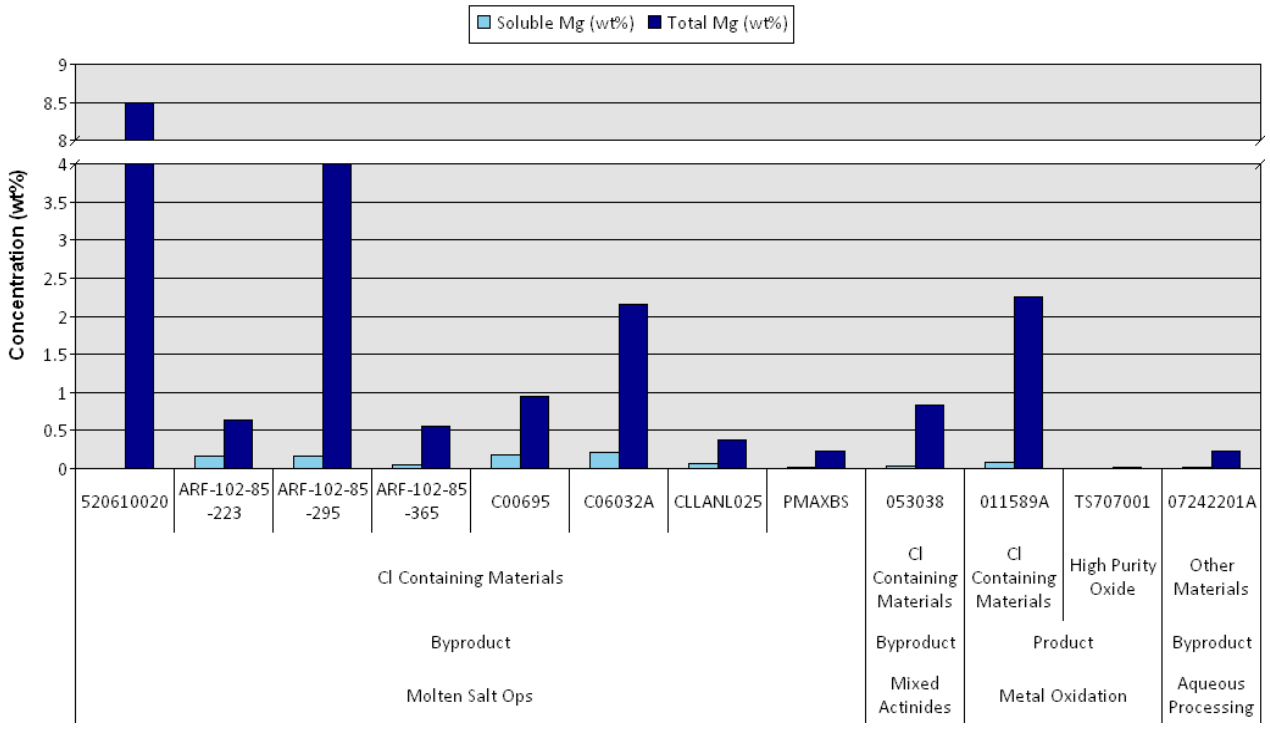

Figure 41. Comparison of soluble magnesium to total magnesium concentrations in various MIS samples.

Figure 42 compares the calcium concentrations measured by ICP-AES on the filtrate with those obtained on the completely dissolved sample. Calcium solubility ranged from ~30% to 70%. Four samples contained greater than 0.1% total calcium. These samples included three chloride containing materials, 011589A, 520610020, and 053038 and one other oxide, 07242201A. These three chloride containing materials exhibited significant hydrogen generation and 011589A and 053038 exhibited oxygen generation as well. [23].

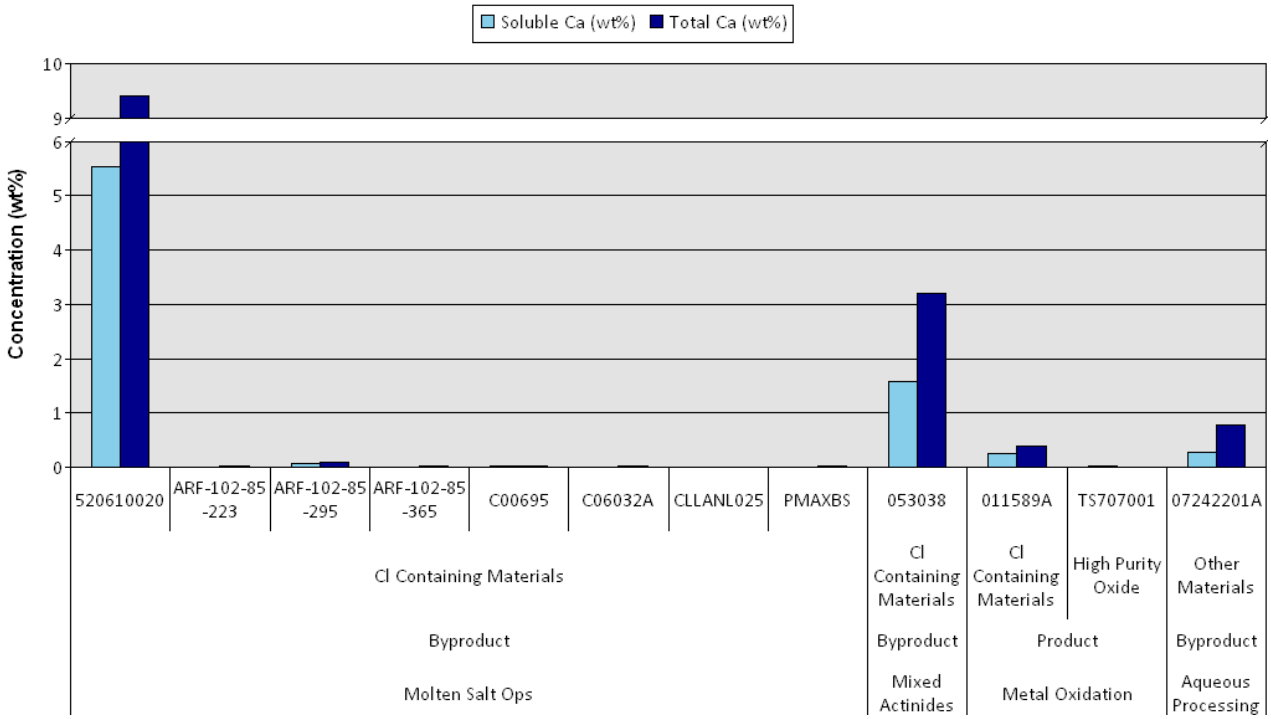

Figure 42. Comparison of soluble calcium to total calcium concentrations in various MIS samples.

Figure 43 compares the fluoride concentrations measured by the ion selective electrode on the filtrate with the fluoride concentrations measured by IC on the completely dissolved sample for several of the items. These results show that the fluorides in these materials are relatively insoluble, with the average solubility of 7% for those items containing at least 0.1% fluoride. Because they are relatively insoluble, fluorides are not expected to contribute to corrosion.[25]

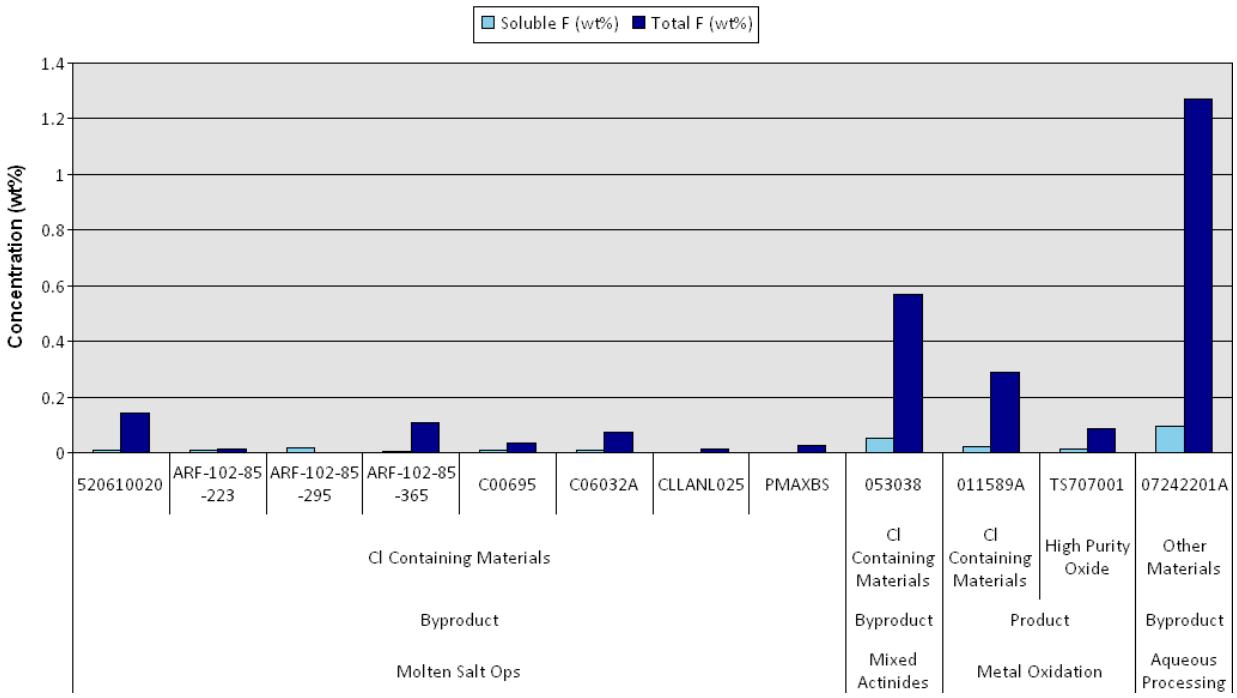

Figure 43. Comparison of soluble fluoride to total fluoride concentrations in various MIS samples.

Shelf-life Surveillance

The shelf-life program identifies early indications of potential degradation mechanisms that provide a focused process for selecting storage containers to examine in the field and determines those factors that are most important in terms of pressurization and corrosion [23]. The shelf-life program provides essential information to the storage sites for the surveillance activities that will be established. The shelf-life program uses both full-scale (i.e. 3013 size) containers and small-scale (1:500 scale) test containers. The large scale experiments were designed to establish the baseline behavior of full-scale containers and bound the behavior of material in extreme cases. The containers in large-scale experiments are instrumented with thermocouples, a pressure transducer, a Raman chamber, and a gas manifold used for extracting gas samples for gas chromatography (GC) analysis. Corrosion specimens are placed at various locations in the material. Some of the containers have double cantilever beam specimens that are monitored electronically for changes that indicate crack growth throughout the experiment. The small scale experiments were designed to establish the behavior of represented materials within the storage conditions allowed by DOE 3013 Standard. The representative materials are exposed to humid conditions that allow the materials to absorb 0.5 wt% moisture. The small-scale test containers are instrumented with pressure transducers and are placed in a heated array maintained at 55°C. Some of the test containers are also fitted with humidity sensors to monitor the relative humidity over time.

The results from the small-scale shelf-life experiments are described and the data for the experiments is available electronically through the LANL library (see Reference 15). A list of representative samples is given in the Appendix 11. As described earlier, the representative materials fit into one of 3 groups: high purity oxides, chloride containing materials, and other materials. In the high purity oxides, mostly nitrogen and carbon dioxide are generated, with minor amounts of hydrogen. One exception is MIS sample, MISSTD-1. This material has a high surface area, and it absorbs moisture at low RH (less than 10%) and generates hydrogen and oxygen from radiolysis of the absorbed moisture while in storage. In the chloride containing materials, hydrogen dominates gas generation, and carbon dioxide, nitrogen,

carbon monoxide, and methane are minor components. The other materials are generally impure oxides that do not contain chlorine, and much less hydrogen is observed.

Summary

This report provides an overview of the results of the comprehensive characterization studies that have been performed on the plutonium samples used to represent the metric ton quantities of similar materials that are currently stored in 3013 containers across the DOE complex. This information is a key element of the technical basis for understanding the behavior of plutonium in the presence of a wide variety of impurities over long periods of time. The broader significance of this work is that it provides the technical underpinnings for major programs with national and international implications that depend on the need to safely store various forms of plutonium, and it provides detailed information on a broad cross-section of plutonium materials that likely represents a bounding case for material contents in storage across the DOE complex.

Acknowledgements

The members of the Material Stabilization and Characterization Team over the history of the MIS program are gratefully acknowledged: Tom Allen, Beverly S. Bender, John Berg, Brian Bluhm, Debbie Dale, Janey Duncan, Lynn Foster, Obie Gillispie, Kirk Hollis, Elizabeth Kelly, Annabelle Lopez, Rebecca Maez, Carl Martinez, Luis Morales, Dennis Padilla, Larry Peppers, Jim Rubin, Dan Schwartz, Leonardo Trujillo, Kirk Veirs, and Edward L. Wilson.

Funding for this work was provided by the US Department of Energy. This work was conducted at Los Alamos National Laboratory operated by Los Alamos National Security, LLC under contract DE-AC52-06NA25396.

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Appendices

Appendix 1	MIS Samples and Process of Origin
Appendix 2	Weight Loss from Calcination
Appendix 3	LOI/TGA Data
Appendix 4	Particle Size Data
Appendix 5	Surface Area Data
Appendix 6	Density Data
Appendix 7	Calorimetry and Isotopic Data
Appendix 8	Prompt Gamma Analysis Data
Appendix 9	Trace Element Analysis Data
Appendix 10	Chloride Salt Mass Balance and Soluble Constituent Data
Appendix 11	Shelf-life Surveillance Representative Samples

Appendix 1, MIS Samples and Process of Origin

Table A1-1. Table of MIS Representative Samples Showing Process of Origin, Major Impurities, Characterization Comments, and Actinide after Stabilization to 950°C.

Process Category	Process Subcategory	MIS Sample	Source Site	Origination Process	Comments	Major Impurities (>1 wt%)	Surface Area / Density	Chem	Sample Available	Prompt Gamma	Small / Large Scale	% Pu	% U	% Pu+U
Aqueous Processing	Byproduct	07032282A	RFETS	Dissolution Residuals (from foundry scrap oxide)	Oxide Dissolution, Building 771, Room 114	F, Ca, C,	Y	Y	Y	Y	Y	67.9	0	67.9
		07242165A	RFETS	Dissolution Residuals (from foundry scrap oxide)	Residue Dissolution, Building 771, Room 149, Line 24	C	Y	Y	Y	Y	Y	34.1	0	34.1
		07242201A	RFETS	Dissolution Residuals (from foundry scrap oxide)	Residue Dissolution, Building 771, Room 149, Line 24.	Cl, F, C, Ni, K, S	Y	Y	Y	Y	Y	63.3	0	63.3
		39-01153A	RFETS	By-product Oxide from Hydroxide Precipitation	Caustic Waste Treatment, Building 371, Room 1115	Cl, Fe, Mg	Y	Y	N	N	N	7.7	0	7.7
		63-88-06-121	HANFORD	Calcined PRF Scrap Oxide	Scrap oxide from aqueous recovery	F, Al, Ca, C, Fe, Mg, Na	Y	Y	Y	Y	Y	35.7	0	35.7
		66-00-11-355	HANFORD	$Mg(OH)_2$ Precipitation/Calcination	From Impure Nitrate (Concentrated Filtrate)	Al, Mg, P	Y	Y	Y	Y	Y	29.0	0	29
		66-01-01-439	HANFORD	$Mg(OH)_2$ Precipitation/Calcination	From Purified Nitrate	Mg, P	Y	Y	Y	Y	Y	63.1	0	63.1
		ARF-102-85-355	HANFORD	Oxide from Residue Processing	WG Oxide received from RFETS	Cl, F, C	Y	Y	Y	Y	Y	65.6	0	65.6
	Product	MISSTD-1*	LANL	Precipitation and Calcination of oxalate	Batch oxalate (III) precipitation from nitrate solution and ion-exchange feed.	None	Y	Y	Y	N	Y	86	0	86.0

Note: MIS Sample MISSTD-1 does not currently represent material in storage because this sample has not been calcined at 950°C.

Table A1-1. Table of MIS Representative Samples Showing Process of Origin, Major Impurities, Characterization Comments, and Actinide Content after Stabilization to 950°C. ***(continued)***

Process Category	Process Subcategory	MIS Sample	Source Site	Origination Process	Comments	Major Impurities (>1 wt%)	Surface Area / Density	Chem	Sample Available	Prompt Gamma	Small / Large Scale	% Pu	% U	% Pu+U
Aqueous Processing *(continued)*	Product	PBO-47-09-012-023	HANFORD	Continuous Oxalate Precipitation/Calcination	Converted from purified nitrate/PUREX N-cell	None	Y	Y	Y	Y	Y	87.5	0	87.5
		PEOF1	LANL	Precipitation and Calcination of oxalate	Batch oxalate (III) precipitation from nitrate solution and ion-exchange feed. Obtained from parent PEOFRBJSTD calc at 975 for 4 hours.	None	Y	Y	Y	Y	Y	87.5	0	87.5
		PEOR3258	LANL	Precipitation and Calcination of oxalate	Batch oxalate (III) precipitation from nitrate solution and ion-exchange feed. Pure Pu oxide standard.	None	Y	Y	N	N	N	87.8	0	87.8
		07161856	RFETS	Precipitation and Calcination of Peroxide	Calcination, Building 771, Room 114	None	Y	Y	Y	Y	Y	84.2	0	84.2
		1000089	RFETS	Precipitation and Calcination of Peroxide	Precipitation/Calcination, Building 371, Room 3511	None	Y	Y	Y	N	Y	84.5	0	84.5
		BLO-39-11-14-004	HANFORD	Continuous Oxalate Precipitation/Calcination	Converted from purified nitrate/PFP RMA Line	None	Y	Y	N	Y	Y	85.2	0	85.2
		CXLNM1	LANL	LANL Chloride line	Pure Pu oxide produced from oxalate precipitated from chloride solution	Unknown	Y	N	Y	N	N	87.3	0	87.3
		CXLOX091802	LANL	Precipitation and Calcination of Oxlate	Pure Pu oxide produced from oxalate precipitated from chloride solution	Cl, F, K, B	Y	Y	Y	N	Y	76.6	0	76.6
		CXLPROD021202	LANL	Precipitation and Calcination of Oxlate	Pure Pu oxide produced from oxalate precipitated from chloride solution	None	Y	Y	Y	N	Y	87.8	0	87.8
		CXLPROD091901	LANL	Precipitation and Calcination of Oxlate	Pure Pu oxide produced from oxalate precipitated from chloride solution	None	Y	Y	Y	N	Y	87.6	0	87.6

Table A1-1. Table of MIS Representative Samples Showing Process of Origin, Major Impurities, Characterization Comments, and Actinide Content after Stabilization to 950°C. ***(continued)***

Process Category	Process Subcategory	MIS Sample	Source Site	Origination Process	Comments	Major Impurities (>1 wt%)	Surface Area / Density	Chem	Sample Available	Prompt Gamma	Small / Large Scale	% Pu	% U	% Pu+U
Metal Oxidation	Byproduct	TS707013	RFETS	Metal Oxidation	Thermal Stabilization, Building 707 J-Module After 1996	Cl, Fe, Mg, K, Na	Y	Y	Y	N	Y	69.8	0	69.8
		ARF-102-85-114-1	HANFORD	Metal Oxidation	WG Foundry Oxide received from RFETS	None	Y	Y	Y	Y	Y	86.3	0	86.3
	Product	MT1490	RFETS	High-Purity Plutonium Oxide Bearing Np	Plutonium Metallurgy R&D, Building 771, Room 182	None	Y	Y	Y	Y	Y	85.6	0	85.6
		TS707001	RFETS	Metal Oxidation	Thermal Stabilization, Building 707 J-Module After 1995	None	Y	Y	Y	Y	Y	87	0	87
		011589A	RFETS	Metal Oxidation	Thermal Stabilization, Building 707, J-Module Before 1990	Cl, Mg	Y	Y	Y	Y	Y	77.7	0	77.7
		011608	RFETS	Metal Oxidation	Thermal Stabilization, Building 707, J-Module Before 1990	None	Y	Y	N	N	N	84.5	0	84.5
		UPOPLOT0003	LANL	Metal Oxidation	Aries Oxide; Direct Metal Oxidation	None	Y	Y	Y	Y	N	87.0	0.4	87.4
		07221730	RFETS	Metal Oxidation	Metal and Chip Burning, Building 771, Room 114	None	Y	Y	N	N	N	85.8	0	85.8
Miscellaneous	Miscellaneous	PPSL-365	HANFORD	Direct de-nitration	Converted from nitrate at PFP PPSL calciner	None	Y	Y	Y	Y	N	83.4	0	83.4
		PuF4-1	LANL	PuF4 Precipitation	PuF4 Line from DP (LANL)	Cl, F	Y	Y	Y	Y	Y	72.7	0	72.7
		64-85-12-1858	HANFORD	Calcined C-Line Scrap Oxide	Scrap oxide for metal reduction (oxalate precipitation, hydrofluorination, and/or reduction to metal)	Ca, Fe	Y	Y	Y	Y	Y	32.7	0	32.7
		41-85-08-1379	HANFORD	Scrap WG oxide from analytical laboratories	Impure oxide (scrap and returns from analytical lab)	Al, F, Mg, Na	N	N	Y	Y	Y	34.1	0	34.1
		04272-CC-220	LANL	Unknown	Plutonium oxide containing Thorium	Cl, K	N	N	Y	Y	Y	82.9	0.2	83.1

Table A1-1. Table of MIS Representative Samples Showing Process of Origin, Major Impurities, Characterization Comments, and Actinide Content after Stabilization to 950°C. ***(continued)***

Process Category	Process Subcategory	MIS Sample	Source Site	Origination Process	Comments	Major Impurities (>1 wt%)	Surface Area / Density	Chem	Sample Available	Prompt Gamma	Small / Large Scale	% Pu	% U	% Pu+U
Mixed Actinide Operations	Byproduct	PSU-84-06-05	HANFORD	Recovery from Pyrolytic Processing	Mixed Oxide recovered from polycube/PFP	None	Y	Y	Y	Y	Y	13.4	65.1	78.5
		PuUOXBC05	LANL	Unknown	Impure Pu/U oxide originally packaged Nov 1983; High Ca, Mg, K, Na	F, Ca, Cu, Mg, Zn	Y	Y	Y	Y	N	36.1	14.7	50.8
		053038	RFETS	By-product Plutonium-Uranium Oxide	Hydroxide Precipitation, Building 771	Cl, Ca, Cr, Fe, K, Na, Zn	Y	Y	Y	Y	Y	60.4	3.7	64.1
		5501407	RFETS	Hydride Oxide / By-product Plutonium-Uranium Oxide	Hydride Operations Building 779, Rooms 152A / 160A	S	Y	Y	Y	Y	Y	65.8	10.9	76.7
		62750	RFETS	By-product Plutonium-Uranium Oxide	Dissolution, Building 371, Room 1115	None	Y	Y	N	N	N	85.9	0.5	86.4
		669194	RFETS	By-product Plutonium-Uranium Oxide	Special Assembly Projects, Building 777	None	Y	Y	Y	Y	Y	13.8	69.2	83
		CAN92	RFETS	By-product Plutonium-Uranium Oxide	Analytical Lab Production Support, Building 559	None	Y	Y	Y	Y	Y	81.6	2.3	83.9
	Product	SCP711-46	LANL	Hot Plate Oxidation	Fuel Pellets and powder	C	Y	Y	Y	Y	N	6.2	78.3	84.5
		SCP711-56	LANL	Hot Plate Oxidation	Fuel Pellets and powder	None	Y	Y	Y	Y	Y	17.5	70	87.5
		5501579	RFETS	Hydride Oxide / By-product Plutonium-Uranium Oxide	Hydride Operations Building 779, Rooms 152A / 160A	Ga	Y	Y	Y	Y	Y	85.1	0.1	85.2
	Miscellaneous	CXL1685	LANL	Mixture of Pu Oxide and Depleted Uranium	Pu / U mixture	None	Y	Y	Y	N	N	6.0	79.9	85.9

Table A1-1. Table of MIS Representative Samples Showing Process of Origin, Major Impurities, Characterization Comments, and Actinide Content after Stabilization to 950°C. ***(continued)***

Process Category	Process Subcategory	MIS Sample	Source Site	Origination Process	Comments	Major Impurities (>1 wt%)	Surface Area / Density	Chem	Sample Available	Prompt Gamma	Small / Large Scale	% Pu	% U	% Pu+U
Molten Salt Operations	Byproduct	PMAXBS	LANL	Oxide from Pyrochemical Process	Mixture of anode heels and ER salts	Cl, Ga, K, Na	Y	Y	Y	Y	Y	71.9	0	71.9
		07242141A	RFETS	Screenings from Oxide packaged for off site shipment	Unknown origin	Fe	Y	Y	Y	Y	Y	43.1	0	43.1
		520610020	RFETS	Oxide from Pyrochemical Processes	Pyrochemistry Technology Development, Building 779	Cl, Al, Ca, Mg, Ni, K, Na	Y	Y	Y	N	Y	33.7	0	33.7
		ARF-102-85-223	HANFORD	Scrap Oxide from Pyrochemical Process	WG Oxide received from RFETS	Cl, K, Na	Y	Y	Y	Y	Y	70.9	0	70.9
		ARF-102-85-295	HANFORD	Scrap Oxide from Pyrochemical Process	WG Oxide received from RFETS	Cl, Fe, Mg, Ni, K, Na	Y	Y	Y	Y	Y	39.6	0	39.6
		ARF-102-85-365	HANFORD	Scrap Oxide from Pyrochemical Process	WG Oxide received from RFETS	Cl, Mg, K, Na	Y	Y	Y	Y	Y	68.4	0	68.4
		ATL27960	LANL	From advanced testing line for actinide separation (ATLAS)	Washed Pyrochemical Salt	C, K, Na	Y	Y	N	N	N	74.2	0	74.2
		C00024A	RFETS	Oxide from Pyrochemical Processes. IDC defined as LOI reject unsure of source	Calcined then stored in the B371 S/R at one time	Cl, Ga, Mg, K, Na	Y	Y	Y	Y	Y	74.3	0	74.3
		C00695	RFETS	Oxide from Pyrochemical (ER tilt pour) Processes	ER tilt pour operations, Building 371. Calcined and stored in the B371 S/R at one time	Cl, Mg, K, Na	Y	Y	Y	Y	Y	74.1	0	74.1
		C06032A	RFETS	Screenings from Oxide packaged for off-site shipment	Split Can, Calcined then stored in the B371 S/R.	Cl, Mg, K, Na	Y	Y	Y	N	Y	65.9	0	65.9
		CLLANL025	RFETS	Oxide from Pyrochemical (ER tilt pour) Processes	ER tilt pour operations, Building 371	Cl, K, Na	Y	Y	Y	Y	Y	77.7	0	77.7

Table A1-2. Table of MIS Nonrepresentative Samples Showing Process of Origin, Major Impurities, Characterization Comments, and Actinide Content
Note: None of the nonrepresentative samples have been included in small and large-scale shelf-life studies. Stabilization temperatures vary.

Process Category	Process Subcategory	MIS Sample	Source Site	Origination Process	Comments	Major Impurities (>1 wt%)	Surface Area / Density	Chem	Sample Available	Prompt Gamma	% Pu	% U	% Pu+U
Aqueous Processing	Byproduct	07242243A	RFETS	Dissolution Residuals (from foundry scrap oxides)	Low Purity Heel	F	N	N	Y	Y	24.6		
		07242326A	RFETS	Dissolution Residuals (from foundry scrap oxides)	Dissolution Heel	F	N	N	Y	Y	15.7		
	Product	MISSTD-2	LANL	Precipitation and Calcination of oxalate	Batch oxalate (III) precipitation from nitrate solution and ion-exchange feed	Unknown	N	N	Y	N	87.3	0	87.3
Metal Oxidation	Byproduct	101707001	RFETS	Metal Oxidation	Foundry Oxide	Cl, Na	N	N	Y	Y	19.6		
Misc	Misc	MISNE4	LANL	Mix of 520610020, 11589, C06032A, TS707013, 1000089, and 39-01153	Impure items (Mg and Ca) combined, V-blended, gypsum added and calcined numerous times	Cl, Ca, Fe, Mg	Y	Y	Y	Y	66.8	0	66.8
		YBG2-NRDL-4	LANL	Standard from the Navy	Unknown origin	F	N	N	Y	Y	77.5	0	77.5
Mixed Actinides	Misc	1685	LANL	Unknown	Depleted Uranium Oxide	Unknown	Y	N	Y	N			
		BMU	LANL	By product uranium oxide	Burned uranium metal	Unknown	Y	N	Y	N	0	84.3	84.3
		BMUCXL70-30	LANL	Mixture of 70% BMU (EU) and 30% CXL (PuO2)	Pu / U mixture	Unknown	N	N	Y	N	23.8	58.3	82.1
		BMUCXL93-7	LANL	Mixture of 93% BMU (EU) and 7% CXL (PuO2)	Pu / U mixture	Unknown	N	N	Y	N	6.1	78.5	84.6
		CXLBMU70-30	LANL	Mixture of 70% CXL (PuO2) and 30% BMU (EU)	Pu / U mixture	Unknown	N	N	Y	N	59.9	23.7	83.6
		CXLBMU93-7	LANL	Mixture of 93% CXL (PuO2) and 7% BMU (EU)	Pu / U mixture	Unknown	N	N	Y	N	80.8	5.9	86.7
		MISNE2	LANL	Mix of 7221730, TS707001, 11608, and 62750	MISNE2 post neutron moderation. Contains borax and gypsum.	None	Y	Y	Y	Y	84.6	2	86.6
	Product	MOXSCP-COM	LANL	Calcined MOX	MOX fuel pellets and powder	Unknown	N	N	Y	N	4.9	81.6	86.5

Appendix 2, Weight Loss from Calcination

Table A2-1. Representative Samples: Summary of Weight Loss Due to Calcination at 950°C Averaged by Process Category

Process Category (Operation)	Process Subcategory (Oxide)	Number of Items	Average % Wt Loss @ 950 C	LCL**	UCL**
Aqueous Processing	Byproduct	8	15.09	-0.6	30.8
	Product	9	2.19	-0.4	4.8
Metal Oxidation	Byproduct	2	2.76	NA	NA
	Product	5	0.96	-0.5	2.4
Miscellaneous	Miscellaneous	5	3.09	-3.2	9.3
Mixed Actinide Operations*	Byproduct	7	3.64	0.5	6.8
	Misc.	1	-2.71	NA	NA
	Product	3	-1.48	-5.1	2.2
Molten Salt Ops.	Byproduct	10	15.63	8.4	22.9

Notes: The NA indicates either there is not enough data or there is an outlier in a small data set.

***Mixed Actinide Materials items contain uranium which tends to gain weight when calcined due to an increase in the oxygen content.**

****The lower 95% confidence limit (LCL) and upper 95% confidence level (UCL) were calculated for many of the averages reported. Small sample sizes made it impossible to determine exact distributions of the data, so these confidence intervals are very approximate, but are better than just reporting averages. At a minimum they give some indication of the variability in the data.**

LCLs and UCLs for Percent Weight Loss and Particle size were calculated using standard theory assuming the data are normally distributed but with unknown standard deviation e.g. mean ± t*SD where t is from t distribution with n-1 degrees of freedom where n is the sample size, SD= Standard deviation.

The LCL and UCLs for average surface area reduction, LOI and packing fractions are based on taking the logit transformation and finding the confidence intervals assuming the transformed data are normal. The inverse logit is used to transform back to the original data. Logit transformation for a measurement x: $logit(x) < -\log\left(\frac{x}{1-x}\right)$

For the LCL and UCLs for average density measurements a natural log transformation was used.

Table A2-2. Weight Loss Due to Calcination Arranged by Process Category and MIS Sample

Process Category	Process Subcategory	MIS Sample	% Wt Loss @ 600°C	% Wt Loss @ 800°C	% Wt Loss @ 950°C	% Wt Loss @ 950°C for Average
Aqueous Processing	Byproduct	07032282A	3.72		4.11	4.11
		07242165A		14.67	15.26	15.26
		07242201A (chunks)			16.46	14.53
		07242201A (powder)			14.53	
		39-01153A		49.40	52.66	52.66
		63-88-06-121			-0.19	-0.19
		66-00-11-355			1.32	1.32
		66-01-01-439			0.00	0.00
		ARF-102-85-355	33.71		-1.68	32.03
	Product	07161856		10.41	10.47	10.47
		1000089		0.85	0.50	0.50
		BLO-39-11-14-004	2.23		0.39	2.63
		CXLNM1			0.93	0.93
		CXLOX091802			4.06	4.06
		CXLPROD021202			0.71	0.71
		CXLPROD091901			0.70	0.70
		PBO-47-09-012-023	0.68		0.01	0.70
		PEOF1			0.83	0.83
Metal Oxidation	Byproduct	ARF-102-85-114-1	-0.27		0.15	0.15
		TS707013	1.86		5.36	5.36
	Product	011589A			2.84	2.84
		011608		-0.92	0.20	0.20
		07221730		0.33	-0.33	-0.33
		MT1490		0.79	0.99	0.99
		TS707001	0.75		0.35	1.10

Note: Shading indicates sequential calcination.

Table A2-2. Weight Loss Due to Calcination Arranged by Process Category and MIS Sample *(continued)*

Process Category	Process Subcategory	MIS Sample	% Wt Loss @ 600°C	% Wt Loss @ 800°C	% Wt Loss @ 950°C	% Wt Loss @ 950°C for Average
Miscellaneous	Miscellaneous	04272-CC-220			2.47	2.47
		41-85-08-1379			11.00	11.00
		64-85-12-1858			-2.70	-2.70
		PPSL-365	0.36		0.24	1.04
		PPSL-365			1.47	
		PuF4-1			4.43	7.44
		PuF4-1			3.01	
Mixed Actinide Operations	Byproduct	053038		2.08	7.10	7.10
		5501407			7.76	7.76
		62750		0.89	0.91	0.91
		669194	-0.49		0.00	0.00
		CAN92			0.69	0.69
		PSU-84-06-05	0.48		1.97	2.45
		PuUOXBC05	4.46		2.12	6.58
	Miscellaneous	CXL1685			-2.71	-2.71
	Product	5501579			0.07	0.07
		SCP711-46			-2.85	-2.85
		SCP711-56			-1.65	-1.65
Molten Salt Operations	Byproduct	07242141A	1.74		30.75	32.49
		520610020		12.61	12.71	12.71
		ARF-102-85-223	0.55		10.17	10.73
		ARF-102-85-295 (chunks)	0.21		31.09	29.10
		ARF-102-85-295 (powder)	0.41		28.70	
		ARF-102-85-365	0.68		13.96	14.64
		ATL27960	1.41		4.23	5.63
		C00024A		4.56	5.00	5.00
		C00695		7.13	10.34	10.34
		C06032A (chunks)			21.46	30.23
		C06032A (powder)	5.18		30.23	
		CLLANL025			8.78	8.78
		PMAXBS		12.22		

Note: Shading indicates sequential calcination.

Table A2-3. Non Representative Samples - Weight Loss Due to Calcination Arranged by Process Category and MIS Sample

Process Category	Process Subcategory	Item Id	Comments	% Wt Loss @ 950 C
Misc	Misc	MISNE4	Impure items (Mg and Ca) combined, V-blended, gypsum added and calcined numerous times	0.88
Mixed Actinides	Misc	1685		-2.66
		BMU		0.09
		MISNE2	Pure items combined, V-blended, borax added and calcined	1.61
		MISNE2	Re-run of moisture analysis on MISNE2. Gypsum added and calcined.	1.03
		MISNE2	MISNE2 post neutron moderation. Contains borax and gypsum.	0.003

Appendix 3, LOI and TGA

Table A3-1. Average LOI Percent Weight Change Arranged by Process Category for samples calcined to 950°C Conditions

Process Category	Process Subcategory	Number of Items	Average LOI (wt%) 950°C	95% LCL	95% UCL
Aqueous Processing	Byproduct	5	1.4	0.24	4.3
	Product	8	1.0	0.21	5.0
Metal Oxidation	Byproduct	2	2.1		
	Product	5	0.6	0.02	2.8
Mixed Actinide Operations	Byproduct	7	0.6	0.11	4.6
	Product	3	0.04	0.01	0.2
Molten Salt Operations	Byproduct	9	5.3	3.2	10.5

Table A3-2. LOI Results Arranged by Process Category and MIS Sample for All Conditions

Process Category	Process Subcategory	MIS Sample	LOI (wt%) AR	LOI (wt%) 600°C	LOI (wt%) 800°C	LOI (wt%) 950°C
Aqueous Processing	Byproduct	07032282A	3.29	4.50		1.16
		07242165A	13.89		0.26	0.13
		07242201A (chunks)				1.20
		07242201A (powder)	13.37			2.15
		39-01153A	54.81		6.40	2.08
		66-00-11-355	0.37			
		66-01-01-439	0.30			
		ARF-102-85-355	64.40	1.07		1.58
	Product	07161856	10.02		0.92	0.16
		1000089	0.90		0.84	1.33
		BLO-39-11-14-004	2.49	0.48		0.05
		CXLOX091802	10.25			5.84
		CXLPROD021202	0.72			0.12
		CXLPROD091901	0.90			0.13
		MISSTD-1	2.35			
		PBO-47-09-012-023	0.75	0.15		0.04
		PEOR3258				0.02
Metal Oxidation	Byproduct	ARF-102-85-114-1	0.51	0.80		0.24
		TS707013	7.42	6.90		3.94
	Product	011589A	4.15			1.82
		011608	1.17		1.62	0.81
		07221730	0.03		0.34	0.25
		MT1490	0.73		0.22	0.21
		TS707001	0.82	0.22		0.01
Misc.	Misc.	PPSL-365	0.35			0.06
		PPSL-365		0.18		0.04

Table A3-2. LOI Results Arranged by Process Category for All Conditions *(continued)*
Note: Multiple results for the same sample and condition are averaged.

Process Category	Process Subcategory	MIS Sample	LOI (wt%) AR	LOI (wt%) 600°C	LOI (wt%) 800°C	LOI (wt%) 950°C
Mixed Actinide Operations	Byproduct	053038	0.08		4.75	3.94
		5501407	7.14			0.13
		62750	29.15		0.20	-0.24
		669194	-0.07	0.05		0.03
		CAN92	0.59			0.02
		PSU-84-06-05	0.14	0.15		0.06
		PuUOXBC05	5.94	1.73		0.32
	Misc.	CXL1685	-2.70			0.47
	Product	5501579	0.36			0.07
		SCP711-46	6.12			0.02
		SCP711-56	-1.31			0.04
		SCP711-56	-3.84			
Molten Salt Operations	Byproduct	07242141A		3.83		
		520610020	4.49		2.81	2.77
		ARF-102-85-223	7.91	8.66		7.31
		ARF-102-85-295 (chunks)	10.92	9.65		9.95
		ARF-102-85-295 (powder)	7.18	8.33		7.48
		ARF-102-85-365	9.32	7.12		6.10
		ATL27960	4.26	2.41		0.99
		C00024A	4.20		2.34	1.71
		C00695	9.14		7.78	6.22
		C06032A (chunks)				9.21
		C06032A (powder)	5.86	10.88		7.32

Table A3-3. TGA-DSC Results Arranged by Process Category and MIS Sample for Given Conditions
Note: RT is Room Temperature

Process Category	Process Subcategory	MIS Sample	Condition	TGA Mass Loss (wt%)	
				RT to 200°C	RT to 1000°C
Aqueous Processing	Byproduct	07242165A	AR	7.62	0.23
		39-01153A	AR	39.62	11.35
	Product	07161856	AR	3.80	8.00
		1000089	AR	0.36	1.23
		BLO-39-11-14-004	AR	2.00	2.30
			600C	0.00	0.37
			950C	0.00	0.09
		PBO-47-09-012-023	950C	0.32	0.32
Metal Oxidation	Product	011608	AR	0.13	1.83
		07221730	AR	0.36	0.36
		MT1490	AR	0.90	0.26
Mixed Actinides	Byproduct	053038	AR	9.88	18.73
		5501407	AR	1.00	4.70
			950C	0.00	0.05
		62750	AR	0.00	0.00
		CAN92	AR	1.00	1.00
			950C	0.00	-0.22
		PSU-84-06-05	AR	0.00	-0.23
			950C	0.00	-0.94
Molten Salt Operations	Byproduct	520610020	AR	8.35	17.44
			800C	8.88	19.28
			950C	11.18	17.96
		ARF-102-85-365	AR	2.17	13.80
		C00024A	AR	1.00	7.20
		C00695	AR	0.62	6.57
			950C	2.06	11.45

Table A3-4. TGA-MS Results Arranged by Process Category and MIS Sample for Given Conditions.

Process Category	Process Subcategory	MIS Sample	Condition	TGA Mass Loss (wt%)			TGA—Mass Spectrometry			
				RT to 200°C	RT to 650°C	RT to 1000°C	H_2O (wt%)	CO_2 (wt%)	NO_2 (wt%)	SO_2 (wt%)
Aqueous Processing	Byproduct	07032282A	950C	0.07	0.22	1.04	0.06	0.17	0.04	
		07242165A	800C / 950C mix	0.13	0.24	0.88	0.09	0.09	0.01	0.000
		07242201A	950C	0.13	0.61	1.52	0.23	0.15	0.17	0.251
		66-00-11-355	950C	0.17	0.18	0.09	0.10	0.19	0.01	0.003
		66-01-01-439	950C	0.18	0.39	0.42	0.19	0.09	0.06	0.002
		ARF-102-85-355	750C	0.35	1.59	9.36	0.23	4.11	0.07	0.079
	Product	07161856	950C	0.41	1.46	1.98	0.63	0.05	0.79	
		1000089	800C / 950C mix	0.28	0.62	1.09	0.36	0.38	0.04	
		BLO-39-11-14-004	950C	0.39	0.76	1.58	0.37	0.08	0.17	0.001
		MISSTD-1	600C	0.35	2.12	2.28	1.18			
			600C	1.12	2.91	3.32	1.29	0.88	0.40	
		PBO-47-09-012-023	950C	0.14	0.25	0.20	0.13	0.21	0.07	0.000
		PEOF1	950C	0.04	0.48	0.48	0.20	0.48	0.01	
Metal Oxidation	Byproduct	ARF-102-85-114-1	950C	0.08	0.14	0.18	0.07	0.12	0.03	
		TS707013	600C / 950C mix	0.05	0.19	1.38	0.07	0.18	0.03	
	Product	011589A	950C	0.40	0.65	1.62	0.47	0.26	0.01	
		MT1490	800C / 950C mix	0.07	0.19	0.35	0.06	0.05	0.09	
		TS707001	950C	0.10	0.26	0.29	0.08	0.01	0.16	
		UPOPLOT0003	950C	0.00	0.01	0.00	0.01			
Miscellaneous	Miscellaneous	04272-CC-220	950C	0.02	0.09	0.09	0.02			
		41-85-08-1379	950C	0.24	0.63	0.77	0.17	0.41	0.02	
		64-85-12-1858	950C	0.08	0.21	0.18	0.10	0.13	0.04	0.000
		PuF4-1	950C	0.07	0.73	3.24	0.03	0.11	0.03	
Mixed Actinides	Byproduct	053038	950C	0.34	0.72	2.29	0.46	0.20	0.04	
		5501407	AR / 950C mix	0.39	1.29	3.55	0.87	0.07	0.09	2.475
			900C	0.05	0.23	0.87				
		669194	600C / 950C mix	0.07	0.19	0.46	0.09	0.03	0.01	0.022
		CAN92	950C	0.05	0.12	0.20	0.05	0.16	0.06	
		PSU-84-06-05	950C	0.11	0.25	0.44	0.12	0.01	0.06	0.000
	Product	5501579	800C / 950C mix	0.10	0.23	0.27	0.09	0.01	0.14	
		SCP711-56	950C	0.04	0.17	0.33	0.04	0.05	0.02	0.000

Table A3-4. TGA-MS Results Arranged by Process Category and MIS Sample for Given Conditions *(continued)*

Process Category	Process Subcategory	MIS Sample	Condition	TGA Mass Loss (wt%)			TGA—Mass Spectrometry			
				RT to 200°C	RT to 650°C	RT to 1000°C	H_2O (wt%)	CO_2 (wt%)	NO_2 (wt%)	SO_2 (wt%)
Molten Salt Operations	Byproduct	07242141A	950C	0.03	0.13	1.21	0.06	0.83	0.02	0.030
		520610020	750C	0.24	0.57	1.46	0.31	0.33	0.07	0.001
		ARF-102-85-223	750C	0.17	0.38	2.30	0.15	0.04	0.02	0.009
		ARF-102-85-295	750C	0.08	0.25	3.55	0.14	0.15	0.02	
			950C	0.28	0.61	2.20	0.34	0.16	0.11	
		ARF-102-85-365	750C	0.15	0.29	1.67	0.16	0.22	0.04	0.000
		C00024A	800C / 950C mix	0.20	0.34	1.14	0.22	0.06	0.03	
		C00695	800C / 950C mix	0.33	0.68	2.03	0.38	0.09	0.03	
		C06032A	950C	0.25	0.44	3.08	0.23	0.05	0.02	0.008
		CLLANL025	750C	0.11	0.25	1.99	0.16	0.06	0.02	0.000
		PMAXBS	800C	0.04	0.19	1.55	0.04	0.08	0.02	

Note 1: RT is Room Temperature
Note 2: Blanks indicate that no measurement was taken.

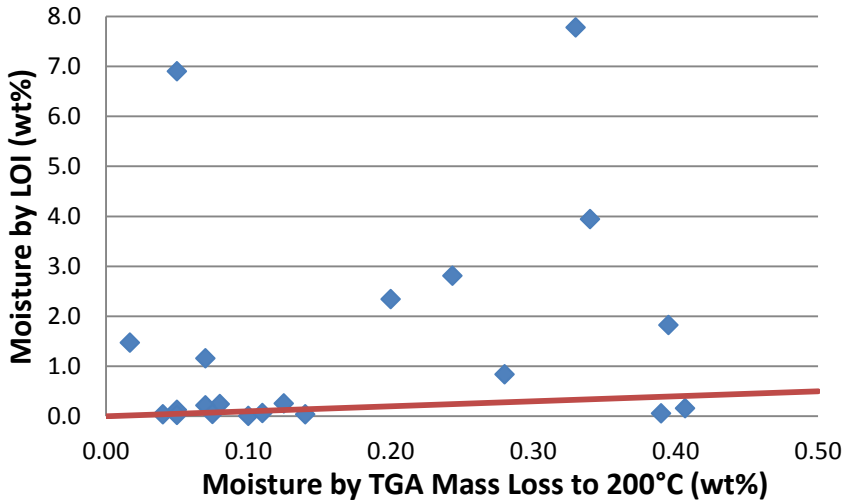

Figure A3-1 Comparison of the weight percent loss measured by LOI with the TGA mass loss measured to 200°C. The solid line with a slope of 1.0 and an intercept of 0.0 is included for reference to highlight deviation from equal LOI and TGA-MS measurements. The largest TGA measurement was 0.42 wt%. Contrary to what would be expected, for seven out of the twenty-one measurements (33%), the TGA-MS weight percent loss exceeded the weight loss by LOI. The discrepancy may result from the heterogeneity of the material sampled or differences in sample handling after the material was stabilized. For example, for some materials, a time lapse of up to five years occurred between stabilization and the TGA measurement.

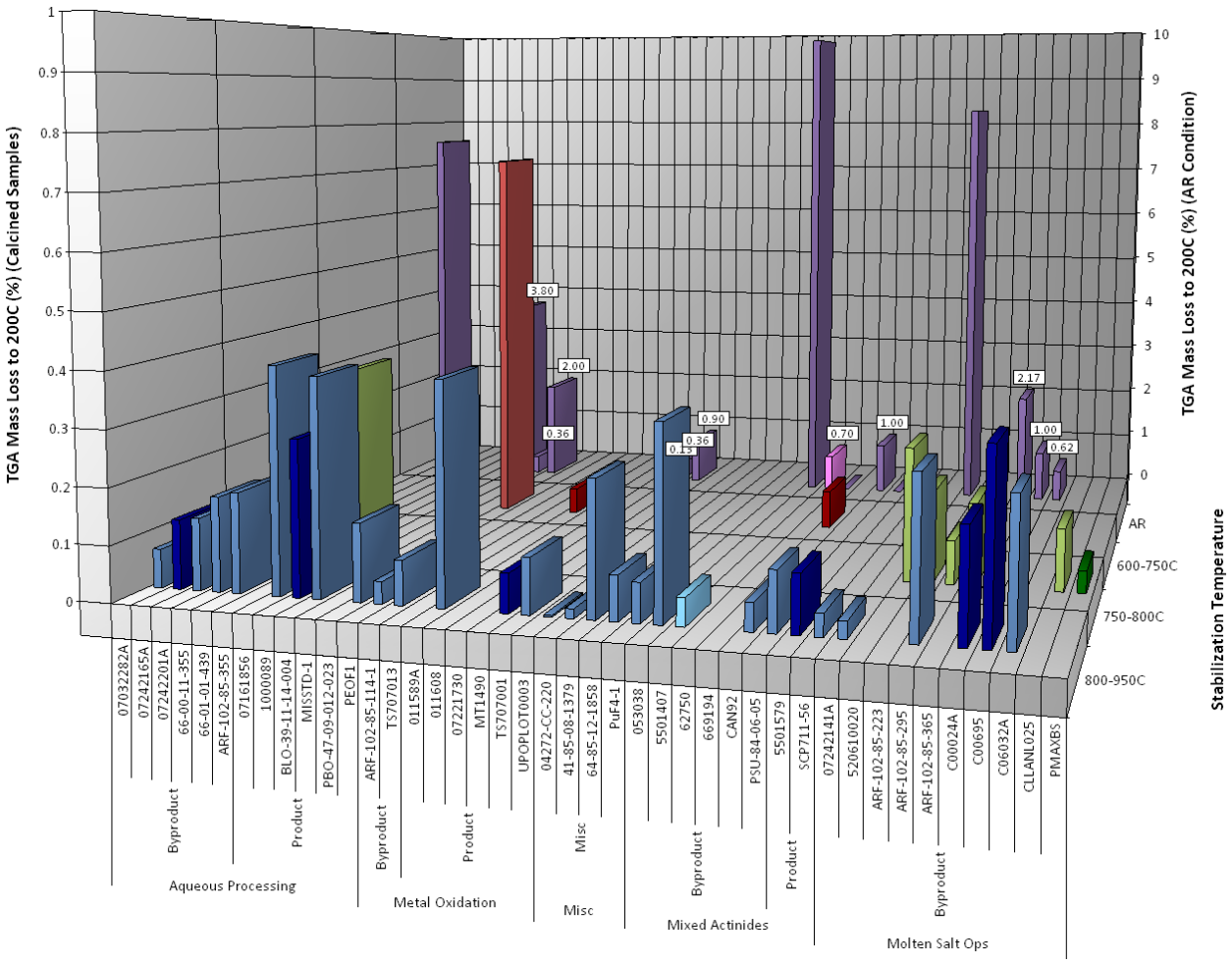

Figure 3A-2. TGA measurements for the MIS samples "as-received" and after calcination at various temperatures.
The light-blue bars indicate samples calcined at 900°C.
The medium-blue bars indicate samples calcined at 950°C.
The dark-blue bars indicate samples containing mixtures of material calcined at 800°C and 950°C.
The light-green bars indicate samples calcined at 750°C.
The dark-green bar indicates sample calcined at 800°C.
The light-red bar indicates a sample calcined at 600°C
The dark-red bars indicate samples containing material from the "as-received" condition mixed with material calcined at 600°C.
The pink bars indicate samples containing mixtures of "as-received" material and material calcined at 950°C.
The data for materials in the "as-received" condition are plotted on the right vertical axis; all other data are plotted on the left vertical axis.

The above figure illustrates the effect of calcination on MIS samples by comparing the moisture estimates based on the mass loss to 200°C for samples calcined at various temperatures. As expected, the moisture estimates for the calcined samples are much lower than those for samples in the "as-received" condition. MIS samples 053038 and 520610020 have the highest mass losses of the "as-received" samples. Both materials are known to contain magnesium chlorides and/or calcium chlorides, which readily absorb moisture from the air. All of the calcined materials with the exception of MISSTD-1 have moisture estimates less than the limit of 0.5 wt% set by DOE-STD-3013. MIS sample MISSTD-1 is a pure oxide that was calcined at 600°C at the time of production. It has a high surface area and readily absorbs moisture, and this material had been exposed to air during storage for at least 10 years prior to sampling for TGA. Several other pure oxide samples (07161856, 1000089, and BLO-39-11-14-004) were calcined about 5 years prior to sampling for TGA, and they are among the highest in moisture for the group of materials calcined between 800 and 950°C. The material was stored in the dry glovebox lines or the vault during this interim time.

Appendix 4, Particle Size Data

Table A4-1. Summary of Particle Size Data Averaged by Process Category for MIS Samples Calcined to 800°C or 950°C (minimal suspension time used)

Process Category	Process Subcategory	Number of Items	Average Volume % under 3 µm (LCL,UCL)	Mean Particle Size (µm) (LCL,UCL)
Aqueous Processing	Product	8	11.5 (7.8, 15.2)	14.2 (10.3, 18.2)
	Byproduct	2	37	11
Metal Oxidation	Byproduct	1	18	7
Mixed Actinide Operations	Byproduct	2	11	16
Molten Salt Operations	Byproduct	3	23	16
Miscellaneous	Miscellaneous	1	11	10

Note: Because of the small sample size, LCL and UCL were not calculated for most process subcategories.

Table A4-2. Particle Size Data Arranged by Process Category and MIS Sample for All Conditions (short suspension times -30 seconds or less)

Process Category	Process Subcategory	MIS Sample	Condition	Sonication Time (min)	Volume % under 3 microns	Number Of Modes	Mean (µm)	Mode 1 Mean (µm)	Mode 2 Mean (µm)	Mode 3 Mean (µm)
Aqueous Processing	Product	07161856	950C	0.25	12.7	3	24.2	1.8	15.8	48.0
		BLO-39-11-14-004	950C	0.18	6.6	2	11.5	1.3	12.3	
		CXLNM1	950C	3.00	9.0	1	9.7			
		CXLOX091802	950C	0.17	6.6	1	13.0			
		CXLPROD091901	950C	0.00	14.6	1	16.7			
		CXLPROD021202	950C	0.22	8.2	1	15.4			
		MISSTD-1	AR	1.00	20.2	1	8.4			
				0.25	18.0	3	10.6	2.1	11.1	60.5
					17.3	3	10.3	2.2	11.5	68.3
					17.9	3	11.0	2.2	10.9	66.7
		PBO-47-09-012-023	950C	0.17	17.1	3	14.0	2.0	13.6	52.0
					16.7	2	13.5	1.9	16.1	
		PEOF1	950C	4.00	17.4	1	9.8			

Table A4-2. Particle Size Data Arranged by Process Category and MIS Sample for All Conditions (minimal suspension time used) *(continued)*

Process Category	Process Subcategory	MIS Sample	Condition	Sonication Time (min)	Volume % under 3 microns	Number Of Modes	Mean (µm)	Mode 1 Mean (µm)	Mode 2 Mean (µm)	Mode 3 Mean (µm)
Aqueous Processing *(continued)*	Byproduct	66-00-11-355	950C	1.00	58.3	2	4.5	1.8	9.0	
		63-88-06-121	950C	1.00	15.7	1	17.5			
			AR	0.50	8.6	2	38.9	13.8	109.1	
Metal Oxidation	Byproduct	ARF-102-85-114-1	950C	0.17	17.2	2	6.4	2.1	7.4	
					18.2	2	7.2	2.3	8.4	
		UPOPLOT0003	950C	0.00	17.0	3	25.7	1.6	13.7	56.3
Mixed Actinide Operations	Byproduct	5501407	950C	0.17	13.0	2	14.1	1.9	14.7	
					14.9	2	13.3	1.9	15.5	
		669194	950C	0.18	7.7	2	17.4	2.1	18.8	
					6.6	3	19.6	2.0	14.4	49.6
Molten Salt Operations	Byproduct	07242141A	950C	0.50	8.8	1	20.8			
		ARF-102-85-365	950C	0.25	22.5	3	19.8	1.9	12.2	66.0
		PMAXBS	800C	0.50	37.0	1	6.8			
Miscellaneous	Miscellaneous	64-85-12-1858	950C	1.00	7.9	2	50.5	13.8	95.4	
			AR	0.50	6.2	2	58.1	14.2	118.5	
		PuF4-1	950C	1.00	22.2	1	11.5			
			950C	0.50	11.4	1	10.0			
			AR	0.50	9.7	1	7.2			

Table A4-2. Representative Samples: Particle Size Data as a Function of Suspension Time by Arranged by Process Category for All Conditions.

Process Category	Process Subcategory	MIS Sample	Condition	Suspension Time (min)	Mean Particle Size (µm)	% Increase in Mean Particle Size with Calcination
Aqueous Processing	Byproduct	63-88-06-121	950C	0.5	74.8	91%
				1.0	79.6	226%
				23.0	15.5	
			AR	0.5	39.2	
				1.0	24.4	
				17.5	18.1	
		66-00-11-355	950C	0.5	8.3	
				1.0	6.2	
				10.0	3.6	
	Product	MISSTD-1	AR	0.5	9.7	
				1.0	8.9	
				11.0	7.5	
Misc.	Misc.	64-85-12-1858	950C	0.5	68.6	6%
				1.0	59.5	1%
				16.0	42.6	
			AR	0.5	65.0	
				1.0	58.8	
				15.0	53.8	
		PuF4-1	950C	0.5	20.6	112%
				0.5	11.6	
				1.0	15.7	81%
				1.0	11.1	
				10.0	9.0	
				12.0	9.0	
			AR	0.5	7.6	
				1.0	7.4	
				8.0	7.1	
Molten Salt Operations	Byproduct	07242141A	950C	0.5	30.6	
				1.0	26.4	
				14.0	15.1	
		PMAXBS	800C	0.5	14.1	
				1.0	9.1	
				15.0	5.1	

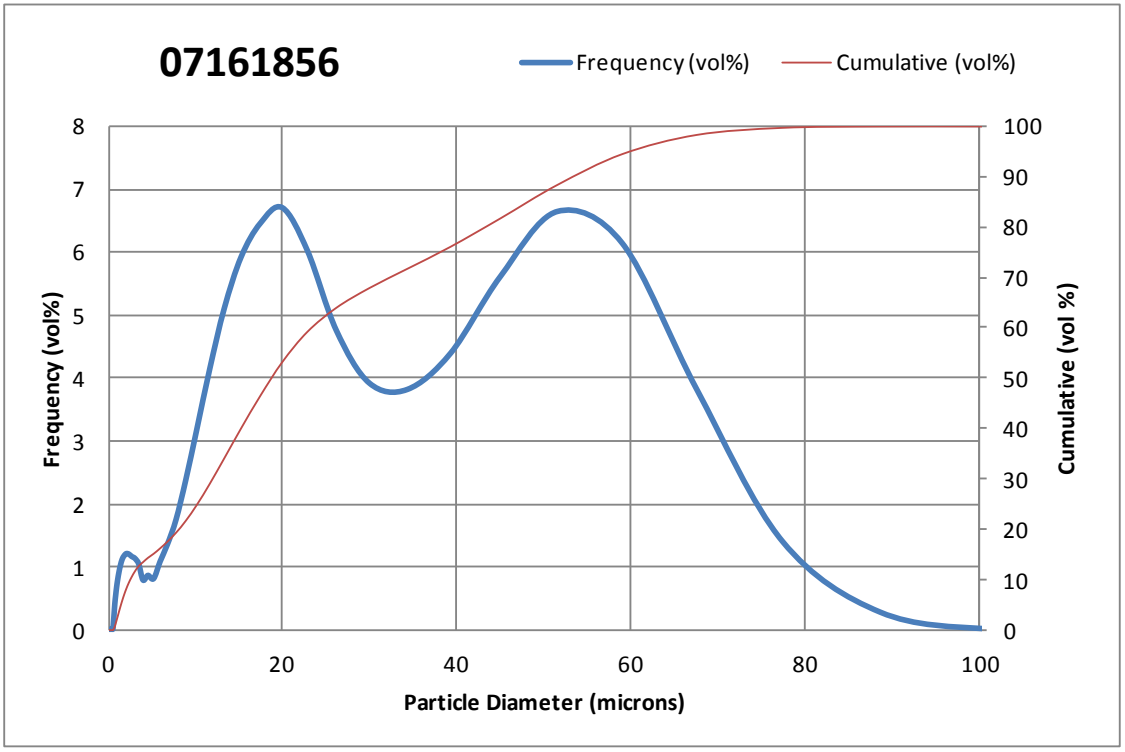

Figure A4-1. Particle size distribution for MIS sample 07161856 (linear scale) by laser diffraction (950°C calcined)

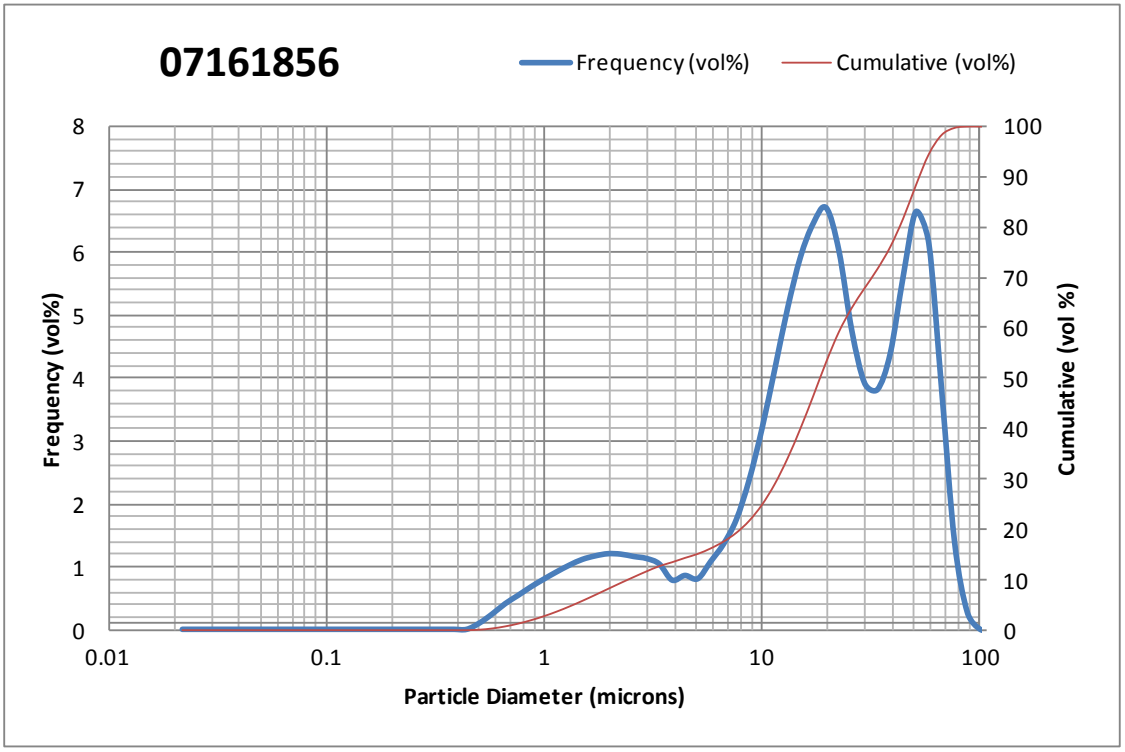

Figure A4-2. Particle size distribution for MIS sample 07161856 (semi-log scale) by laser diffraction (950°C calcined).

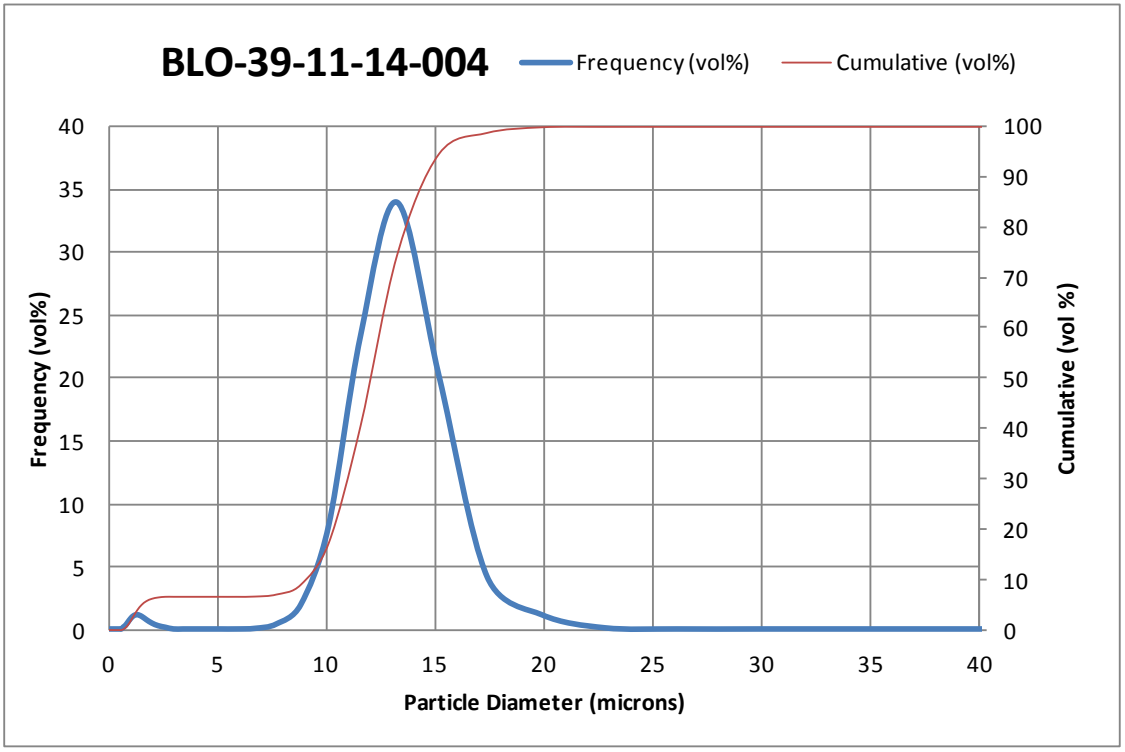

Figure A4-3. Particle size distribution for MIS sample BLO-39-11-14-004 (linear scale) by laser diffraction (950°C calcined).

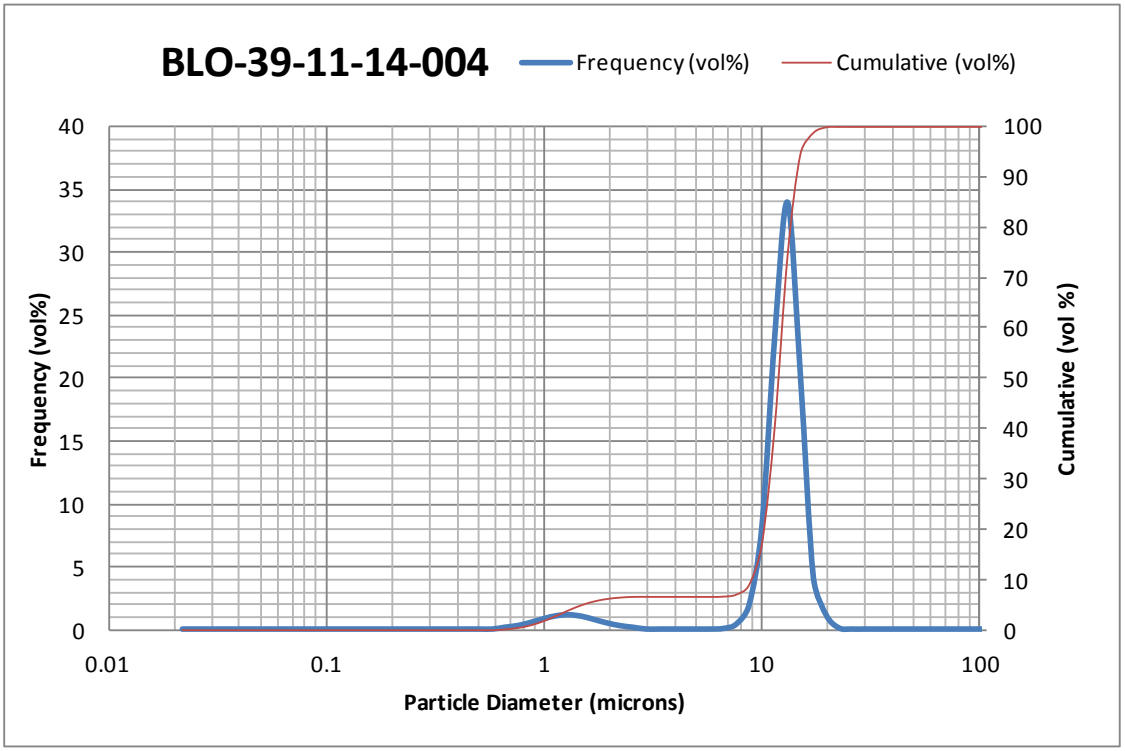

Figure A4-4. Particle size distribution for MIS sample BLO-39-11-14-004 (semi-log scale) by laser diffraction (950°C calcined).

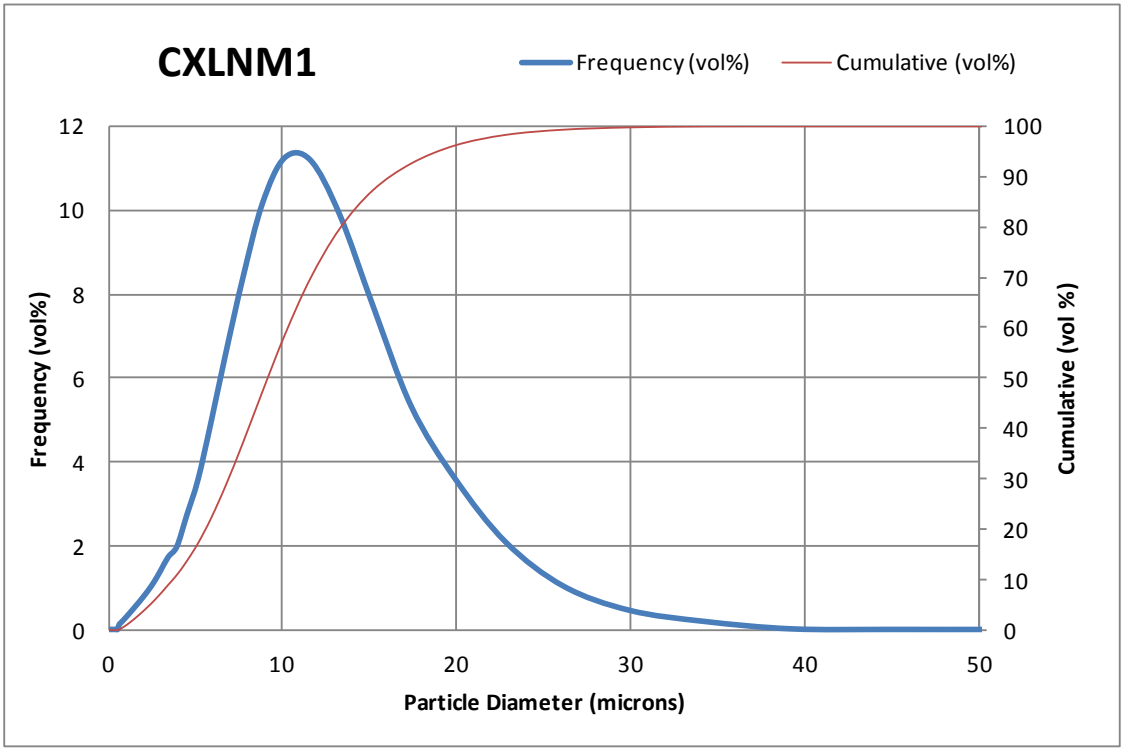

Figure A4-5. Particle size distribution for MIS sample CXLNM1 (linear scale) by laser diffraction (950°C calcined).

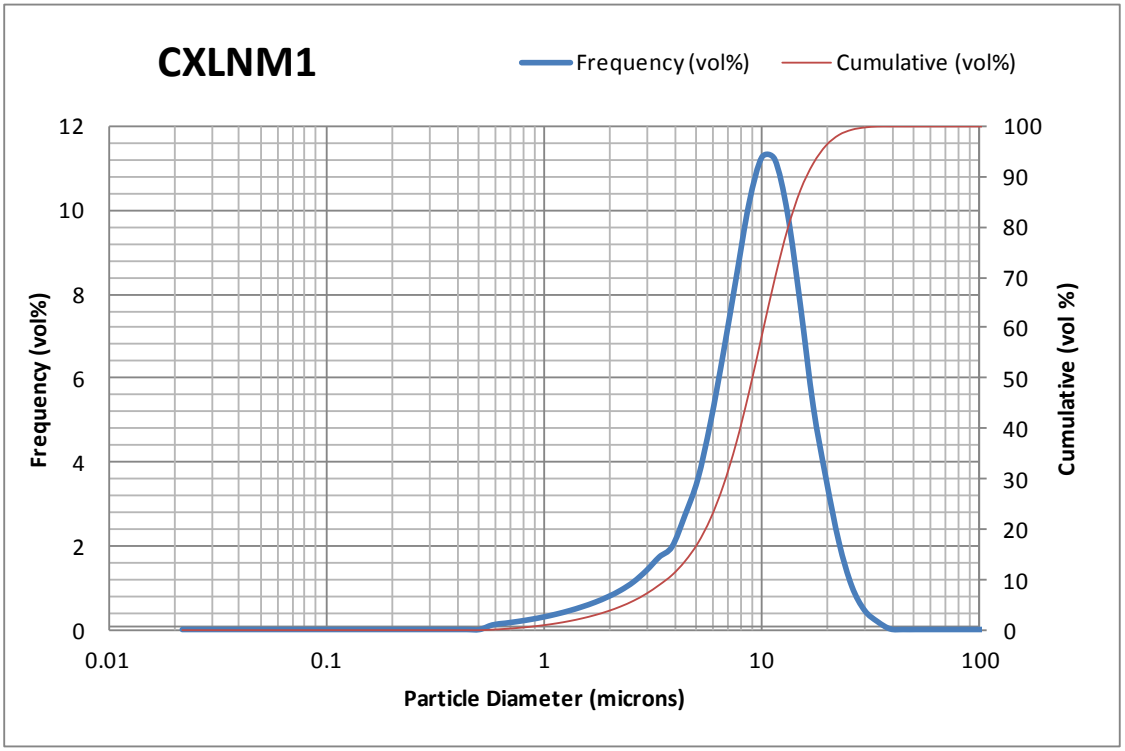

Figure A4-6. Particle size distribution for MIS sample CXLNM1 (semi-log scale) by laser diffraction (950°C calcined).

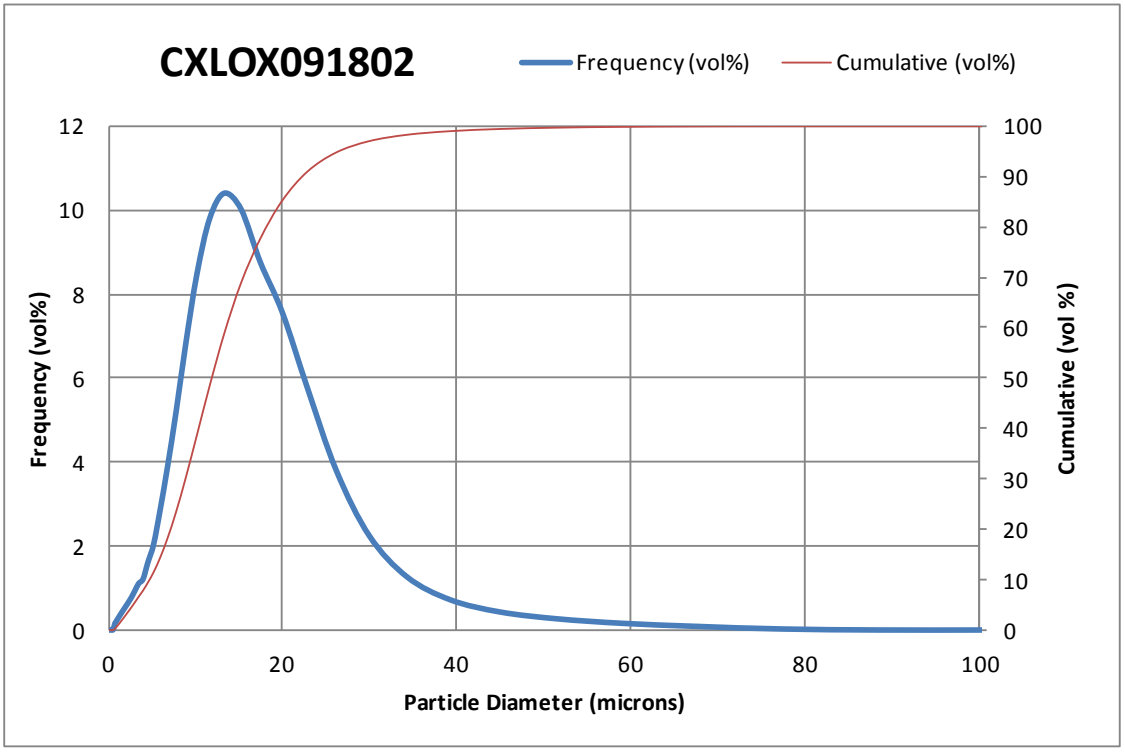

Figure A4-7. Particle size distribution for MIS sample CXLOX091802 (linear scale) by laser diffraction (950°C calcined).

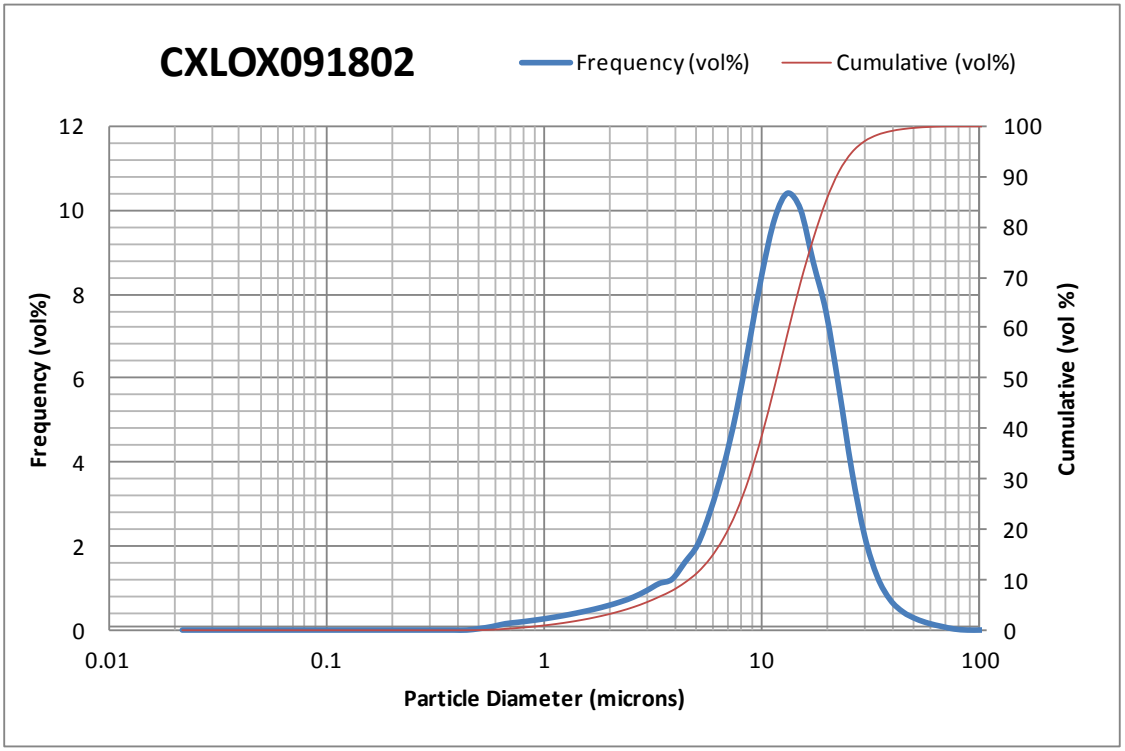

Figure A4-8. Particle size distribution for MIS sample CXLOX091802 (semi-log scale) by laser diffraction (950°C calcined).

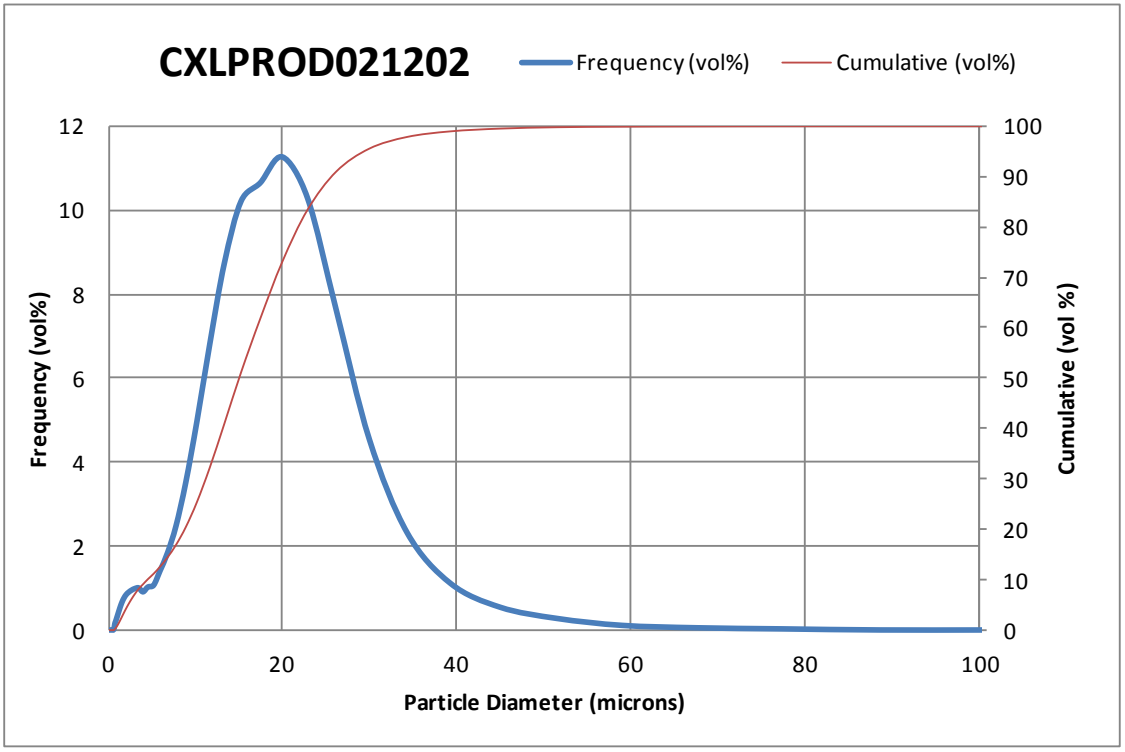

Figure A4-9. Particle size distribution for MIS sample CXPROD021202 (linear scale) by laser diffraction (950°C calcined).

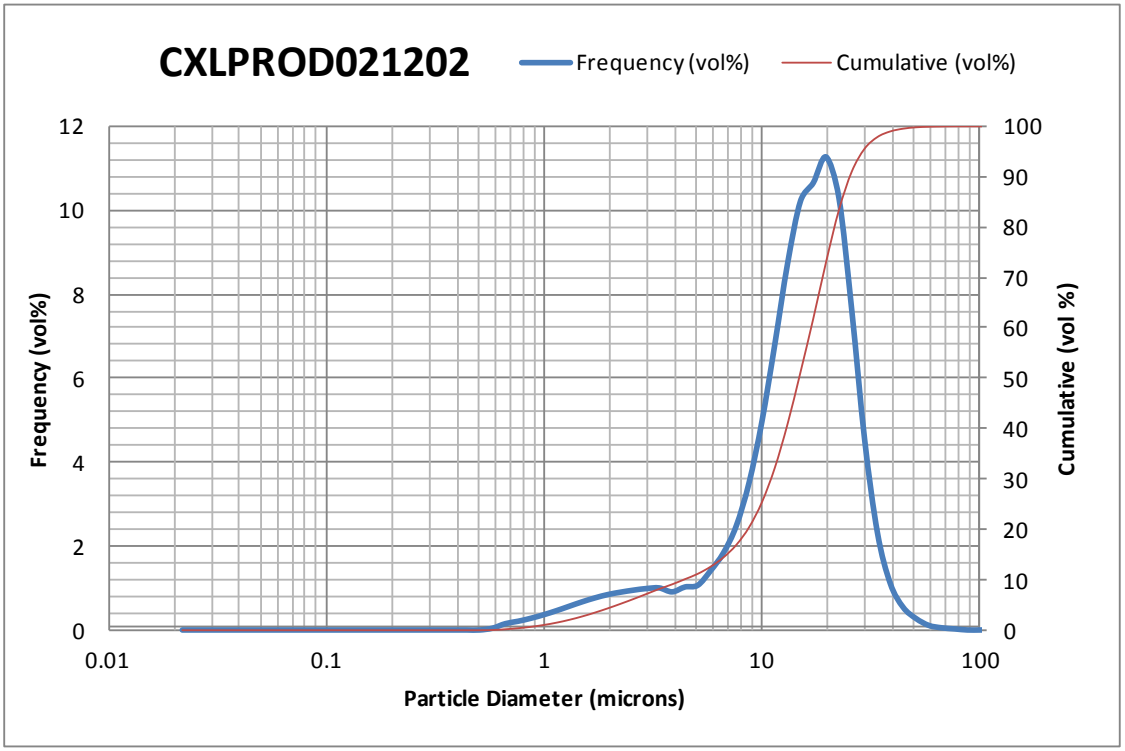

Figure A4-10. Particle size distribution for MIS sample CXPROD021202 (semi-log scale) by laser diffraction (950°C calcined).

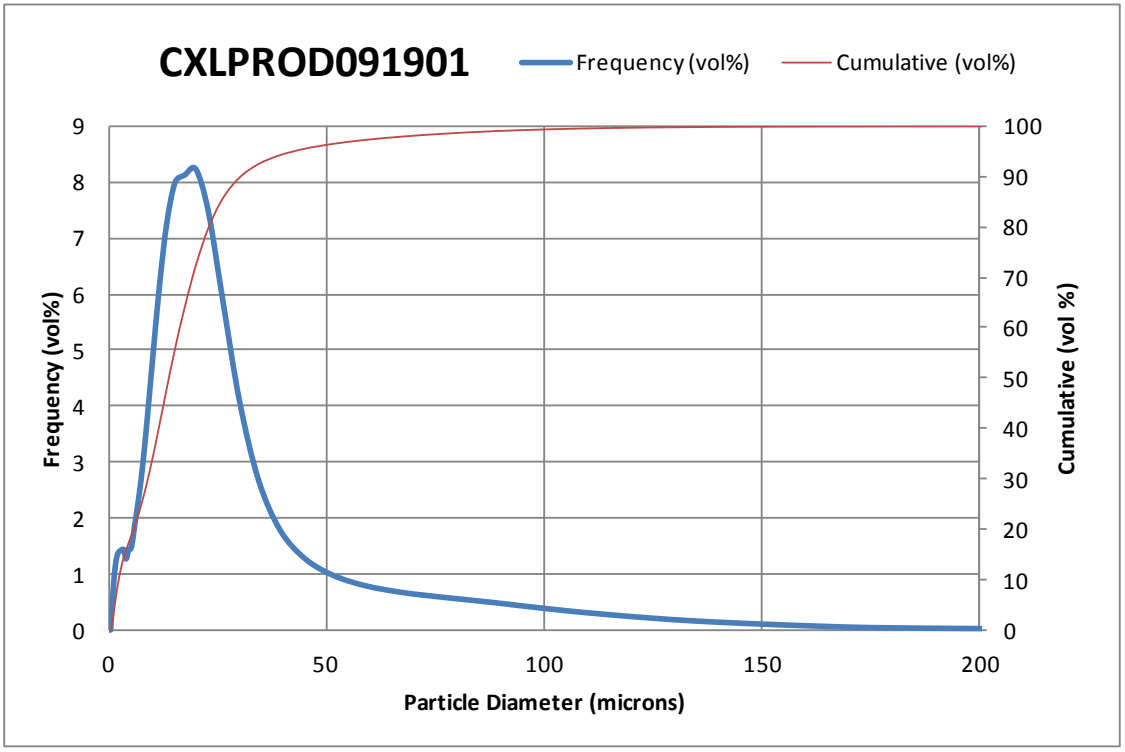

Figure A4-11. Particle size distribution for MIS sample CXPROD091901 (linear scale) by laser diffraction (950°C calcined).

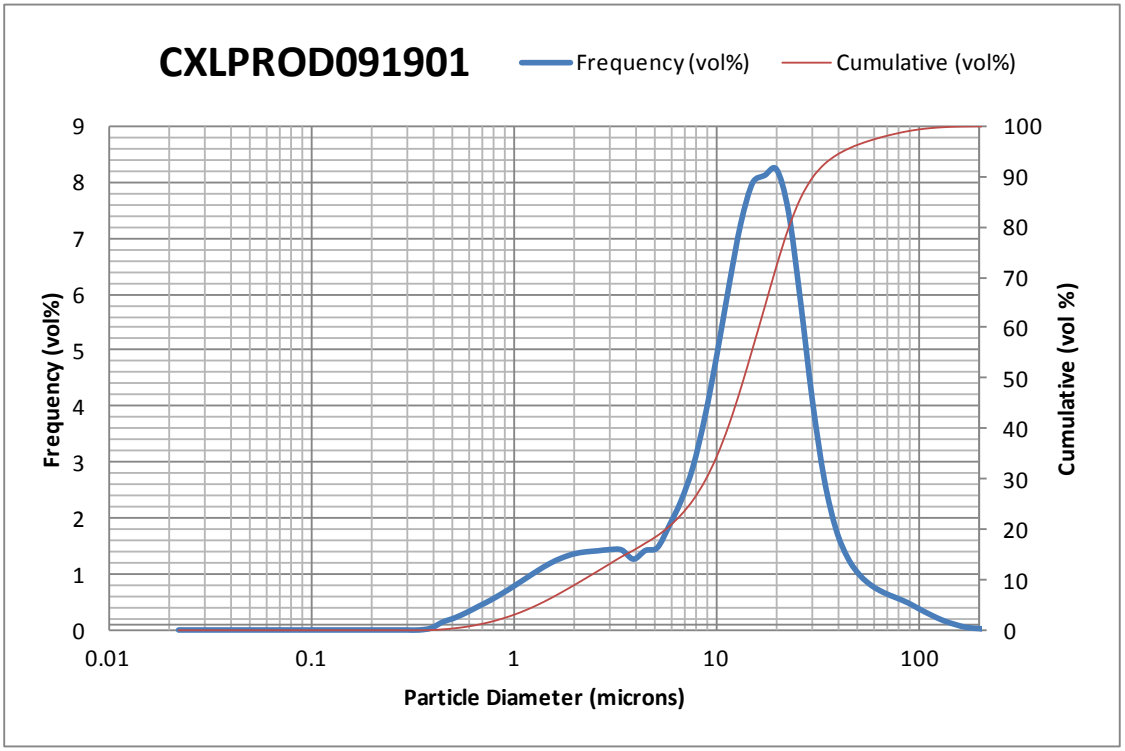

Figure A4-12. Particle size distribution for MIS sample CXPROD091901 (semi-log scale) by laser diffraction (950°C calcined).

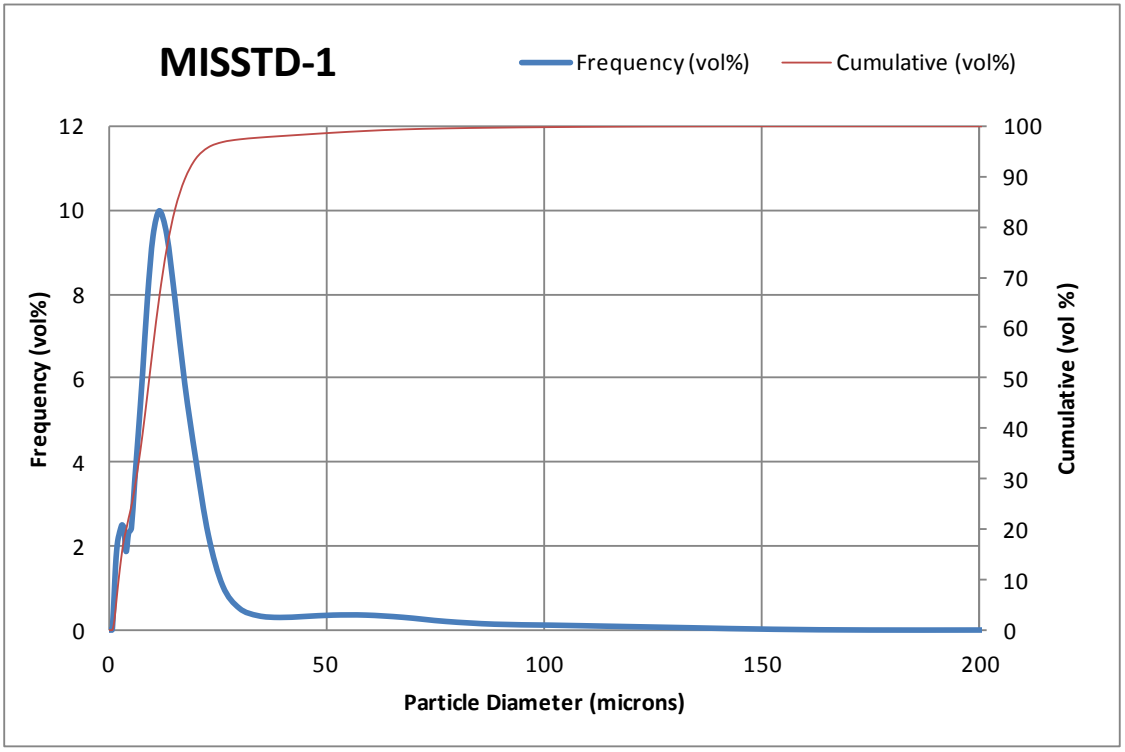

Figure A4-13. Particle size distribution for MIS sample MISSTD-1 (linear scale) by laser diffraction ("as-received").

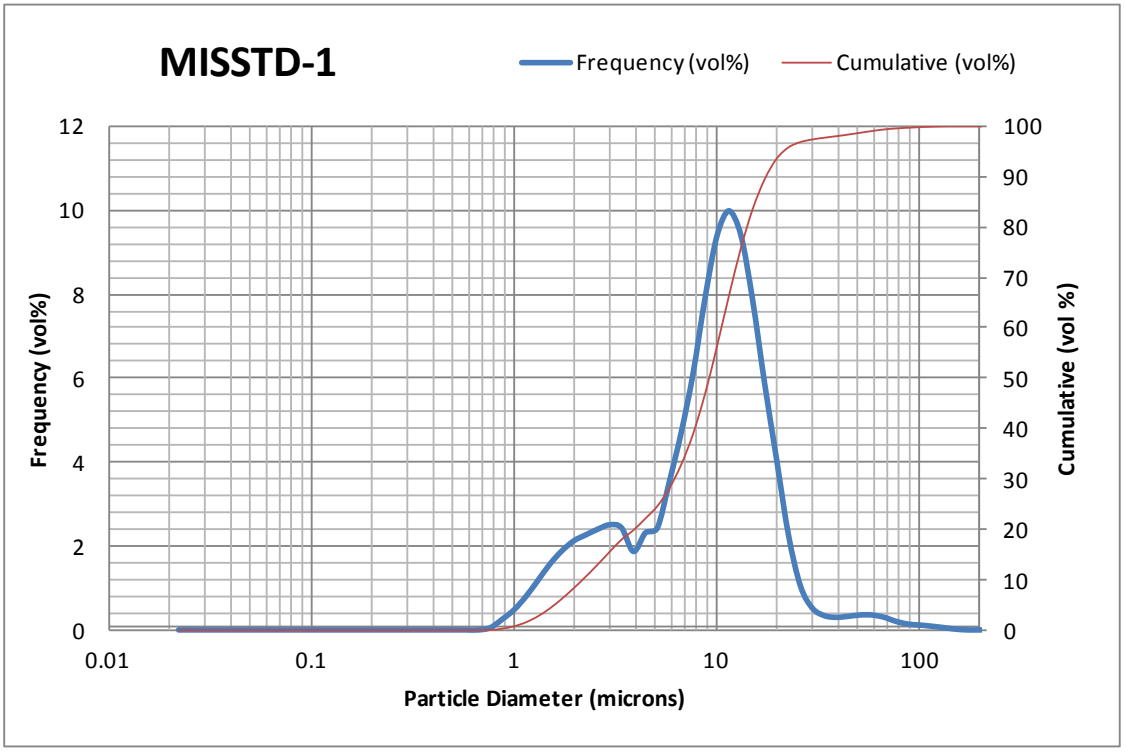

Figure A4-14. Particle size distribution for MIS sample MISSTD-1 (semi-log scale) by laser diffraction ("as-received").

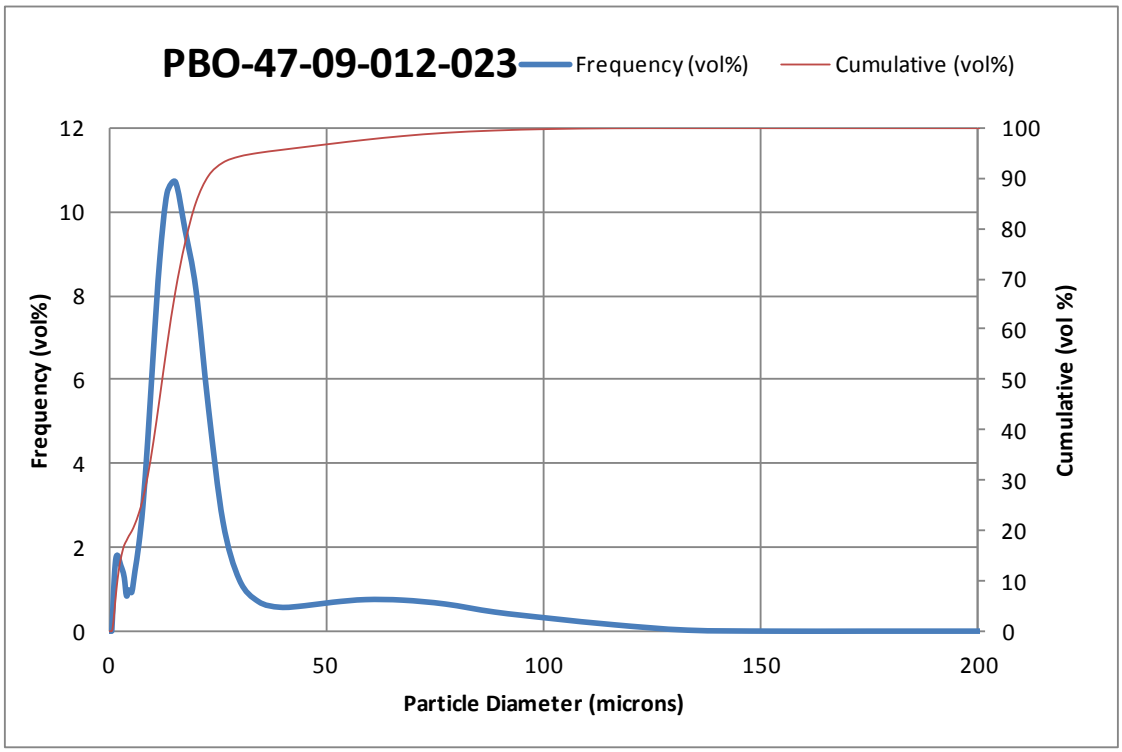

Figure A4-15. Particle size distribution for MIS sample PBO-47-09-012-023 (linear scale) by laser diffraction (950°C calcined).

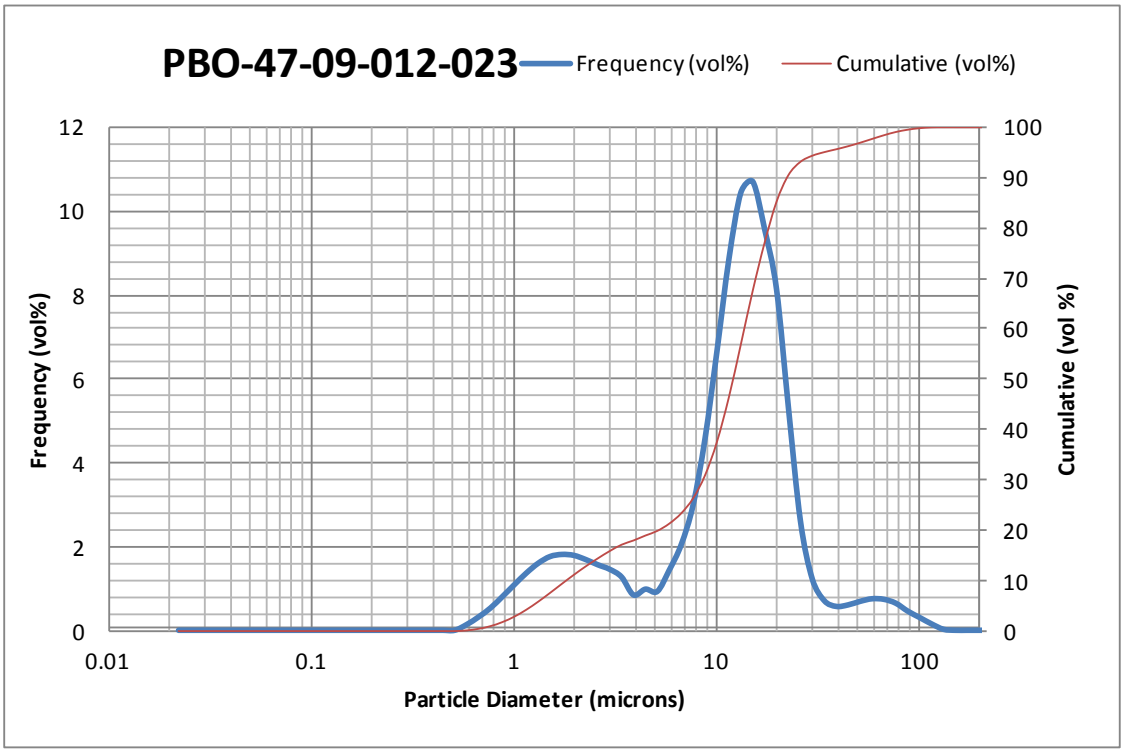

Figure A4-16. Particle size distribution for MIS sample PBO-47-09-012-023 (semi-log scale) by laser diffraction (950°C calcined).

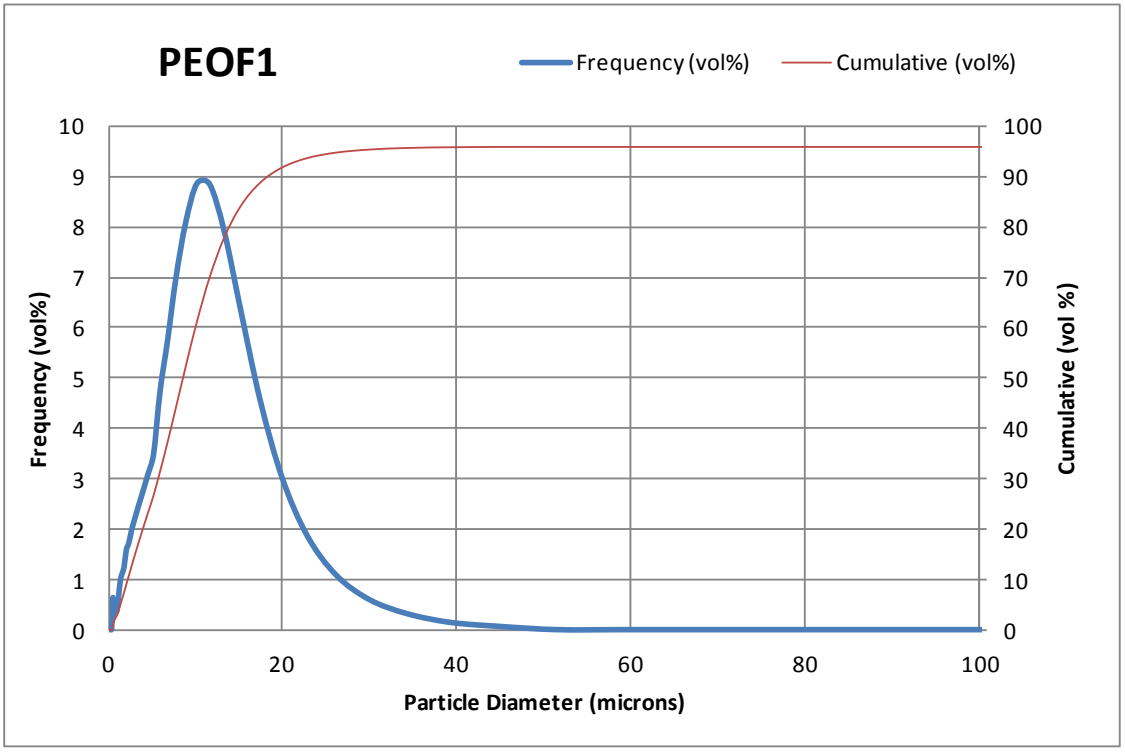

Figure A4-17. Particle size distribution for MIS sample PEOF1 (linear scale) by laser diffraction (950°C calcined).

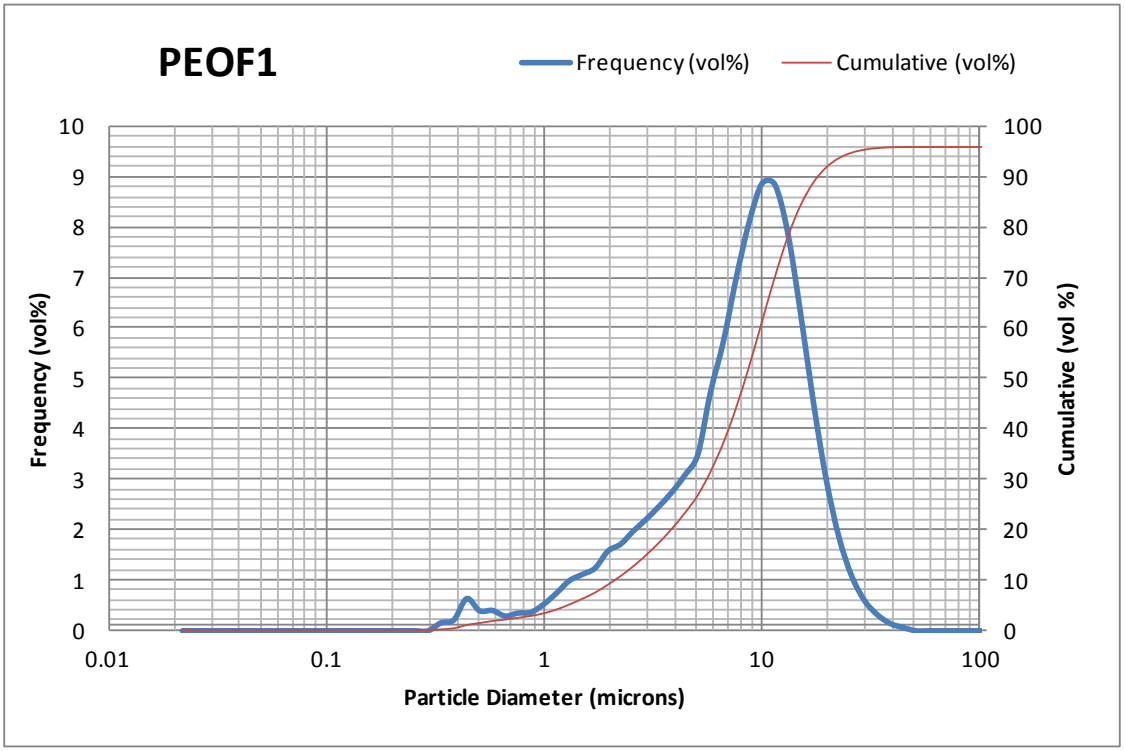

Figure A4-18. Particle size distribution for MIS sample PEOF1 (semi-log scale) by laser diffraction (950°C calcined).

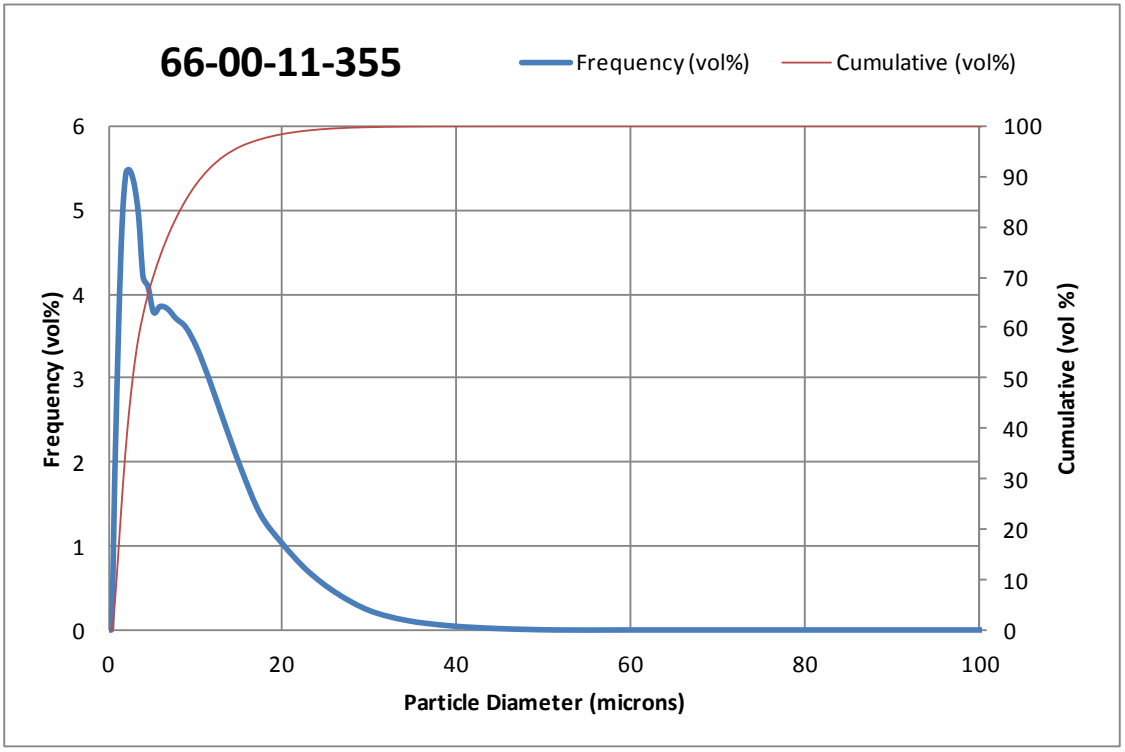

Figure A4-19. Particle size distribution for MIS sample 66-00-11-355 (linear scale) by laser diffraction (950°C calcined).

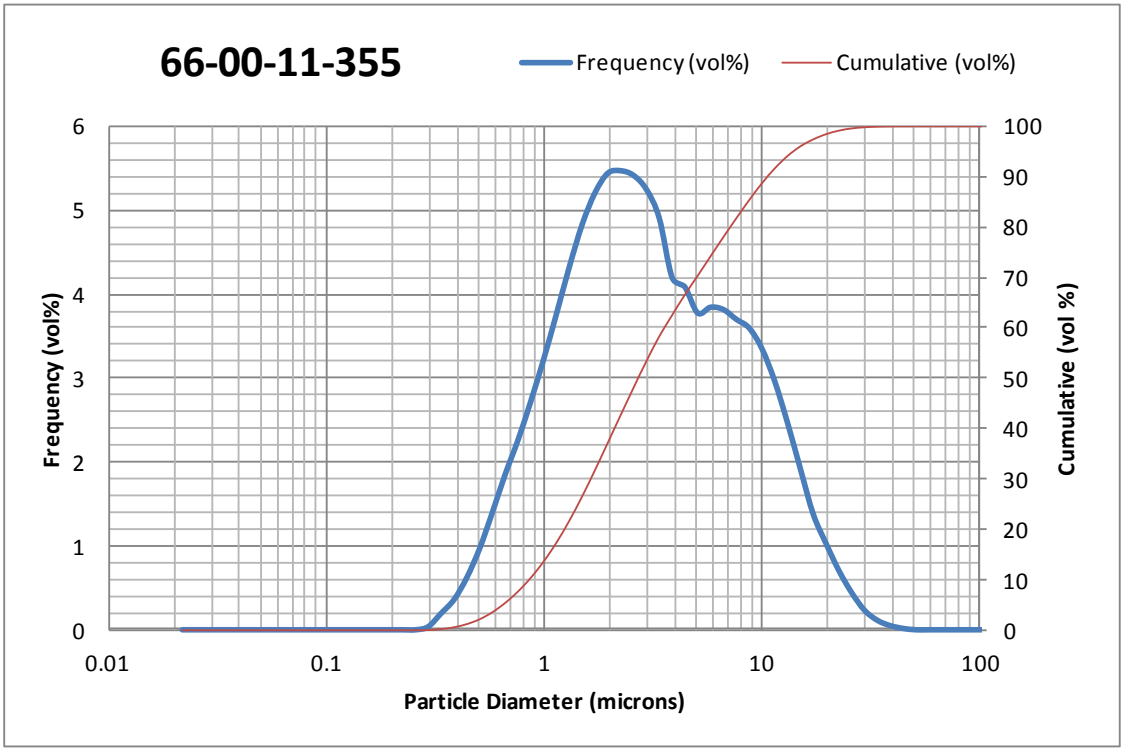

Figure A4-20. Particle size distribution for MIS sample 66-00-11-355 (semi-log scale) by laser diffraction (950°C calcined).

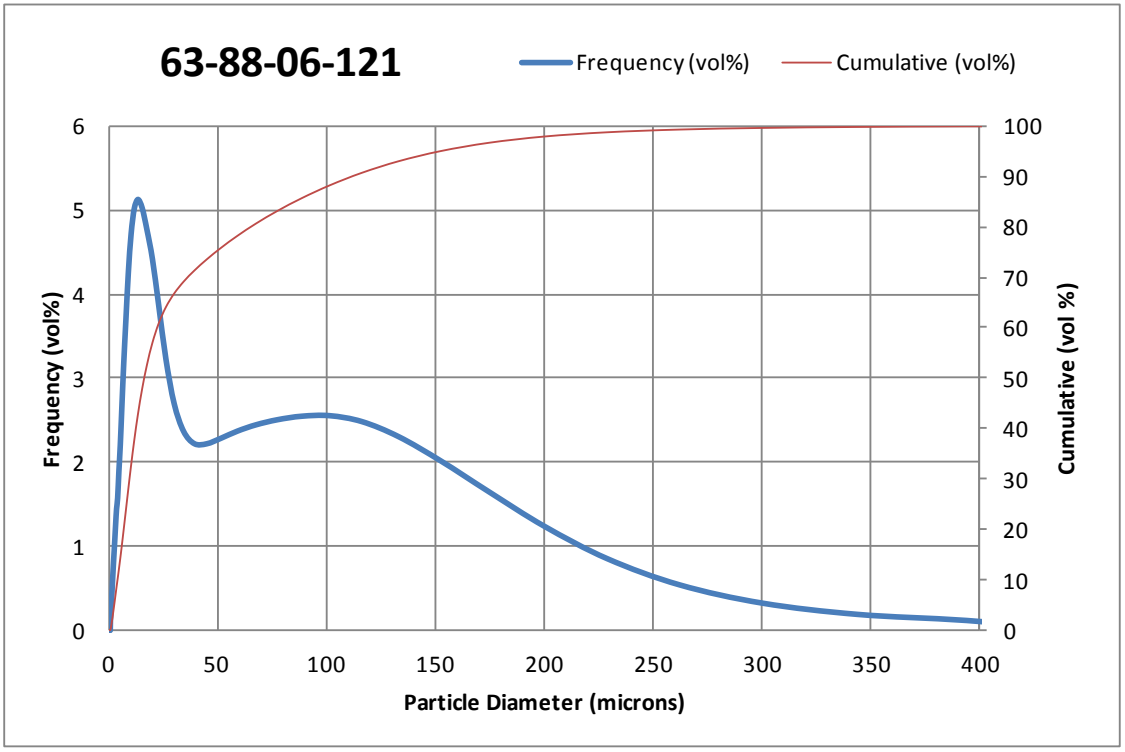

Figure A4-21. Particle size distribution for MIS sample 63-88-06-121 (linear scale) by laser diffraction ("as-received").

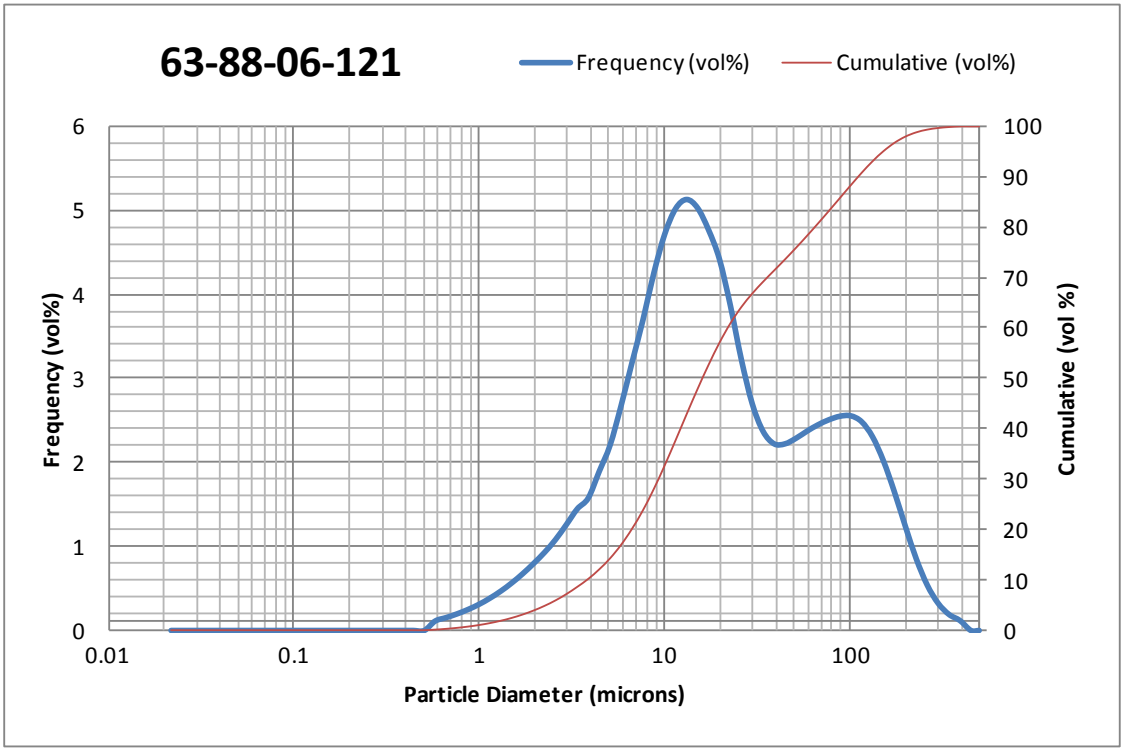

Figure A4-22. Particle size distribution for MIS sample 63-88-06-121 (semi-log scale) by laser diffraction ("as-received").

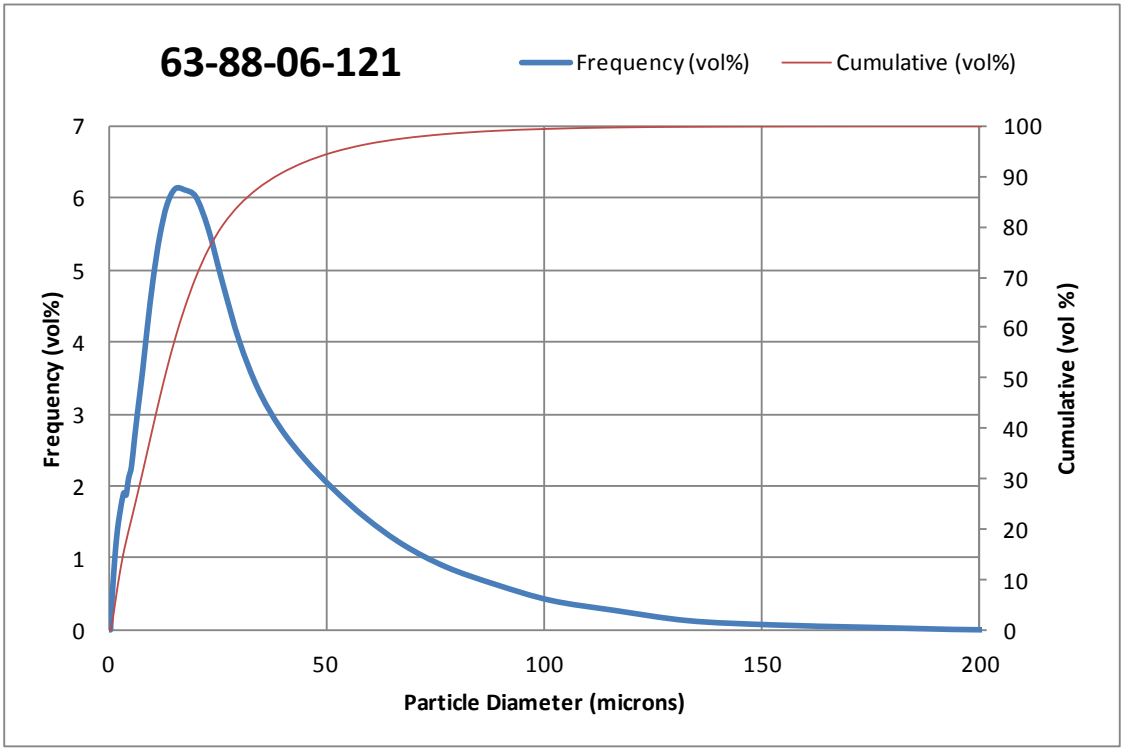

Figure A4-23. Particle size distribution for MIS sample 63-88-06-121 (linear scale) by laser diffraction (950°C calcined).

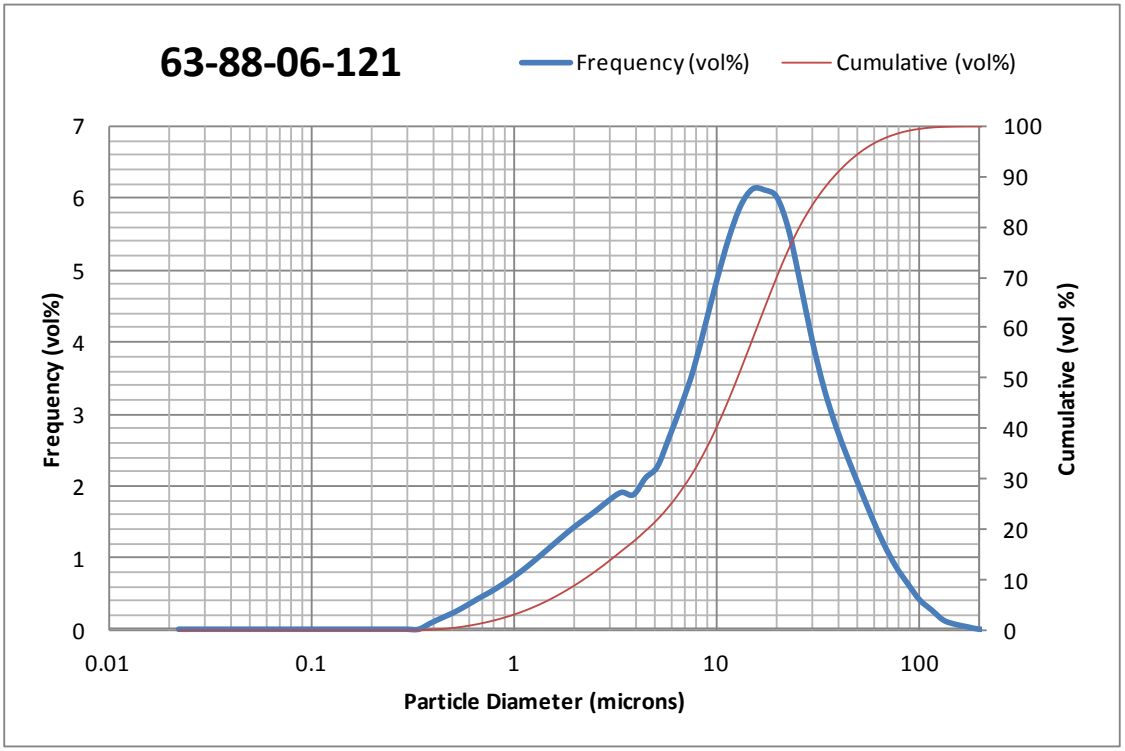

Figure A4-24. Particle size distribution for MIS sample 63-88-06-121 (semi-log scale) by laser diffraction (950°C calcined).

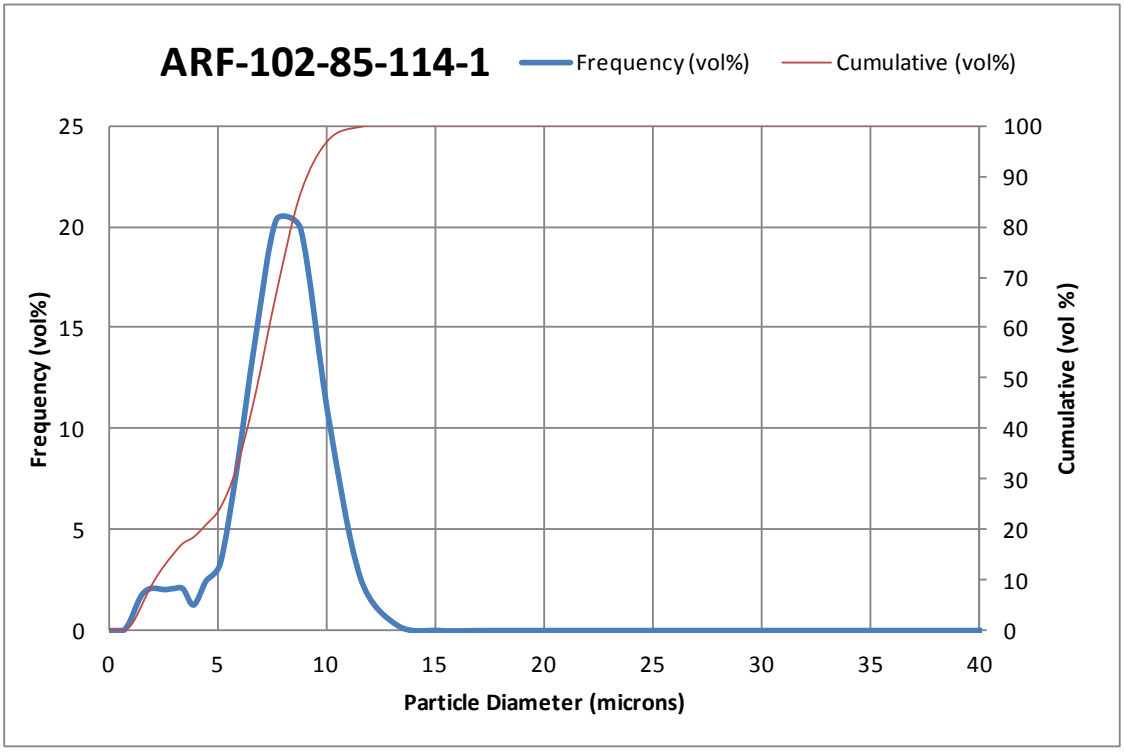

Figure A4-25. Particle size distribution for MIS sample ARF-102-85-114-1 (linear scale) by laser diffraction (950°C calcined).

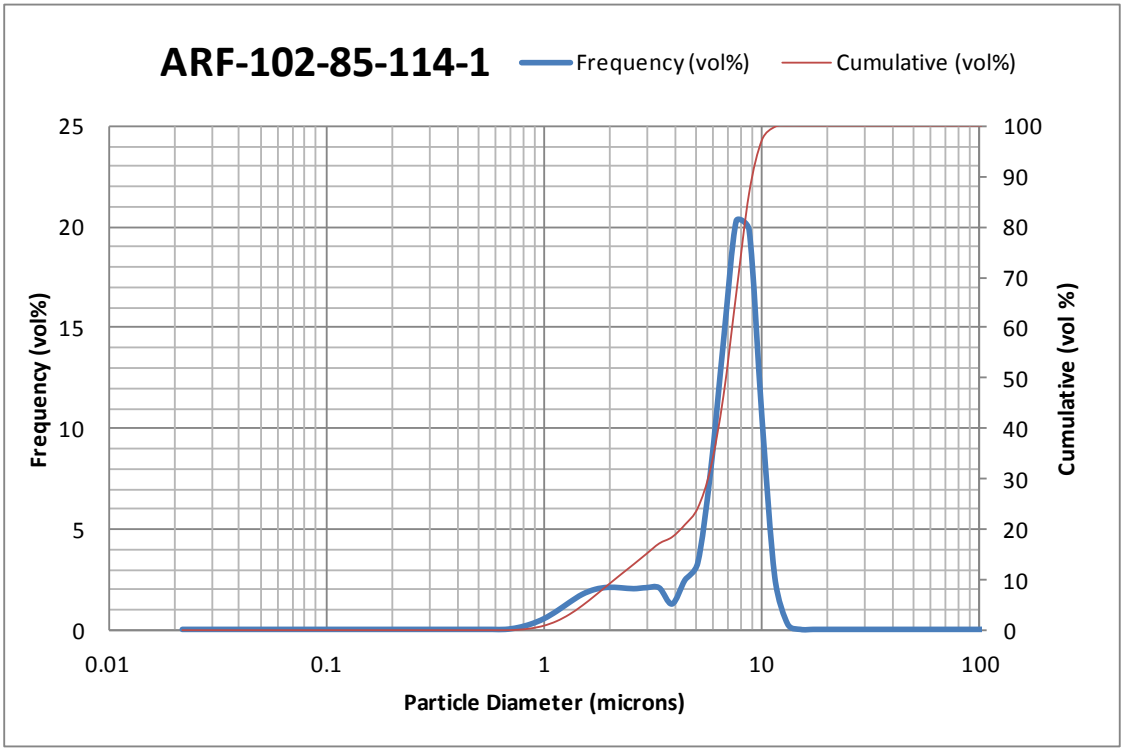

Figure A4-26. Particle size distribution for MIS sample ARF-102-85-114-1 (semi-log scale) by laser diffraction (950°C calcined).

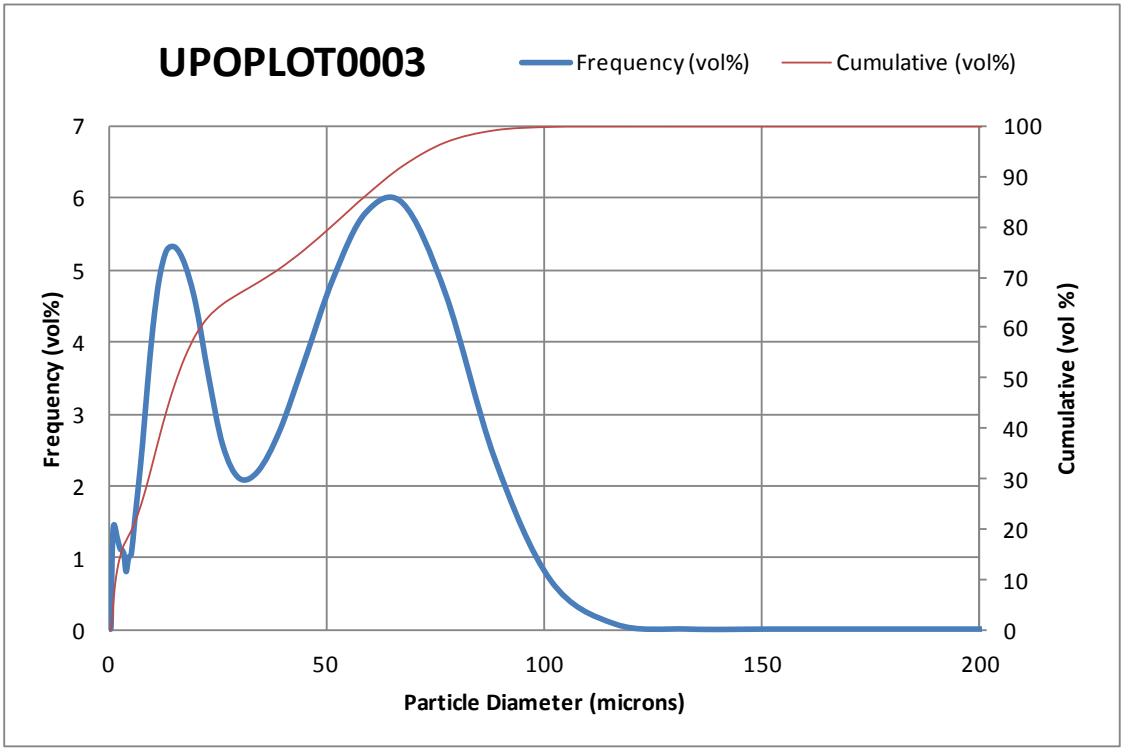

Figure A4-27. Particle size distribution for MIS sample UPOPLOT0003 (linear scale) by laser diffraction (950°C calcined).

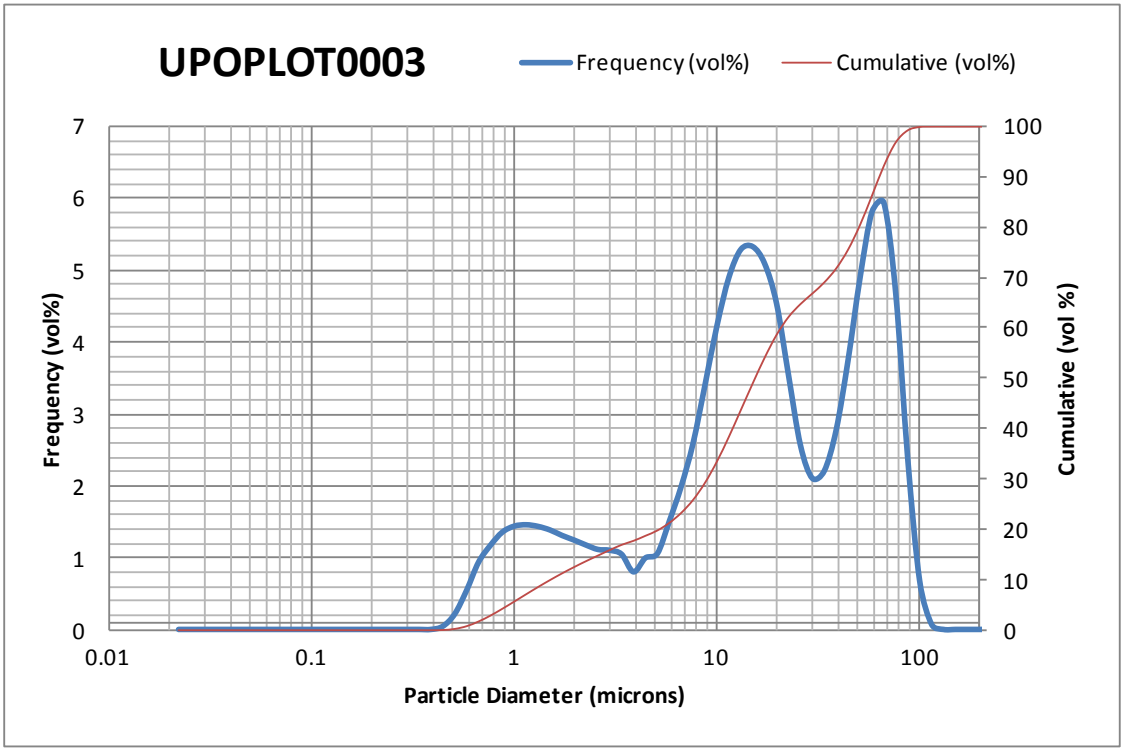

Figure A4-28. Particle size distribution for MIS sample UPOPLOT0003 (semi-log scale) by laser diffraction (950°C calcined).

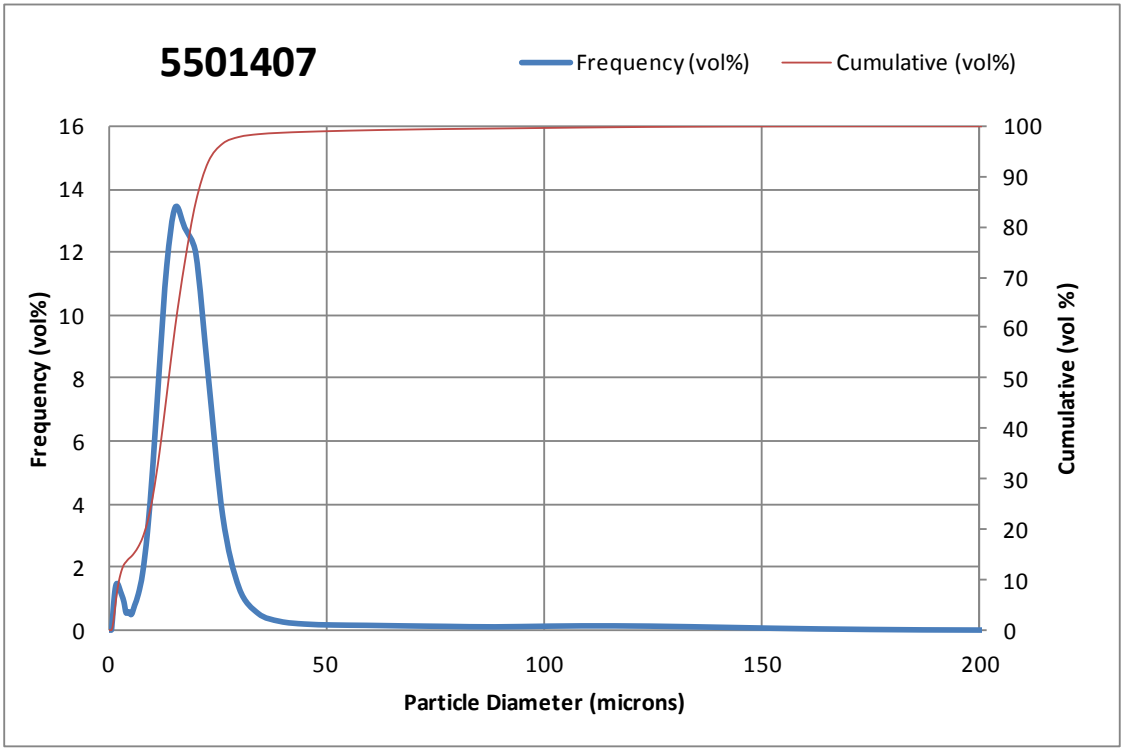

Figure A4-29. Particle size distribution for MIS sample 5501407 (linear scale) by laser diffraction (950°C calcined).

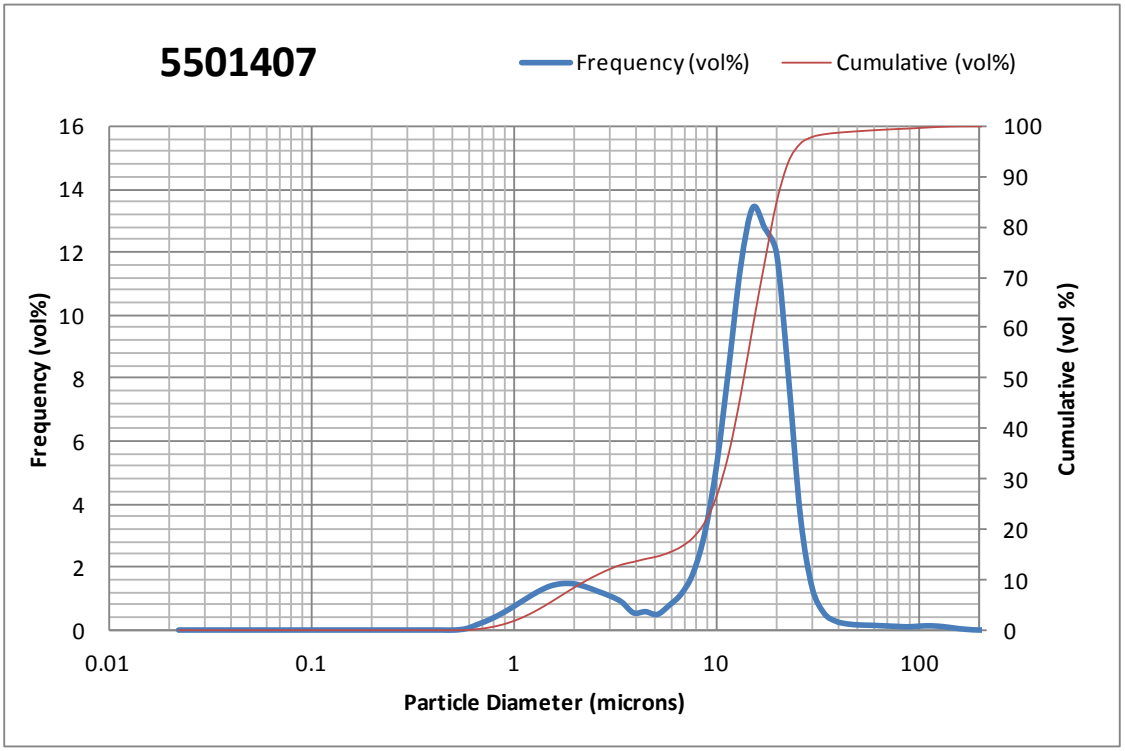

Figure A4-30. Particle size distribution for MIS sample 5501407 (semi-log scale) by laser diffraction (950°C calcined).

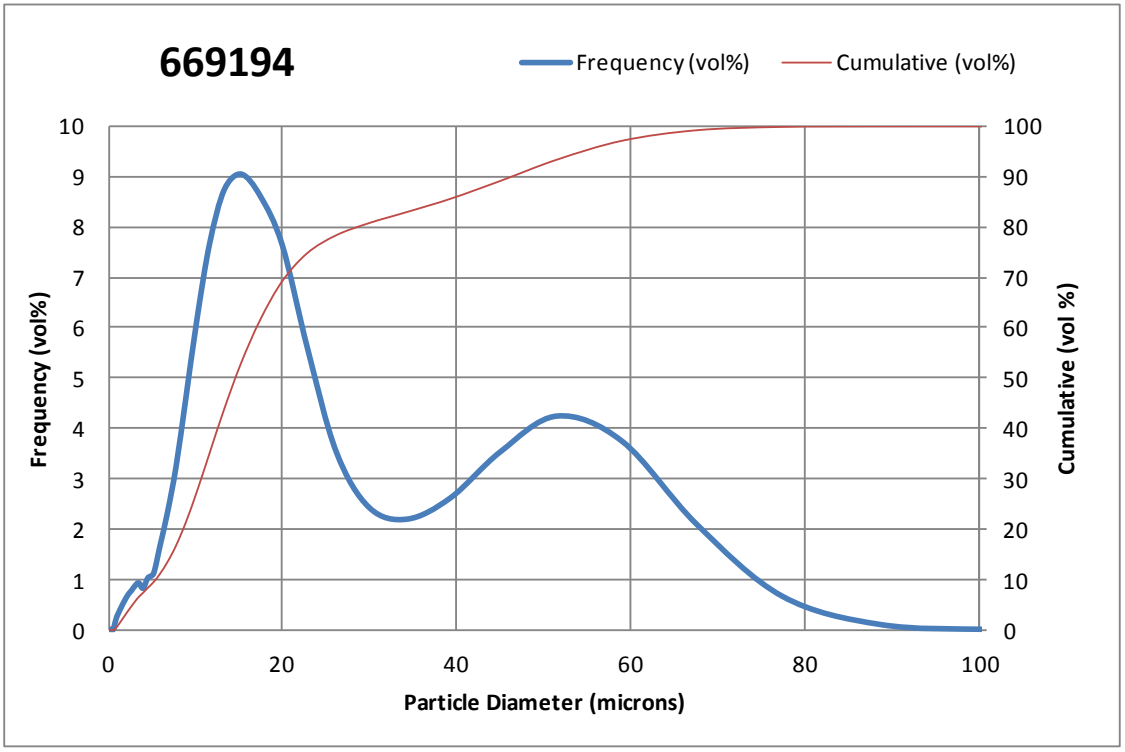

Figure A4-31. Particle size distribution for MIS sample 669194 (linear scale) by laser diffraction (950°C calcined).

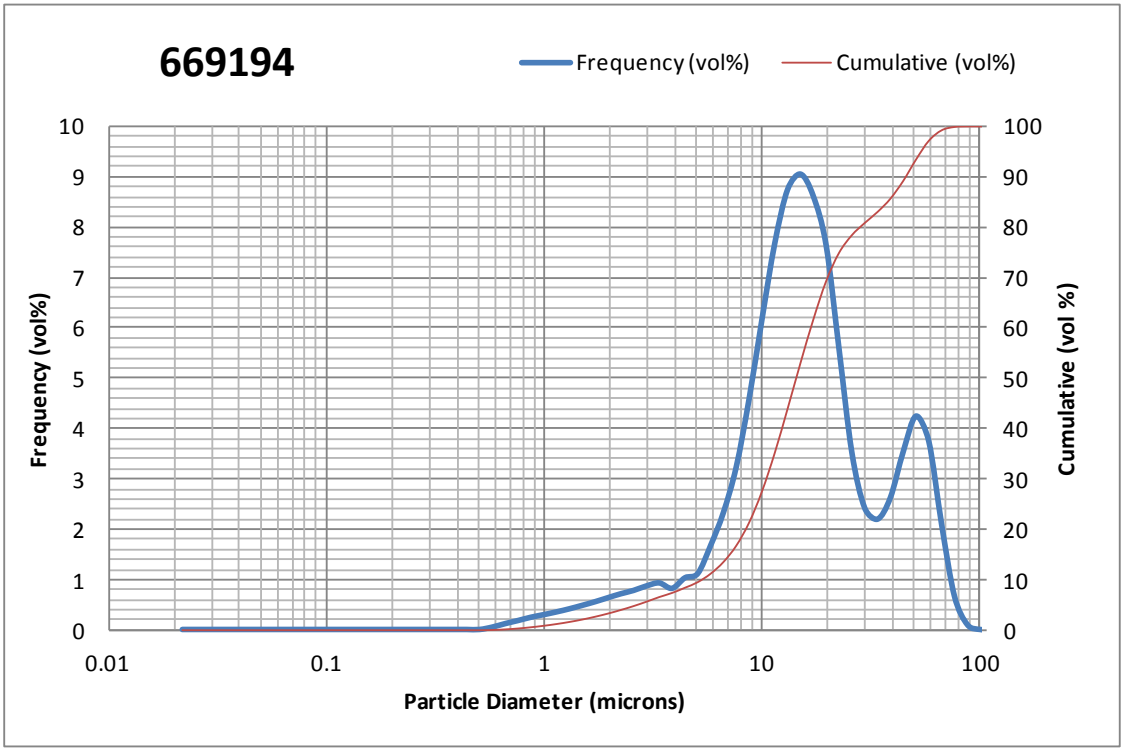

Figure A4-32. Particle size distribution for MIS sample 669194 (semi-log scale) by laser diffraction (950°C calcined).

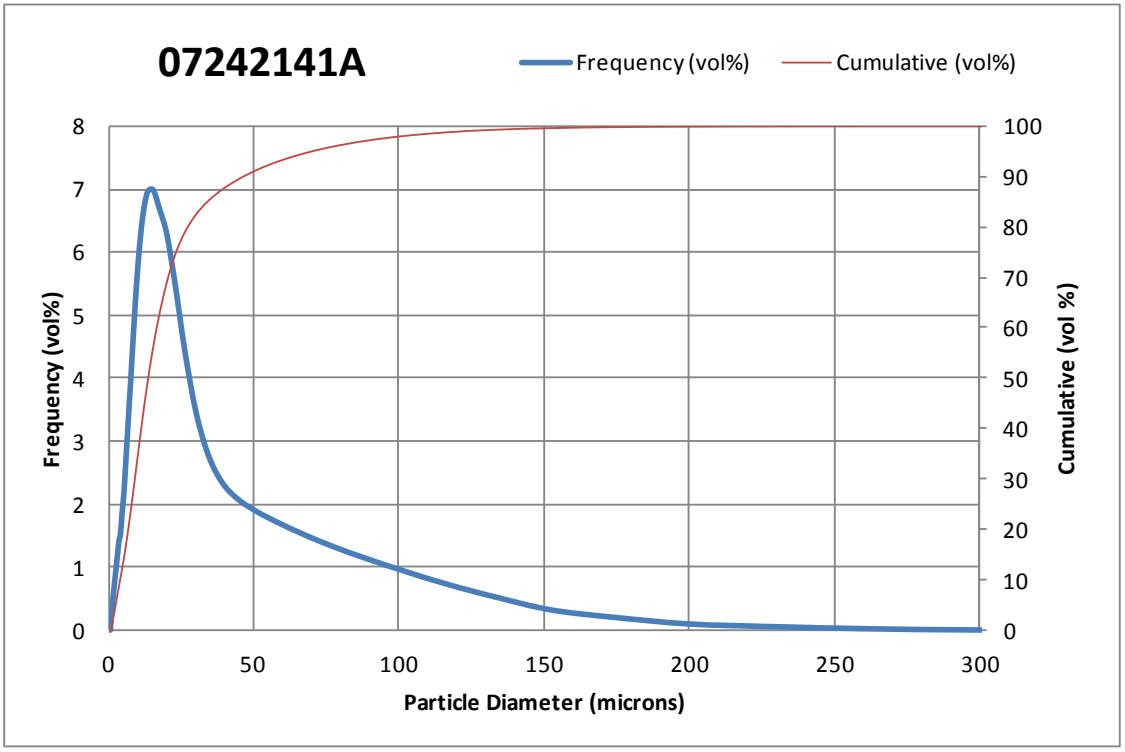

Figure A4-33. Particle size distribution for MIS sample 07242141A (linear scale) by laser diffraction (950°C calcined).

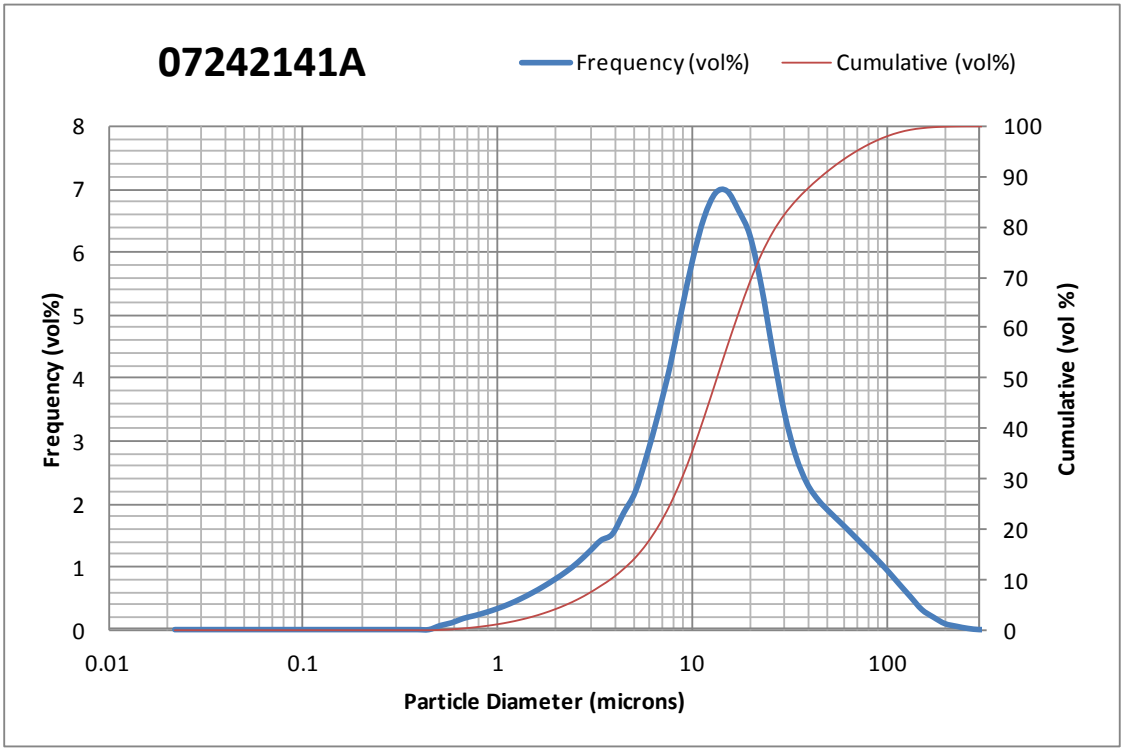

Figure A4-34. Particle size distribution for MIS sample 07242141A (semi-log scale) by laser diffraction (950°C calcined).

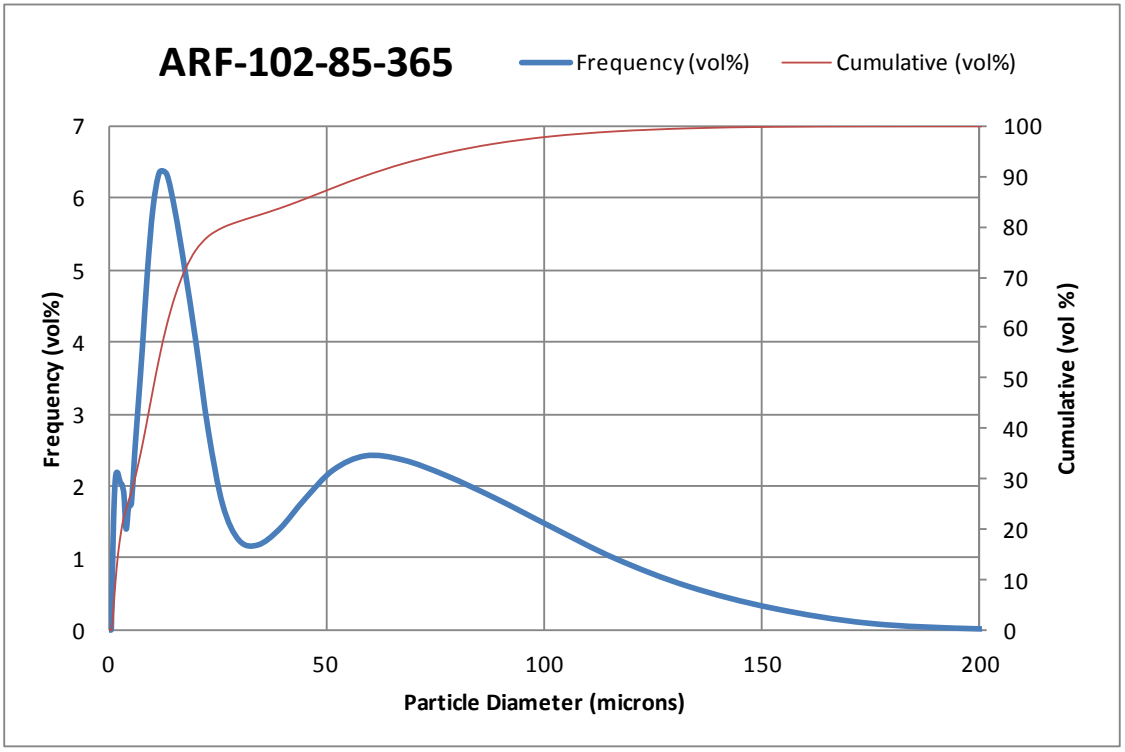

Figure A4-35. Particle size distribution for MIS sample ARF-102-85-365 (linear scale) by laser diffraction (950°C calcined).

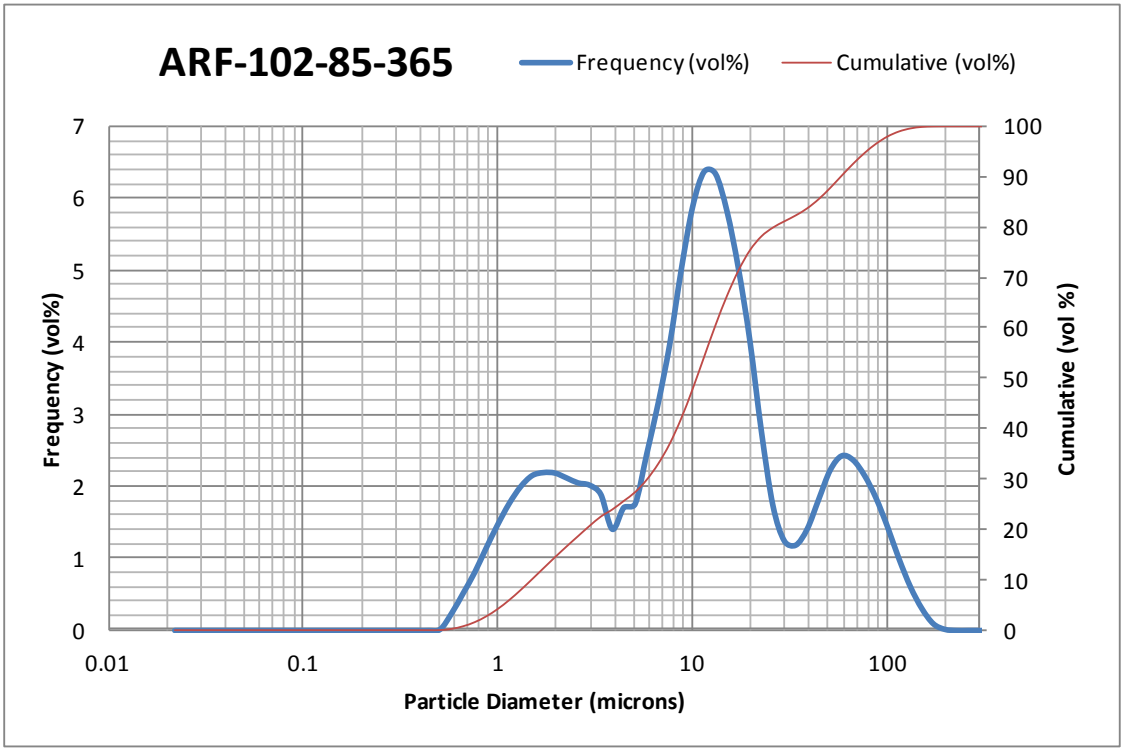

Figure A4-36. Particle size distribution for MIS sample ARF-102-85-365 (semi-log scale) by laser diffraction (950°C calcined).

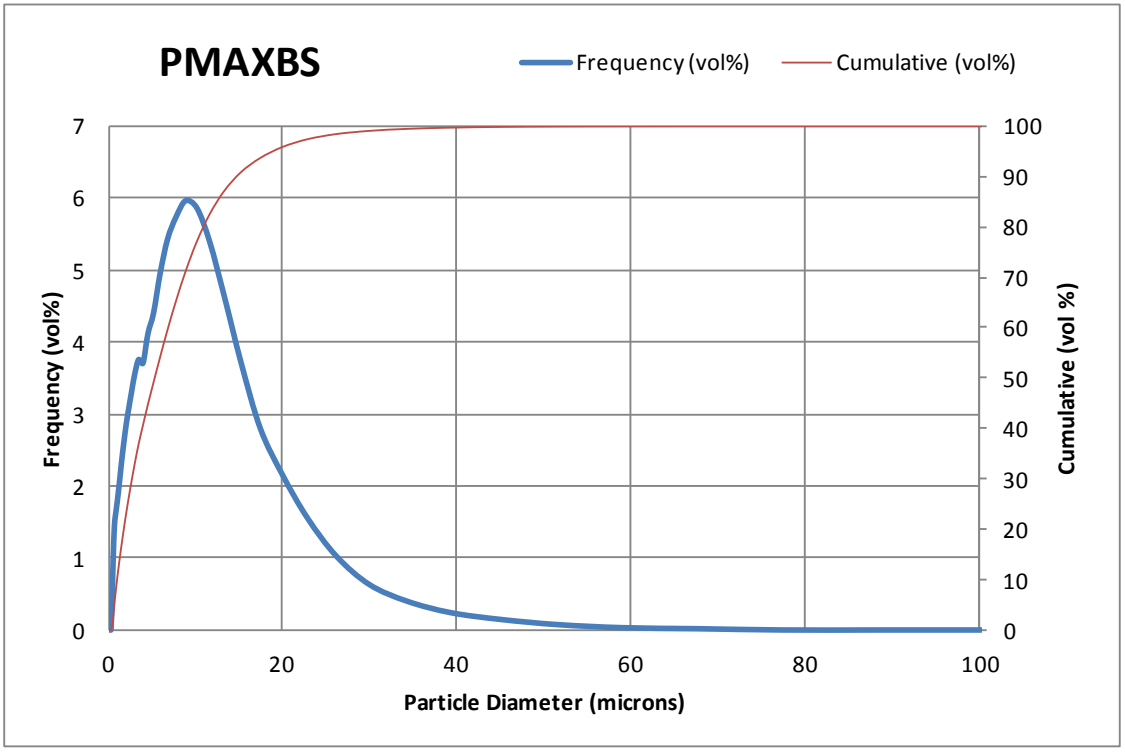

Figure A4-37. Particle size distribution for MIS sample PMAXBS (linear scale) by laser diffraction (800°C calcined).

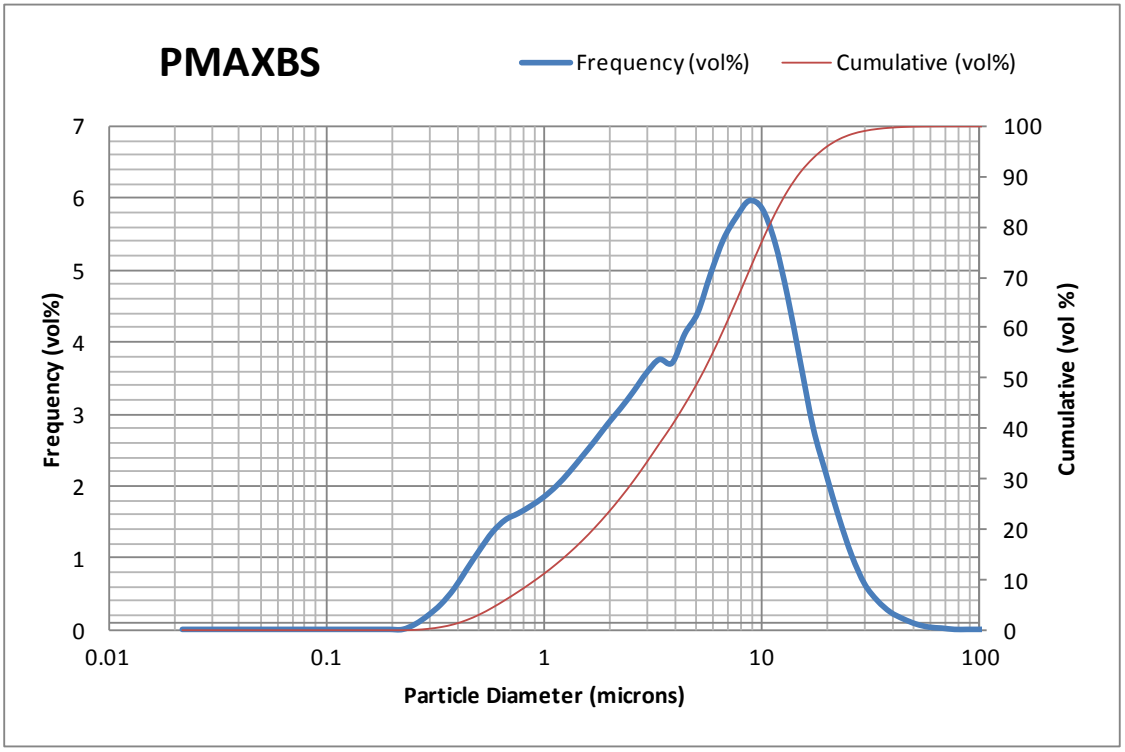

Figure A4-38. Particle size distribution for MIS sample PMAXBS (semi-log scale) by laser diffraction (800°C calcined).

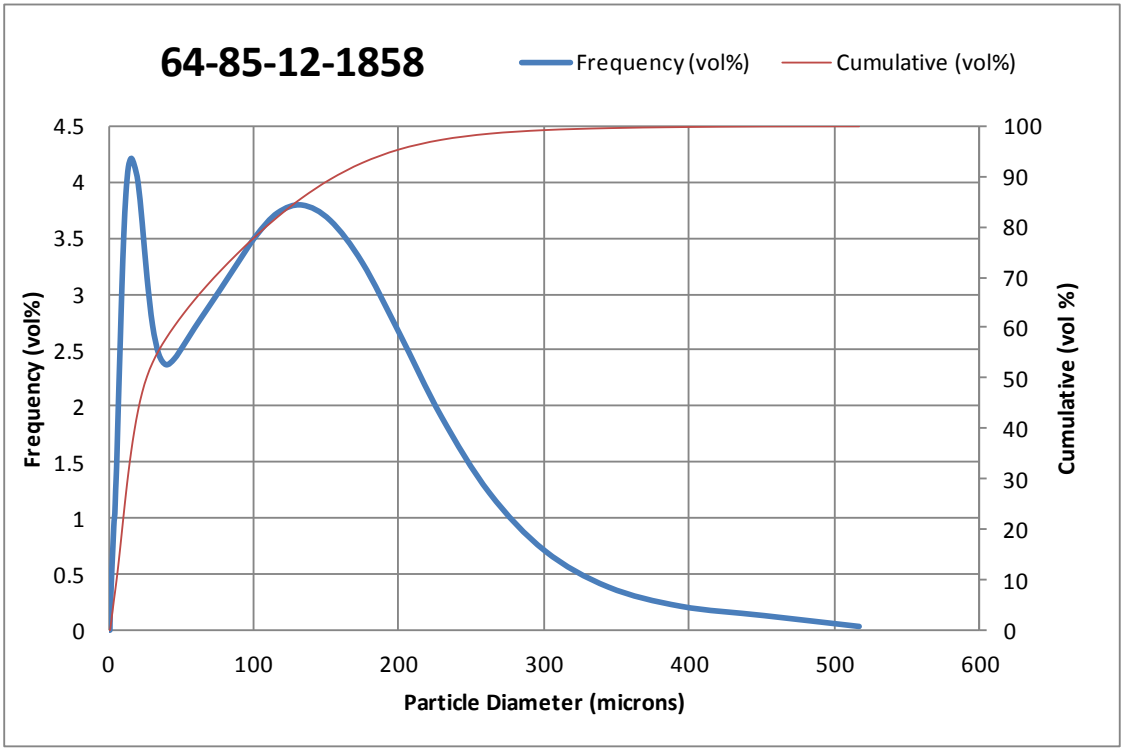

Figure A4-39. Particle size distribution for MIS sample 64-85-12-1858 (linear scale) by laser diffraction ("as-received").

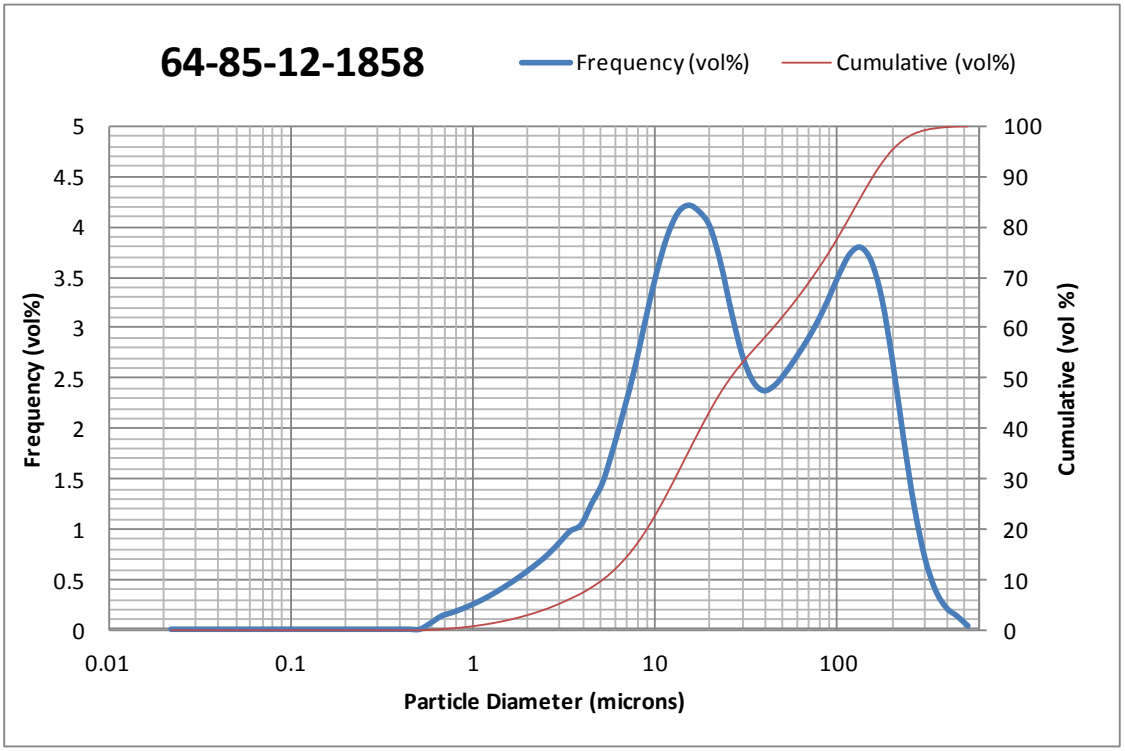

Figure A4-40. Particle size distribution for MIS sample 64-85-12-1858 (semi-log scale) by laser diffraction ("as-received").

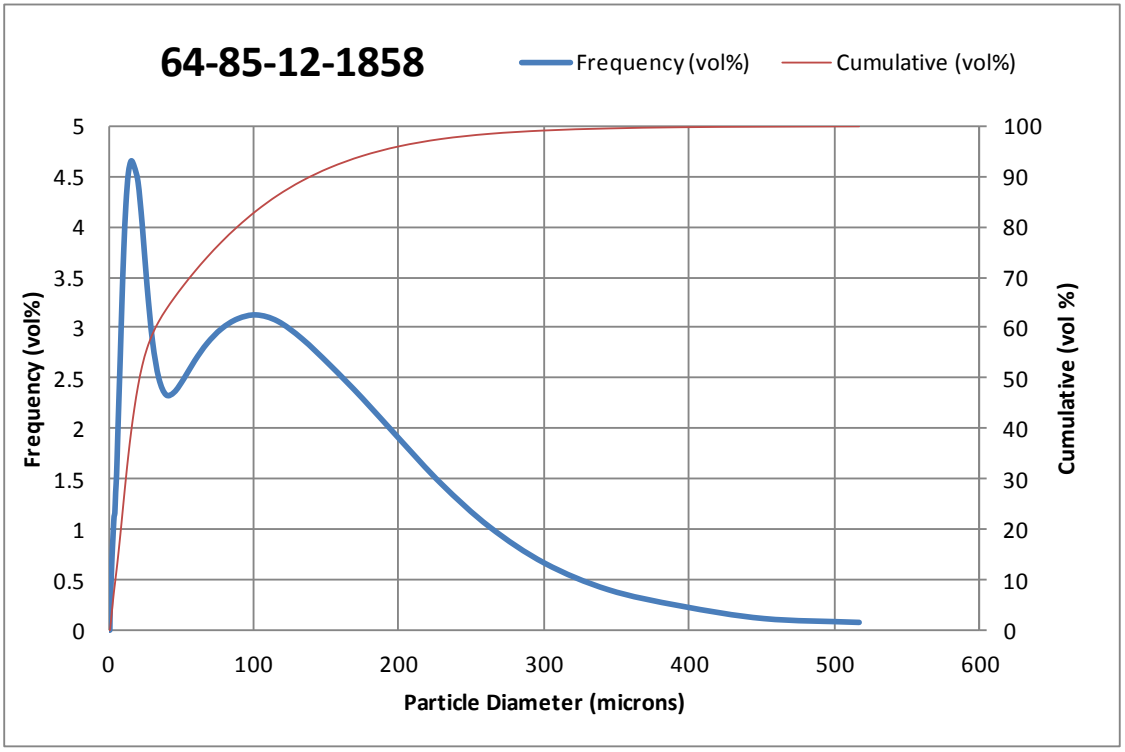

Figure A4-41. Particle size distribution for MIS sample 64-85-12-1858 (linear scale) by laser diffraction (950°C calcined).

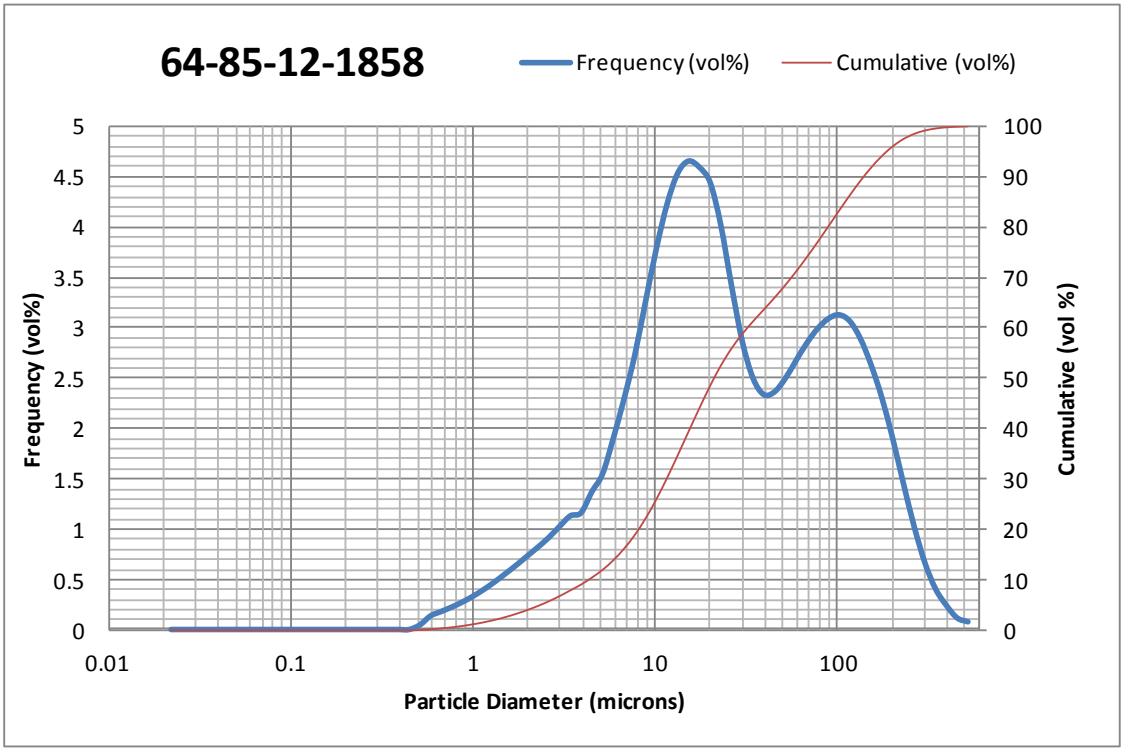

Figure A4-42. Particle size distribution for MIS sample 64-85-12-1858 (semi-log scale) by laser diffraction (950°C calcined).

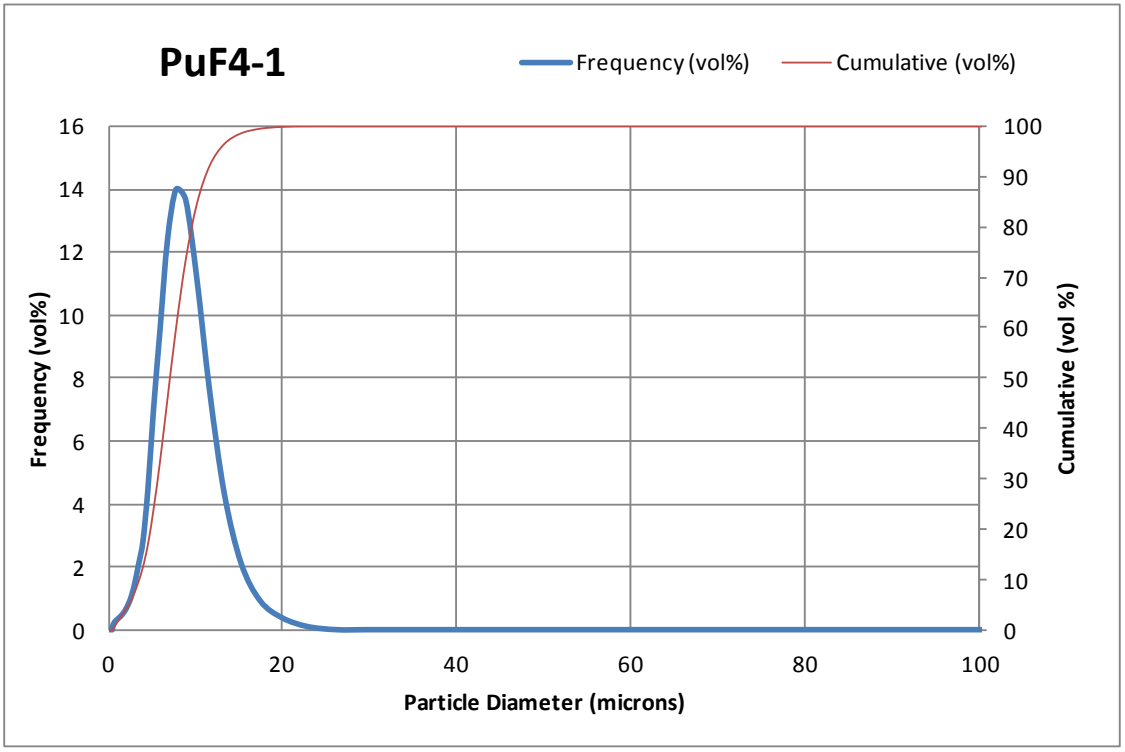

Figure A4-43. Particle size distribution for MIS sample PuF4-1 (linear scale) by laser diffraction ("as-received").

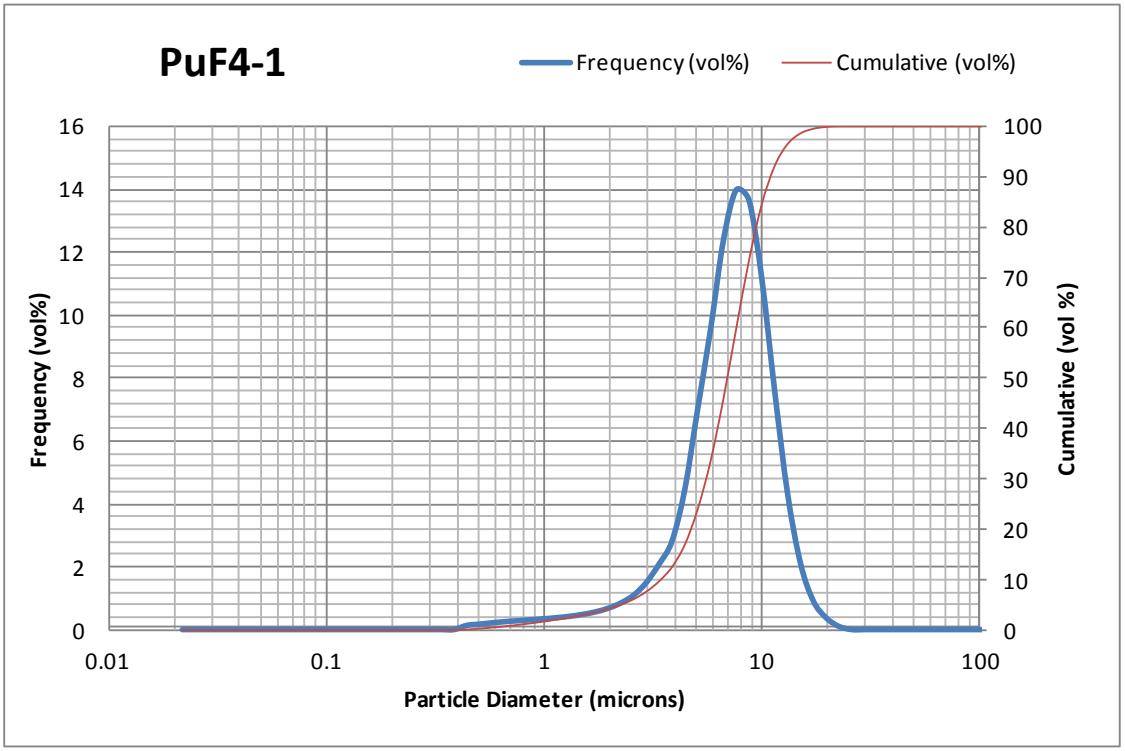

Figure A4-44. Particle size distribution for MIS sample PuF4-1 (semi-log scale) by laser diffraction ("as-received").

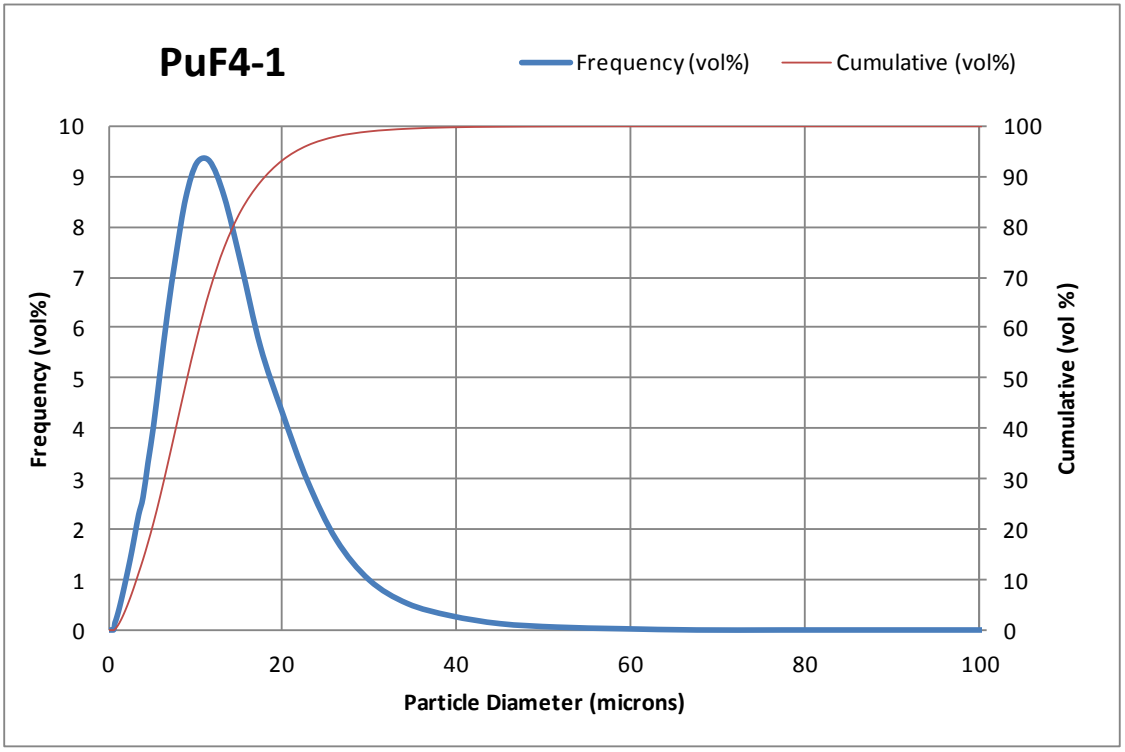

Figure A4-45. Particle size distribution for MIS sample PuF4-1 (linear scale) by laser diffraction (950°C calcined).

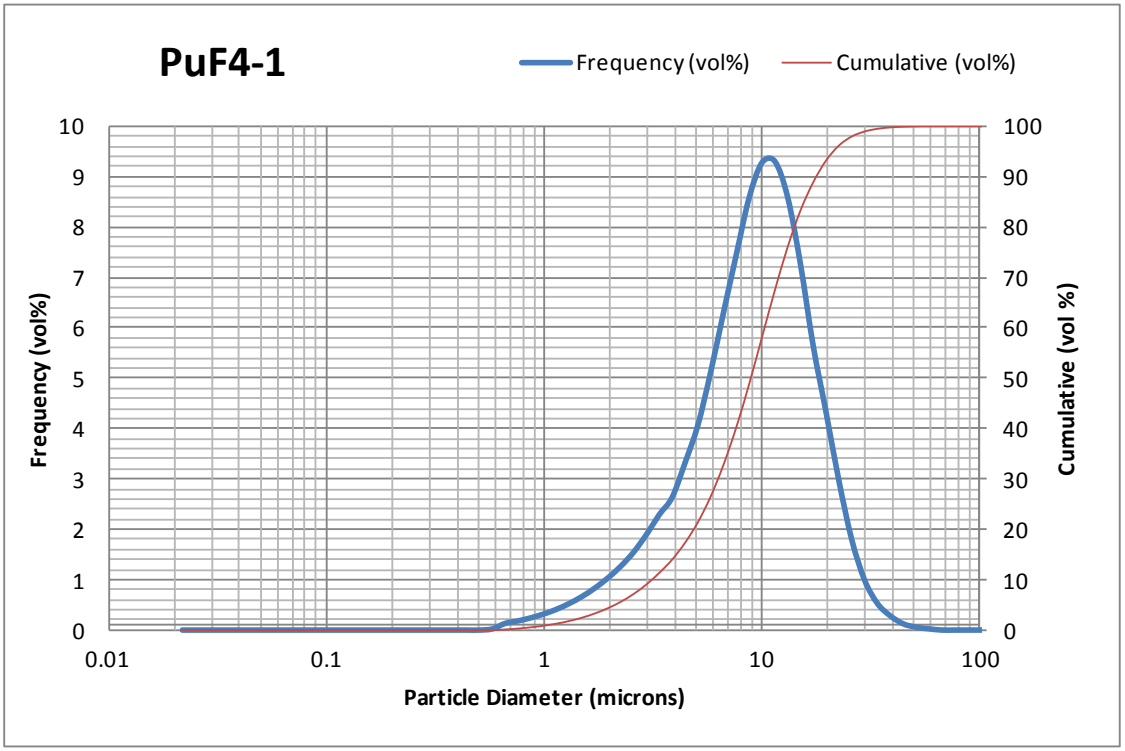

Figure A4-46. Particle size distribution for MIS sample PuF4-1 (semi-log scale) by laser diffraction (950°C calcined).

Appendix 5, Surface Area Data

Table A5-1. Summary of Surface Area Data Averaged by Process Category for MIS Samples in the AR and 950°C Conditions

Process Category	Process Subcategory	Number of Items	Average Surface Area (m^2/g) AR	Average Surface Area (m^2/g) 950°C (Cox 95% LCL,UCL)	Median Surface Area (m^2/g) 950°C (95% Cox LCL,UCL)	Average % Reduction -Due to 950°C calcination (95% Cox LCL,UCL)
Aqueous Processing	Byproduct	4-7	19.2	2.7 (1.1, 8.4)	2.7 (0.6, 4.7)	72% (46,91)
	Nitrate Product	1-4	17.8	2.0		
	Product	7-10	11.5	2.7 (1.6,4.8)	2.3 (1.2,3.6)	62% (32,89)
Metal Oxidation	Byproduct	2	4.6	1.3	NA	68%
	Product	4-5	4.7	1.7 (0.5,15.7)	2.3 (0.2,6.0)	55% (47,63)
Misc.	Misc	2-3	13.1	1.3	NA	73%
Mixed Actinide	Byproduct	7	3.1	0.6 (0.3,1.1)	0.5 (0.2,0.9)	70% (44,94)
	Product	1-3	11.2	0.6	0.6	90%
Molten Salt	Byproduct	8-9	5.3	1.0 (0.6,2.3)	0.7 (0.4,1.5)	67% (45,89)

Notes:

% Reduction is the average of the % reductions for each item = $(1 - SA_{950}/SA_{AR})$

For several subcategories the LCL and UCL were not calculated due to small number of data points for the process category/subcategory. Because there were outliers in the data, the median value was also calculated for comparison.

Table A5-2. Representative Samples: Surface Area Data Arranged by Process Category for All Conditions

Process Category	Process Subcategory	MIS Sample	Surface Area (m^2/g) AR	Surface Area (m^2/g) 600°C	Surface Area (m^2/g) 950°C	% Reduction Due to 950°C calcination
Aqueous Processing	Byproduct	07032282A	7.0	6.9	2.9	58%
		07242165A	1.5		0.4	71%
		07242201A (chunks)			18.9	
		07242201A (powder)	9.5		2.7	71%
		39-01153A	72.8		8.8	
		63-88-06-121	5.4		0.6	89%
		66-00-11-355	3.3		2.9	
		66-01-01-439	3.9			
		ARF-102-85-355	50.1	3.2	0.9	
	Product	07161856	7.2		1.0	86%
		1000089	2.3		0.7	71%
		BLO-39-11-14-004			3.5	
		CXLNM1	24.6		2.7	89%
		CXLOX091802*	9.2		4.5	52%
		CXLPROD021202*	5.3		4.4	18%
		CXLPROD091901*	7.9		5.6	29%
		MISSTD-1*	20.6			
		PBO-47-09-012-023	15.1		1.2	92%
		PEOF1			1.1	
		PEOR3258			2.0	
Metal Oxidation	Byproduct	ARF-102-85-114-1	5.8	3.0	1.0	82%
		TS707013	3.5	1.4	1.7	53%
	Product	011589A	7.4		3.2	57%
		011608	4.8		2.3	51%
		07221730	1.5		0.8	51%
		MT1490	3.5			
		TS707001	6.1		2.3	61%
		UPOPLOT0003			0.1	
Misc.	Misc.	64-85-12-1858	35.4		2.9	
		PPSL-365	2.3		0.8	67%
		PuF4-1	1.5		0.3	79%

Table A5-2. Surface Area Data Arranged by Process Category and MIS Sample for All Conditions *(continued)*

Process Category	Process Subcategory	MIS Sample	Surface Area (m^2/g) AR	Surface Area (m^2/g) 600°C	Surface Area (m^2/g) 950°C	% Reduction Due to 950°C calcination
Mixed Actinides	Byproduct	053038	5.6		0.2	97%
		5501407	4.9		1.2	76%
		62750	3.9		0.5	87%
		669194	3.1	2.2	0.8	74%
		CAN92	2.8		0.2	93%
		PSU-84-06-05	0.6	1.2	0.5	28%
		PuUOXBC05	1.0	1.6	0.7	35%
	Misc	CXL1685	3.9		2.7	31%
	Product	5501579*	5.1		0.6	89%
		SCP711-46	27.0			
		SCP711-56	1.4			
Molten Salt Operations	Byproduct	07242141A			0.5	
		520610020	3.0		1.8	42%
		ARF-102-85-223	3.5	1.3	0.5	86%
		ARF-102-85-365	10.5	2.0	2.0	81%
		ATL27960	15.6	4.3	0.8	95%
		C00024A	1.0		0.1	86%
		C00695	0.8		0.6	32%
		C06032A (chunks)			3.1	
		C06032A (powder)	3.8	1.2	2.6	32%
		CLLANL025	3.9		0.7	83%

Notes:

*** Indicates 3-Point Specific Surface Area measurements; all others are 5-Point measurements.**
Multiple results for the same sample and condition are averaged.

Table A5-3. Nonrepresentative Samples: Surface Area Data Arranged by Process Category and MIS Sample for All Conditions

Process Category	Process Subcategory	MIS SAMPLE	Surface Area (m^2/g) AR	Surface Area (m^2/g) 950°C	% Reduction Due to 950°C calcination
Mixed Actinides	Misc	1685	0.32	0.70	-1.2
		BMU	0.19		
		MISNE2	0.83		
Misc	Misc	MISNE4	0.84		

Note: MISNE2 is a mixture of product quality oxide samples. Much of the material had been previously calcined at 950°C

Appendix 6, Density Data

Table A6-1. Representative Samples: Summary of Density Data, % Compaction and Packing Fraction Averaged by Process Category in the 950°C Condition

Process Category	Process Subcategory	Number of Items	Bulk Density (g/mL)	95% LCL, UCL	Tap Density (g/mL)	95% LCL, UCL	Pycnometer density (g/mL)	95% LCL, UCL	% Compaction	Packing Fraction
Aqueous Processing	Byproduct	6-7	2.5	1.3, 3.8	3.2	1.6, 4.8	8.2	5.9, 10.5	22%	0.33
	Product	8-9	3.1	1.9, 4.3	3.8	2.5, 5.1	10.7	9.5, 11.9	21%	0.30
Metal Oxidation	Byproduct	2	3.2	NA	4.2	NA	9.4	NA	25%	0.36
	Product	5-6	4.7	3.6, 5.9	5.6	4.2, 7.0	10.5	9.4, 11.7	15%	0.44
Misc.	Misc.	2-3	3.7	NA	3.2	NA	5.1	NA	18%	0.45
Mixed Actinide Operations	Byproduct	6	3.6	2.7, 4.6	4.5	3.3, 5.7	8.7	7.0, 10.4	20%	0.42
	Misc.	0-1	NA	NA	NA	NA	8.7	NA	28%	0.30
	Product	2-3	2.7	2.3, 3.1	3.8	3.1, 4.4	9.4	NA	15%	0.44
Molten Salt Operations	Byproduct	8-9	3.0	2.3, 3.6	3.5	2.7, 4.2	6.8	5.9, 7.7	22%	0.33

Notes:

NA indicates that density was not measured or results not reported due to large uncertainty in result or small sample size.

The theoretical density of pure plutonium oxide is 11.5 g/mL

$\%\boldsymbol{Compaction} = \mathbf{1} - \left(\boldsymbol{\rho_{bulk}}/\boldsymbol{\rho_{tap}}\right)$ **X 100**

$\boldsymbol{Packing\ Fraction} = \left(\boldsymbol{\rho_{bulk}}/\boldsymbol{\rho_{particle}}\right)$

$\boldsymbol{Void\ space} = \mathbf{1} - \left(\boldsymbol{\rho_{bulk}}/\boldsymbol{\rho_{particle}}\right)$

Table A6-2. Representative Samples: Density Data and Packing Fraction Arranged by Process Category in the AR Condition

Process Category	Process Subcategory	MIS Sample	Bulk Density (g/mL)	Tap Density (g/mL)	Pycnometer density (g/mL)	Packing Fraction
Aqueous Processing	Byproduct	07032282A	3.64	4.21	8.52	0.43
		07242165A	2.37	2.97	5.60	0.42
		07242201A (powder)	2.41	3.35		
		39-01153A	0.63	0.83	2.05	0.31
		63-88-06-121			4.14	
		66-00-11-355	0.94	1.37	4.63	0.20
		66-01-01-439	2.32	2.94	7.60	0.31
		ARF-102-85-355	1.81	2.02	6.10	0.30
	Product	07161856	3.96	4.72	7.38	0.54
		1000089	4.69	5.87	10.10	0.46
		BLO-39-11-14-004	3.04	3.61		
		CXLNM1	1.57	2.07	11.53	0.14
		CXLOX091802	1.37	1.58	6.12	0.22
		CXLPROD021202	2.94	3.13	11.48	0.26
		CXLPROD091901	2.50	3.13	11.35	0.22
		MISSTD-1	1.78	2.28	9.50	0.19
		PBO-47-09-012-023	1.95	2.37		
Metal Oxidation	Byproduct	ARF-102-85-114-1	2.74	4.03	11.63	0.24
		TS707013	3.09	3.96	6.55	0.47
	Product	011589A	2.69	3.92		
		011608	4.50	5.77	10.07	0.45
		07221730	5.03	6.14	11.06	0.46
		MT1490	5.54	6.92	11.56	0.48
		TS707001	1.77	2.95		
Miscellaneous	Miscellaneous	64-85-12-1858	0.51	0.71	3.31	0.15
		PPSL-365	4.34	4.76		
		PuF4-1	0.96	1.15	6.00	0.16
Mixed Actinide Operations	Byproduct	053038	2.71	3.23	5.88	0.46
		5501407	2.31	2.95		
		62750	5.01	6.11	10.68	0.47
		669194	3.49	3.96	7.85	0.44
		CAN92	4.05	4.68		
		PSU-84-06-05	2.09	2.95		
	Miscellaneous	CXL1685	3.40	4.72	10.98	0.31
	Product	5501579	2.58	3.52		
		SCP711-46	3.34	4.30	11.49	0.29
		SCP711-56	4.38	5.56	10.60	0.41
Molten Salt Operations	Byproduct	520610020	1.87	2.28	4.39	0.43
		ARF-102-85-223	2.55	3.27	5.50	0.46
		ARF-102-85-365	2.72	3.49		
		C00024A	3.43	4.29		
		C00695	2.31	2.75	5.72	0.40
		C06032A (powder)	2.00	2.78		
		CLLANL025	2.37	3.59		

Table A6-3. Representative Samples: Density Data and Packing Fraction Arranged by Process Category in the 950°C Condition

Process Category	Process Subcategory	MIS Sample	Bulk Density (g/mL)	Tap Density (g/mL)	Pycnometer density (g/mL)	Packing Fraction
Aqueous Processing	Byproduct	07032282A	3.7	4.5	7.6	0.48
		07242165A	3.1	3.8	10.4	0.30
		07242201A (chunks)	3.6	3.8	7.4	0.48
		07242201A (powder)	3.3	4.3	7.4	0.45
		39-01153A	0.6	0.7	10.4	0.05
		66-00-11-355	1.0	1.3	4.6	0.21
		66-01-01-439	1.9	2.7		
		ARF-102-85-355	4.1	5.1	8.7	0.47
	Product	07161856	6.2	7.0	10.0	0.62
		1000089	5.1	6.4	10.4	0.49
		BLO-39-11-14-004	2.7	3.4	10.8	0.25
		CXLNM1	1.5	2.4	12.0	0.13
		CXLOX091802	1.9	2.4	7.6	0.25
		CXLPROD021202	2.6	3.1	11.9	0.22
		CXLPROD091901	2.2	2.9	11.2	0.19
		PBO-47-09-012-023	2.4	3.0		
		PEOF1	3.1	3.9	11.5	0.27
Metal Oxidation	Byproduct	ARF-102-85-114-1	2.8	4.2	10.9	0.26
		TS707013	3.6	4.3	7.8	0.47
	Product	011589A	4.1	4.6	9.0	0.46
		011608	5.1	6.2	10.5	0.48
		07221730	4.6	5.0	10.8	0.42
		MT1490	6.2	7.5	10.9	0.57
		TS707001	3.0	3.8	11.4	0.26
		UPOPLOT0003	5.4	6.4		
Miscellaneous	Miscellaneous	64-85-12-1858		0.7	3.3	
		PPSL-365	4.4	4.9		
		PuF4-1	3.0	4.0	6.8	0.45
Mixed Actinide Operations	Byproduct	053038	3.4	3.8	6.5	0.52
		5501407	2.4	3.5	8.3	0.28
		62750	5.2	6.7	10.8	0.48
		669194	3.6	4.2	8.0	0.45
		CAN92	3.8	4.7	10.5	0.36
		PSU-84-06-05	3.3	4.2	8.3	0.40
	Miscellaneous	CXL1685			8.7	
	Product	5501579	2.6	3.8	10.4	0.25
		SCP711-46	2.6	3.5		
		SCP711-56	2.9	4.0	8.4	0.35
Molten Salt Operations	Byproduct	07242141A			7.4	
		520610020	2.2	2.5	4.8	0.45
		ARF-102-85-223	3.3	3.9	6.8	0.49
		ARF-102-85-295 (powder)	1.5	1.7	4.9	0.30
		ARF-102-85-365	3.6	4.2	7.1	0.50
		C00024A	3.7	4.2	7.9	0.47
		C00695	3.3	3.7	7.4	0.45
		C06032A (chunks)			5.5	
		C06032A (powder)	2.8	3.5	6.9	0.40
		CLLANL025	3.4	4.4	7.8	0.44

Table A6-4. Nonrepresentative samples: Density Data and Packing Fraction Arranged by Process Category and MIS Sample

Process Category	Process Subcategory	MIS Sample	Condition	Bulk Density (g/mL)	Tap Density (g/mL)	Pycnometer density (g/mL)	Packing Fraction
Misc	Misc	MISNE4	AR			10.0	
Mixed Actinides	Misc	1685	AR			11.0	
			950			8.7	
		BMU	AR	3.5	4.3	7.7	0.45
		MOXSCP-COM	AR	5.3	7.2		

Appendix 7, Calorimetry and Isotopic Data

Table A7-1. Representative Samples: Calorimetry and Gamma-Ray Isotopic Data Arranged by Process Category

Process Category	Process Sub-category	MIS Sample	Pu-238 /Pu wt%	Pu-239 /Pu wt%	Pu-240 /Pu wt%	Pu-241 /Pu wt%	Pu-242 /Pu wt%	Analysis Date	Specif. Pwr. W/kg	Pu wt%	U wt%	Am wt%	Np wt%
Aqueous Processing	By-product.	07032282A	0.015	93.934	5.839	0.187	0.025	11-Sep-97	1.66	66.7	0.0	0.10	0.00
		07242165A	0.012	94.014	5.760	0.189	0.025	24-Sep-97	0.74	29.5	0.0	0.05	0.00
		07242201A	0.013	93.873	5.890	0.199	0.025	17-Sep-97	1.35	54.7	0.0	0.07	0.00
		39-01153A	0.011	93.823	5.969	0.172	0.025	25-Sep-97	0.10	3.9	0.0	0.01	0.00
		63-88-06-121	0.006	96.615	3.320	0.040	0.018	23-Oct-03	0.83	35.7	0.0	0.05	0.00
		66-00-11-355	0.028	93.814	5.804	0.328	0.025	19-Jul-01	0.79	29.1	0.0	0.08	0.01
		66-01-01-439	0.031	93.755	5.830	0.360	0.025	19-Jul-01	1.69	61.4	0.0	0.19	0.01
		ARF-102-85-355	0.013	93.770	6.010	0.185	0.023	25-Jul-96	1.17	46.4	0.0	0.08	0.09
	Product	07161856	0.015	94.014	5.731	0.215	0.025	4-Jun-97	1.92	79.0	0.0	0.09	0.00
		1000089	0.010	93.915	5.887	0.163	0.025	18-Jun-97	2.11	84.2	0.0	0.16	0.00
		BLO-39-11-14-004	0.860	74.145	20.604	2.936	1.454	25-Jul-96	13.40	85.2	0.0	5.86	0.16
		CXLNM1	***0.011***	***93.926***	***5.896***	***0.141***	***0.025***	***4-Feb-03***	***2.04***	***88.4***	***0.0***	***0.01***	***0.01***
		CXLOX091802	0.011	93.853	5.973	0.138	0.025	13-Aug-03	2.37	71.0	0.0	0.64	0.02
		CXLPROD021202	***0.010***	***93.644***	***6.220***	***0.101***	***0.025***	***22-Aug-08***	***2.06***	***87.6***	***0.1***	***0.03***	***0.00***
		CXLPROD091901	0.009	93.885	6.009	0.102	0.025	6-Nov-01	2.01	87.6	0.0	0.00	0.01
		MISSTD-1	0.018	93.412	6.319	0.226	0.025	3-Jun-97	2.09	86.0	0.0	0.05	0.00
		PBO-47-09-012-023	0.079	86.112	12.602	0.960	0.247	25-Jul-96	3.14	87.5	0.0	0.43	0.02
		PEOF1	***0.011***	***93.804***	***6.005***	***0.155***	***0.025***	***20-Oct-01***	***2.04***	***88.6***	***0.0***	***0.00***	***0.00***
Metal Oxidation	By-product	ARF-102-85-114-1	0.009	93.544	6.304	0.118	0.026	6-Aug-96	2.19	85.9	0.0	0.18	0.02
		TS707013	0.011	94.269	5.561	0.135	0.025	18-Jun-97	1.69	67.3	0.0	0.13	0.00
	Product	011589A	0.015	93.647	6.089	0.224	0.025	1-Oct-97	1.93	77.3	0.0	0.11	0.00
		011608	0.017	93.927	5.808	0.223	0.025	5-Jun-97	2.14	85.5	0.0	0.13	0.00
		07221730	0.013	93.634	6.125	0.203	0.025	18-Jun-97	2.14	87.5	0.0	0.10	0.00
		MT1490	0.009	94.169	5.663	0.133	0.025	5-Jun-97	2.17	85.6	0.0	0.19	0.60
		TS707001	0.019	93.745	5.981	0.230	0.025	11-Sep-97	2.18	86.3	0.0	0.13	0.00
		UPOPLOT0003	***0.007***	***94.402***	***5.521***	***0.050***	***0.020***	***19-Aug-10***	***2.40***	***87.0***	***0.4***	***0.38***	***0.02***
Misc.	Misc.	***04272-CC-220***	***0.015***	***91.810***	***8.027***	***0.129***	***0.020***	***6-Jan-09***	***2.44***	***82.9***	***0.2***	***0.36***	***0.02***
		41-85-08-1379	0.029	93.222	6.460	0.264	0.025	23-Oct-03	1.24	41.4	0.0	0.21	0.00
		64-85-12-1858	0.018	92.150	7.587	0.173	0.071	23-Oct-03	1.05	32.7	0.0	0.22	0.01
		PPSL-365	0.030	92.994	6.513	0.434	0.029	29-Jul-96	2.26	83.4	0.0	0.19	0.00
		PUF4-1	0.019	94.532	5.325	0.098	0.025	10-Nov-99	1.87	71.6	0.0	0.18	0.00
Mixed Actinides	By-product	053038	0.009	93.986	5.836	0.144	0.025	18-Jun-97	2.10	52.9	3.7	0.78	0.00
		5501407	0.015	94.030	5.729	0.201	0.025	6-Jun-97	1.61	65.7	10.9	0.09	0.00
		62750	0.015	93.838	5.925	0.197	0.025	5-Jun-97	2.17	86.1	0.5	0.15	0.00
		669194	0.010	95.690	4.111	0.164	0.025	6-Jun-97	0.37	15.3	69.2	0.03	0.00
		CAN92	0.012	93.750	6.049	0.164	0.025	3-Jun-97	2.08	81.6	2.3	0.17	0.00
		PSU-84-06-05	0.017	90.612	9.090	0.198	0.084	2-Aug-96	0.52	15.7	65.1	0.11	0.00
	Misc.	***CXL1685***	***0.013***	***93.755***	***6.058***	***0.149***	***0.025***	***27-Mar-02***	***0.14***	***6.0***	***79.9***	***0.00***	***0.00***
	Product	5501579	0.013	93.647	6.123	0.192	0.025	5-Jun-97	2.10	86.4	0.1	0.08	0.00
		SCP711-46	***0.041***	***88.543***	***10.797***	***0.594***	***0.025***	***10-Nov-99***	***0.21***	***6.2***	***78.3***	***0.04***	***0.00***
		SCP711-56	0.091	88.588	10.389	0.712	0.220	3-Oct-01	0.63	17.5	70.0	0.09	0.00
Molten Salt Operations	By-product	07242141A	0.015	94.020	5.721	0.220	0.025	11-Sep-97	1.08	42.9	0.0	0.08	0.00
		520610020	0.012	93.877	5.924	0.162	0.025	6-Jun-97	0.89	30.9	0.0	0.16	0.00
		ARF-102-85-223	0.008	93.379	6.457	0.127	0.028	7-Aug-96	1.58	64.2	0.0	0.09	0.02
		ARF-102-85-295	0.008	94.181	5.686	0.107	0.019	7-Aug-96	0.69	28.0	0.0	0.05	0.01
		ARF-102-85-365	0.010	93.473	6.371	0.119	0.027	7-Aug-96	1.45	58.9	0.0	0.08	0.00
		C00024A	0.013	93.586	6.167	0.210	0.025	16-Sep-97	1.78	69.5	0.0	0.15	0.00
		C00695	0.010	93.963	5.890	0.112	0.025	4-Jun-97	1.67	69.2	0.0	0.07	0.00
		C06032A	0.010	94.000	5.848	0.117	0.025	1-Oct-97	1.29	51.8	0.0	0.09	0.00
		CLLANL025	0.014	93.918	5.871	0.172	0.025	23-Sep-97	1.79	71.3	0.0	0.13	0.00
		PMAXBS	***0.014***	***93.937***	***5.885***	***0.139***	***0.025***	***14-Aug-03***	***1.69***	***71.8***	***0.0***	***0.03***	***0.00***

Note: Data for samples in AR condition unless indicated by *Bold/Italic*. Those data are for the 950°C condition (800°C for MIS Sample PMAXBS) due to unavailability of data.

Table A7-2. Nonrepresentative Samples: Calorimetry and Gamma-Ray Isotopic Data Arranged by Process Category

Process Category	Process Sub-category	MIS Sample	Cond-ition	Pu-238 /Pu wt%	Pu-239 /Pu wt%	Pu-240 /Pu wt%	Pu-241 /Pu wt%	Pu-242 /Pu wt%	Analysis Date	Specif. Pwr. W/kg	Pu wt%	U wt%	Am wt%	Np wt%
Metal Oxidation	Byproduct	101707001	AR	0.013	93.71	6.11	0.13	0.025	05-Aug-03	0.50	19.7	0.0	0.04	0.00
Mixed Actinide	Misc.	MISNE2	950C	0.017	94.16	5.67	0.13	0.025	24-Apr-01	2.10	83.0	0.9	0.15	0.00
Aqueous Processing	Byproduct	07242243A	AR	0.012	94.09	5.73	0.14	0.025	06-Aug-03	0.63	24.6	0.0	0.06	0.00
Misc	Misc	YBG2-NRDL-4	AR	0.007	94.38	5.53	0.06	0.025	01-Jul-03	2.10	77.5	0.0	0.31	0.02
		MISNE4	950C	0.012	94.06	5.77	0.13	0.025	24-Oct-01	1.69	66.2	0.0	0.13	0.00

Table A7-3. Representative Samples: Comparison of Calorimetry and Gamma-Ray Isotopic Data Measured Before and After Calcination at 950°C

Process Category	Process Subcategory	MIS Sample	Condition	Analysis Date	Specific Power (W/kg)	Pu wt%
Aqueous Processing	Byproduct	07032282A	950C	14-Oct-98	1.68	67.9
		07032282A	AR	11-Sep-97	1.66	66.7
		07242165A	950C	24-Nov-98	0.87	34.1
		07242165A	AR	24-Sep-97	0.74	29.5
		07242201A	950C	13-Oct-98	1.57	64.1
		07242201A	AR	17-Sep-97	1.35	54.7
		39-01153A	950C	30-Jan-98	0.20	7.7
		39-01153A	AR	25-Sep-97	0.10	3.9
		66-01-01-439	950C	25-Nov-03	1.78	63.7
		66-01-01-439	AR	19-Jul-01	1.69	61.4
		ARF-102-85-355	950C	2-Dec-98	1.68	65.6
		ARF-102-85-355	AR	25-Jul-96	1.17	46.4
	Product	07161856	950C	24-Nov-98	2.08	84.2
		07161856	AR	4-Jun-97	1.92	79.0
		1000089	950C	25-Nov-98	2.12	84.6
		1000089	AR	18-Jun-97	2.11	84.2
		BLO-39-11-14-004	950C	30-Apr-02	13.69	77.3
		BLO-39-11-14-004	AR	25-Jul-96	13.40	85.2
		CXLPROD091901	950C	3-Feb-10	2.04	87.0
		CXLPROD091901	AR	6-Nov-01	2.01	87.6
Metal Oxidation	Byproduct	ARF-102-85-114-1	950C	24-Nov-98	2.18	86.3
		ARF-102-85-114-1	AR	6-Aug-96	2.19	85.9
		TS707013	950C	14-Oct-98	1.71	69.8
		TS707013	AR	18-Jun-97	1.69	67.3
	Product	011589A	950C	6-Aug-98	1.98	77.7
		011589A	AR	1-Oct-97	1.93	77.3
		011608	950C	19-Nov-98	2.12	84.5
		011608	AR	5-Jun-97	2.14	85.5
		07221730	950C	10-Mar-99	2.13	85.8
		07221730	AR	18-Jun-97	2.14	87.5
		MT1490	950C	26-Nov-01	2.18	87.8
		MT1490	AR	5-Jun-97	2.17	85.6
		TS707001	950C	23-Jul-98	2.20	87.0
		TS707001	AR	11-Sep-97	2.18	86.3

Table A7-3. Representative Samples: Comparison of Calorimetry and Gamma-Ray Isotopic Data Measured Before and After Calcination at 950°C *(continued)*

Process Category	Process Subcategory	MIS Sample	Condition	Analysis Date	Specific Power (W/kg)	Pu%
Misc.	Misc.	PPSL-365	950C	27-Nov-01	2.33	84.9
		PPSL-365	AR	29-Jul-96	2.26	83.4
		PUF4-1	950C	17-Mar-04	1.55	63.0
		PUF4-1	AR	10-Nov-99	1.87	71.6
Mixed Actinide Operations	Byproduct	053038	950C	30-Nov-98	2.13	60.4
		053038	AR	18-Jun-97	2.10	52.9
		5501407	950C	7-Jul-99	1.61	66.7
		5501407	AR	6-Jun-97	1.61	65.7
		669194	950C	9-Jul-98	2.16	85.9
		669194	AR	5-Jun-97	2.17	86.1
		CAN92	950C	13-Oct-98	0.33	13.9
		CAN92	AR	6-Jun-97	0.37	15.3
		PSU-84-06-05	950C	29-Nov-01	2.14	83.6
		PSU-84-06-05	AR	3-Jun-97	2.08	81.6
		62750	950C	23-Jul-98	0.47	14.4
		62750	AR	2-Aug-96	0.52	15.7
	Product	5501579	950C	29-Nov-01	2.20	88.1
		5501579	AR	5-Jun-97	2.10	86.4
		SCP711-56	950C	25-Nov-03	0.62	17.2
		SCP711-56	AR	3-Oct-01	0.63	17.5
Molten Salt Operations	Byproduct	ARF-102-85-223	950C	19-Nov-98	0.95	33.7
		ARF-102-85-223	AR	6-Jun-97	0.89	30.9
		ARF-102-85-295	950C	30-Nov-98	1.74	70.9
		ARF-102-85-295	AR	7-Aug-96	1.58	64.2
		ARF-102-85-365	950C	28-Nov-01	0.99	39.7
		ARF-102-85-365	AR	7-Aug-96	0.69	28.0
		C00024A	950C	6-Aug-98	1.68	68.4
		C00024A	AR	7-Aug-96	1.45	58.9
		C00695	950C	27-Jun-01	1.90	73.4
		C00695	AR	16-Sep-97	1.78	69.5
		C06032A	950C	1-Dec-98	1.79	71.1
		C06032A	AR	4-Jun-97	1.67	69.2
		CLLANL025	950C	15-Oct-98	1.57	65.9
		CLLANL025	AR	1-Oct-97	1.29	51.8

Appendix 8, Prompt Gamma Analysis Data

Table A8-1. Representative Samples: Summary of Impurity Concentrations Measured by Prompt Gamma and Averaged by Process Category in the 950°C Condition

Process Category	Process Subcategory	Cl (wt%) *(LCL, UCL)*	Mg (wt%) *(LCL, UCL)*	Na (wt%) *(LCL, UCL)*	K (wt%) *(LCL, UCL)*	F (wt%) *(LCL, UCL)*
Aqueous Processing	Byproduct	0.6 *(0.1,1.7)*	8.5 *(0.5,10.0)*	0.4 *(0.1,0.5)*	NA	0.6 *(0.1,1.4)*
	Product	1.5	0.1	0.04 *(0.0,0.1)*	NA	NA
Metal Oxidation	Byproduct	NA	NA	0.1	NA	NA
	Product	1.0	1.6	0.1 *(0,5)*	NA	0.4
Miscellaneous	Miscellaneous	1.2	0.3 *(0.1, 0.5)*	0.7 *(0.1,1.9)*	2.0	3.4 *(0, 5.0)*
Mixed Actinide Operations	Byproduct	5.7	0.5 *(0, 4.8)*	0.2 *(0,0.5)*	2.4	0.6 *(0.1,1.3)*
	Product	NA	NA	0.1	NA	NA
Molten Salt Operations	Byproduct	5.4 *(3.4,7.1)*	0.9 *(0.9, 1.3)*	1.7 *(0.5, 2.8)*	3.1 *(2.8, 4.1)*	0.2 *(0,0.7)*

Table A8-2.Representative Samples: Impurity Concentrations Measured by Prompt Gamma Arranged by Process Category in the 950°C Condition

Process Category	Process Subcategory	MIS Sample	Al (wt%)	Be (wt%)	Cl (wt%)	F (wt%)	Mg (wt%)	Na (wt%)	K (wt%)	P (wt%)
Aqueous Processing	Byproduct	07032282A	0.1		0.7	0.9	0.1	0.17		
		07242165A	0.3			0.1	0.1	0.10		
		07242201A	0.3		0.3	1.0	0.1	0.25		0.1
		63-88-06-121	1.4			0.6	0.9	1.52		0.2
		66-00-11-355	0.4				16.2	0.24		0.7
		66-01-01-439					10.8			
		ARF-102-85-355			0.7	0.3	0.1	0.23		
	Product	07161856		0.03		0.1		0.05		
		BLO-39-11-14-004						0.01		
		CXLOX091802	0.1		1.5	1.9	0.1	0.09		0.0
		CXLPROD021202				0.0		0.00		
		CXLPROD091901								
		PBO-47-09-012-023						0.02		

Table A8-2. Representative Samples: Impurity Concentrations Measured by Prompt Gamma Arranged by Process Category in the 950°C Condition *(continued)*

Process Category	Process Subcategory	MIS Sample	Al (wt%)	Be (wt%)	Cl (wt%)	F (wt%)	Mg (wt%)	Na (wt%)	K (wt%)	P (wt%)
Metal Oxidation	Byproduct	ARF-102-85-114-1		0.10				0.07		
	Product	011589A	0.3		1.9	0.7	3.2	0.36		
		MT1490						0.04		
		TS707001			0.1	0.2	0.1	0.01		
		UPOPLOT0003								
Misc.	Misc.	04272-CC-220			1.2	0.0		0.10	2.0	
		41-85-08-1379	0.8			0.5	0.2	0.91		
		64-85-12-1858	0.2			0.2	0.1	0.15		
		PPSL-365								
		PuF4-1				18.7				
Mixed Actinide Operations	Byproduct	053038	0.5	0.04	5.0	0.9	0.2	0.62		
		053038 (chunks)		0.01	3.0	0.5	0.1	0.29		
		053038 (powder)		0.04	6.4	0.8	0.2	0.59	2.4	
		5501407		0.05			0.0	0.02		
		669194		0.41				0.13		
		CAN92		0.34				0.02		
	Product	PSU-84-06-05								
Molten Salt Operations	Byproduct	PuUOXBC05				0.2	1.6	0.08		
		5501579								
		SCP711-46						0.06		
		SCP711-56								
		07242141A				0.1		0.04		
		ARF-102-85-223			9.2		1.6	3.40	3.3	
		ARF-102-85-223 (chunks)			5.0		0.1	2.92	1.6	
		ARF-102-85-223 (powder)			5.5		0.7	1.88	4.7	
		ARF-102-85-295	0.2		3.9	0.3	1.2	1.28	2.9	
		ARF-102-85-365			3.8	0.1	0.7	1.55	2.4	

Note: Multiple results for the same sample and condition are averaged.

Table A8-3. Nonrepresentative Samples: Impurity Concentrations Measured by Prompt Gamma Arranged by Process Category in the 950°C Condition

Process Category	Process Subcategory	MIS SAMPLE	Al (wt%)	Be (wt%)	Cl (wt%)	F (wt%)	Mg (wt%)	Na (wt%)	K (wt%)	P (wt%)
Aqueous Processing	Byproduct	07242243A				1.0				
		07242326A				2.6				
Metal Oxidation	Byproduct	101707001			7.3			1.4		
Mixed Actinides	Misc	MISNE2						0.1		
Misc	Misc	MISNE4	0.4		4.6	0.1	2.5	1.0	2.1	
		YBG2-NRDL-4				13.6				

Note: For all prompt gamma tables, no entry indicates elemental wt% was below detectable limits.

Appendix 9, Trace Element Analysis Data

Table A9-1. Representative Samples: Summary of Trace Element Analysis Data Averaged by Process Category for MIS Samples in the 950°C Condition

Process Category	Process Subcategory	Cl (wt%)	F (wt%)	Ca (wt%)	Mg (wt%)	Na (wt%)	K (wt%)
Aqueous Processing	Byproduct	0.4 (0.04,0.9)	0.7 (0.2,1.2)	0.7 (0.2,1.1)	10.8 (0.1,13.8)	1.1 (0.1,1.3)	0.4 (0.1,0.8)
	Product	0.3 (0.04,0.8)	0.3 (0,0.4)	0.5 (0,1.2)	0.3 (0,0.9)	0.1 (0,0.5)	0.3 (0,0.4)
Metal Oxidation	Byproduct	1.0	0.03	0.1	0.7	0.4	0.5
	Product	0.3 (0.01,0.9)	0.2 (0,0.6)	0.2 (0,1.0)	0.5 (0,1.7)	0.1 (0,0.2)	0.7 (0,1.0)
Miscellaneous	Miscellaneous	0.6	9.6	0.8	0.4	0.3	1.1
Mixed Actinide Operations	Byproduct	0.9 (0.01,2.0)	0.2	0.7 (0,2.6)	0.9 (0,1.3)	0.2 (0,0.3)	0.3 (0,0.4)
	Miscellaneous	NA	0.01	NA	0.01	NA	0.01
	Product	0.07	NA	0.0	0.01	0.1	0.03
Molten Salt Operations	Byproduct	5.1 (2.7,7.5)	0.07 (0,0.1)	*0.1 (0,0.2)	1.7 (0.3, 2.1)	1.9 (0.7,2.7)	2.7 (1.1,3.4)

Note: *One outlier (8.6) removed

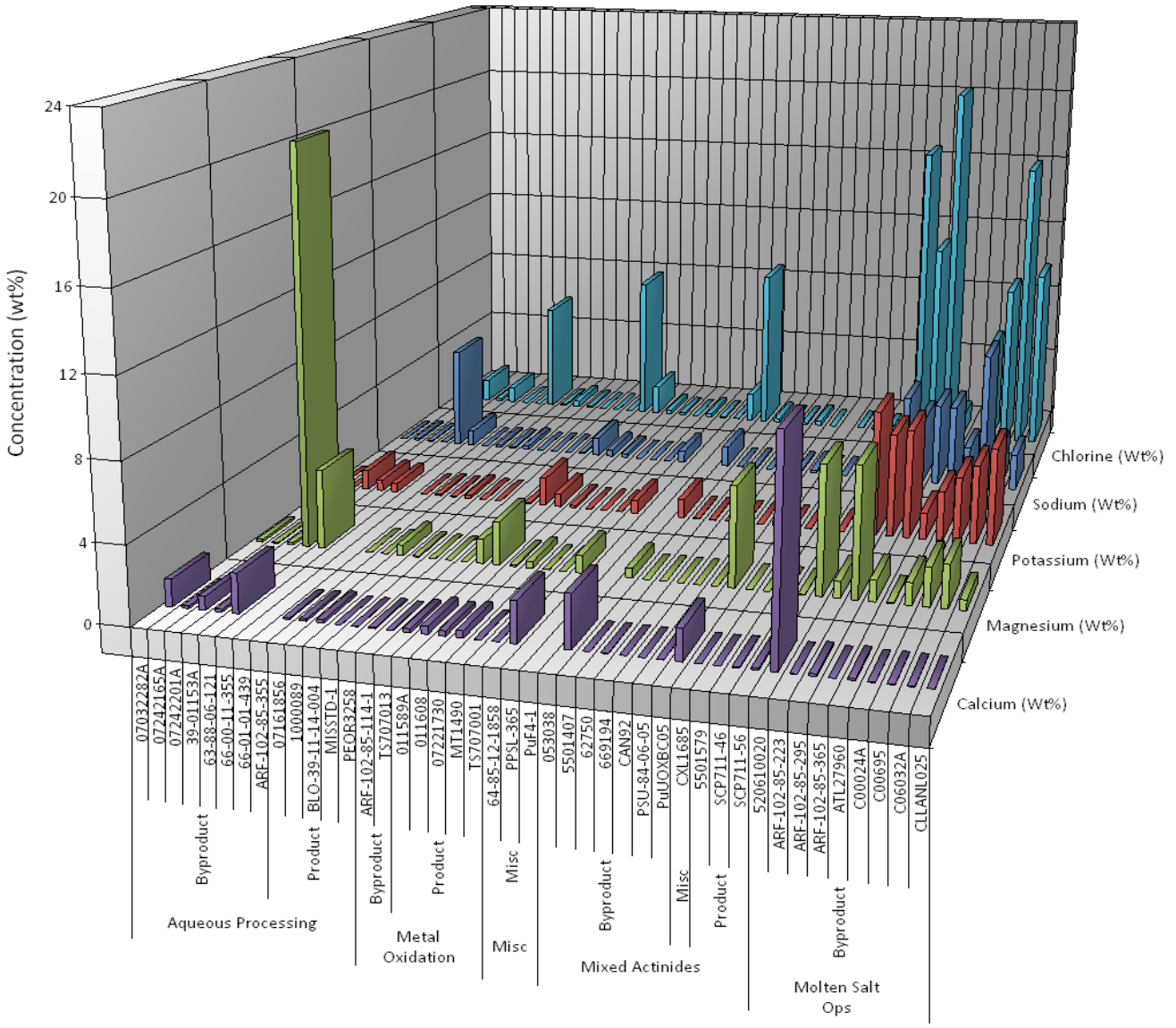

Figure A9-1. Representative Samples: Trace element analysis data arranged by process category and MIS sample for select alkali and alkaline-earth metals and chlorine in "as-received" samples.
Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

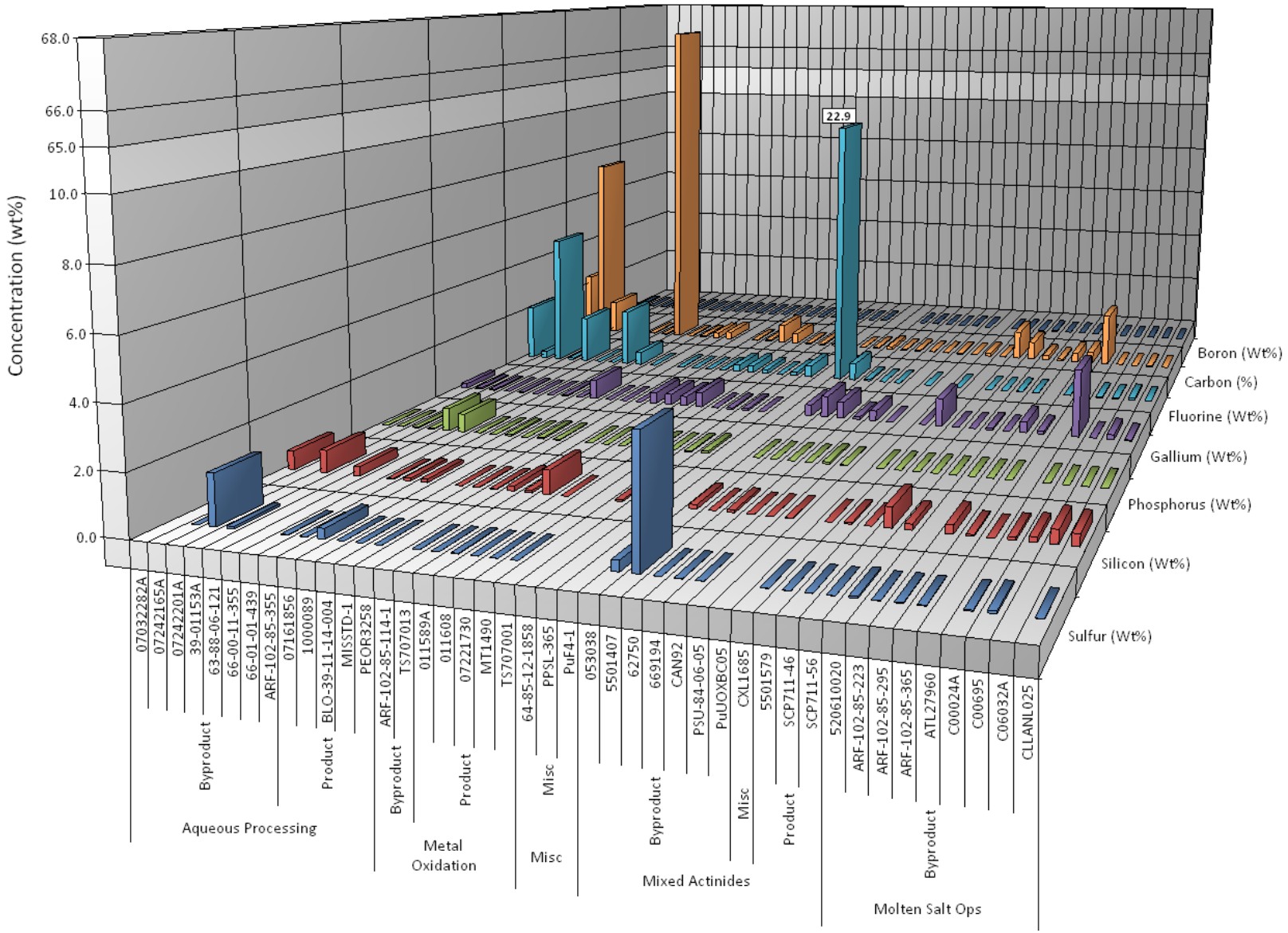

Figure A9-2. Representative Samples: Trace element analysis data arranged by process category and MIS sample for select nonmetal elements in "as-received" samples. Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

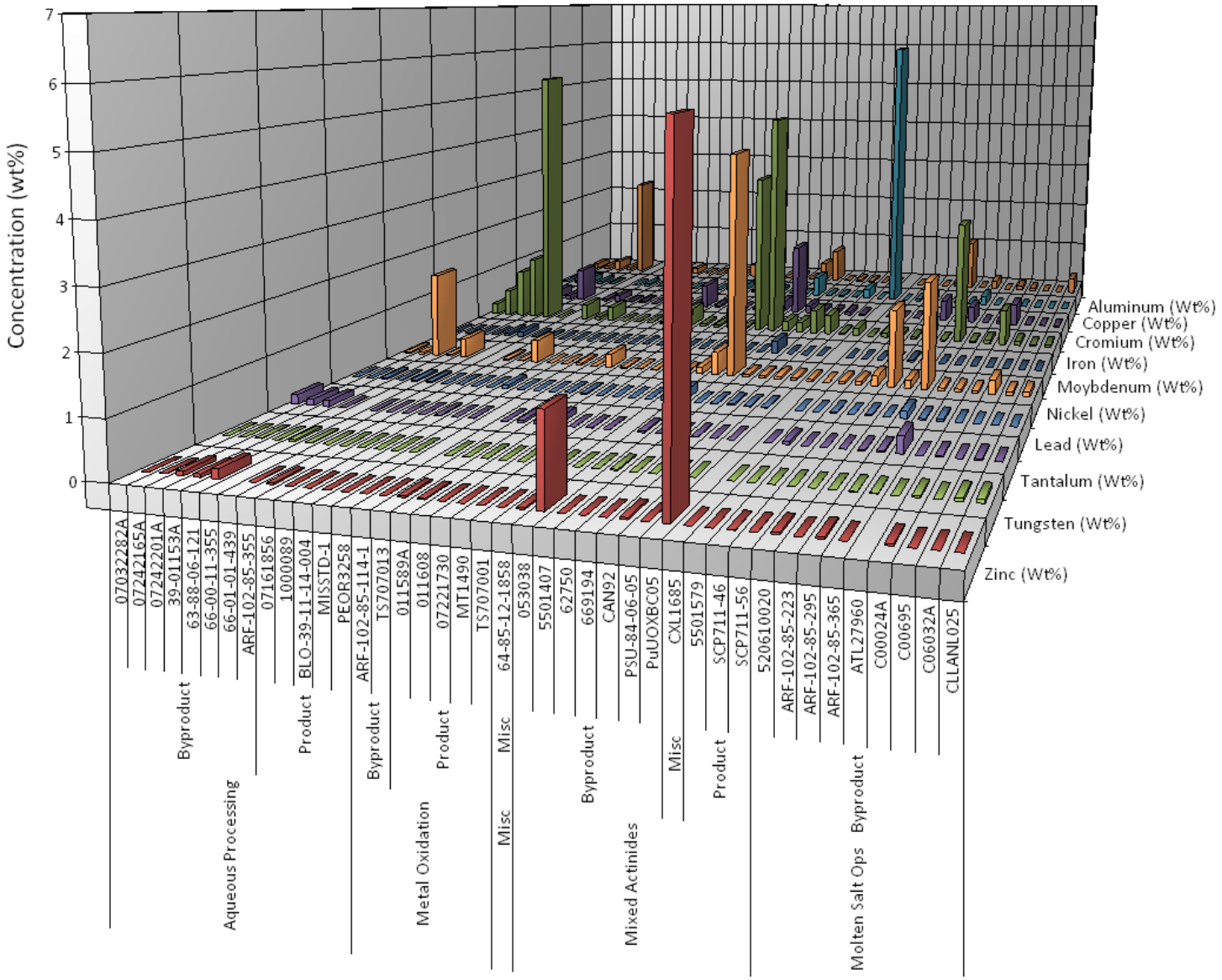

Figure A9-3. Representative Samples: Trace element analysis data arranged by process category and MIS sample for select metal elements in in "as-received" samples. Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

Table A9-2. Representative Samples: Trace Element Analysis Data Arranged by Process Category and MIS Sample for All Conditions

Process Category	Process Sub-category	MIS Sample	Cond	Cl ppm	F ppm	Al ppm	Be ppm	B ppm	Ca ppm	C ppm	Cr ppm	Cu ppm	Ga ppm	Fe ppm	Pb ppm	Mg ppm	Mo ppm	Ni ppm	K ppm	Si ppm	Na ppm	S ppm	Ta ppm	W ppm	Zn ppm
Aqueous	Product	07161856	AR	7.5E+2	5.0E+3	1.0E+2	1.2E+0	2.4E+1	1.1E+3	3.0E+2	1.6E+2	2.4E+1	1.0E+2	5.9E+2	5.6E+0	9.5E+1	1.5E+1	1.2E+2	3.7E+2	1.1E+3	6.5E+2	3.2E+3	4.2E+1	1.2E+1	1.1E+1
			800C	5.2E+2	2.3E+3	9.0E+1	2.6E+0	6.0E+1	1.2E+3	4.5E+1	5.2E+1	1.4E+1	4.0E+1	4.3E+2	7.1E+0	1.0E+2	3.2E+1	7.2E+1	2.0E+2	1.7E+3	5.9E+2	7.0E+1	6.3E+0	7.1E+0	3.9E+1
			950C	2.4E+2	1.0E+3	1.2E+2	6.1E+0	7.7E+1	1.3E+3	2.9E+2	3.0E+2	9.3E+0	4.5E+1	1.4E+3	1.2E+0	1.3E+2	3.8E+1	1.4E+2	4.0E+2	9.0E+2	4.4E+2	1.1E+2	3.3E+0	1.6E+1	1.4E+1
		1000089	AR	3.4E+3	1.8E+1	1.2E+3	8.9E-1	6.7E+1	1.7E+3	5.4E+2	3.1E+2	6.2E+1	7.1E+3	3.0E+3	2.4E+1	5.5E+3	5.7E+0	4.4E+3	1.6E+3		2.1E+3	5.0E+1	1.6E+2	1.7E+0	2.8E+1
			800C	3.2E+3	1.5E+2	1.7E+3	2.0E-1	6.8E+1	2.6E+3	2.0E+2	1.9E+2	2.6E+1	7.8E+2	5.7E+2	3.2E+0	4.5E+3	2.9E+0	6.7E+2	8.6E+2	4.9E+2	1.3E+3		4.8E+1	1.9E+0	7.0E+0
			950C	2.6E+3	5.0E+2	2.4E+3	3.6E+0	1.7E+2	2.0E+3	1.7E+2	4.3E+2	2.6E+1	2.2E+3	9.9E+2	2.4E+0	1.2E+4	1.6E+1	1.6E+3	5.8E+2	3.7E+2	1.0E+3		2.5E+2	9.2E+0	1.3E+1
		BLO-39-11-14-004	AR	1.0E+3		2.5E+1	2.6E+0	5.1E+1	6.3E+1	2.1E+3	8.0E+0	5.0E+0	3.0E+0	1.0E+2	6.0E+2	6.4E+2	1.4E+0	3.0E+0	3.1E+2	4.0E+1	3.0E+1	5.0E+1	2.3E+0	5.7E+0	1.2E+1
			600C	2.3E+3		5.3E+1	2.4E+0	2.8E+1	8.9E+1	3.4E+2	7.0E+1	7.0E+1	2.6E+1	2.7E+2	6.7E+2	8.2E+2	1.2E+1	1.5E+2	5.3E+2	7.2E+1	1.3E+2	6.0E+1	1.6E+1	1.0E+1	2.3E+1
			950C	1.5E+3		2.0E+1	3.3E+0	9.3E+1	8.3E+1	9.0E+1	7.8E+1	5.0E+0	2.0E+1	4.9E+2	4.3E+1	8.4E+2	1.7E+1	2.0E+0	4.9E+2	2.1E+2	3.0E+1	5.0E+1	1.3E+1	7.0E+1	8.0E+0
		CXLOX091802	950C	2.2E+4	1.5E+4	7.0E+2	6.9E-1		2.9E+4		7.3E+2	1.7E+2	2.0E+1	3.4E+3	5.5E+1	2.0E+3		6.7E+2	2.4E+4	9.7E+2	2.2E+3		9.2E+2	1.5E+2	
		CXLPROD021202	950C	2.0E+2	1.3E+2	5.8E+1	2.0E-1		1.0E+1		4.2E+1	2.0E+1	3.2E+0	1.3E+2	2.0E+2	3.0E+0		4.8E+1	1.2E+2	8.8E+1	2.5E+1				
		CXLPROD091901	950C	3.4E+2	1.1E+2	1.4E+2	2.0E-1		2.6E+1		2.7E+1	2.0E+1	2.0E+2	7.9E+1	1.2E+2	1.1E+1		5.7E+1	1.2E+2	1.9E+2	2.8E+1				
		MISSTD-1	AR	7.5E+1	2.1E+2	7.9E+1	2.5E+0	3.4E+1	1.9E+2	2.9E+3	3.0E+1	1.1E+1	6.8E+0	4.8E+1	1.6E+1	6.7E+1	8.3E+0	2.1E+1	1.3E+2	2.0E+2	1.2E+2	4.0E+1	3.1E+1	3.4E+1	5.1E+0
		PBO-47-09-012-023	950C	7.9E+2	4.7E+1	8.0E+1	1.4E+0	5.9E+0	6.4E+1	2.0E+1	4.7E+1	5.2E+1	4.4E+1	1.6E+2	2.0E+0	5.9E+1	3.0E+0	1.3E+2	9.8E+1	6.2E+1	7.0E+1	6.0E+1	3.0E+0	7.0E+0	2.0E+0
		PEOF1	950C	3.0E+1	3.0E+1	2.5E+1	5.0E-1	7.9E+0	6.1E+1	8.0E+1	9.9E+0	5.0E+0	5.9E+0	3.7E+1	3.0E+0	4.7E+0	2.7E+0	1.5E+1	7.7E+1	1.4E+2	2.2E+1	7.9E+0	4.3E+0	2.0E+0	5.5E+1
		PEOR3258	AR	3.6E+1	4.0E+1	2.0E+1	1.5E+0	6.5E+0	6.3E+2		1.4E+2	3.3E+0	1.7E+1	1.6E+2	1.8E+1	3.1E+1	2.0E+1	4.2E+2		1.6E+2	6.7E+1				5.0E+0
			950C	1.0E+2	1.2E+2	1.9E+1	8.0E-1		1.6E+2				6.4E+0	9.8E+1		3.0E+1		2.5E+2		2.7E+2			2.4E+2	
	By-product	07032282A	AR	1.7E+3	2.1E+4	1.2E+3	1.0E+1	4.2E+2	1.4E+4	2.4E+4	9.6E+2	6.5E+1	1.7E+3	2.3E+3	4.6E+1	1.2E+3	1.1E+1	2.9E+2	8.4E+1	6.3E+3	2.6E+2		1.8E+3	1.6E+1	2.0E+1
			950C	6.0E+3	1.2E+4	1.8E+3	1.0E+1	1.1E+2	1.1E+4	4.3E+2	5.5E+2	1.0E+1	5.6E+2	1.6E+3	1.0E+1	1.7E+3	1.0E+1	2.1E+2	1.2E+3	9.0E+3	2.5E+3		6.1E+3	9.3E+1	1.0E+1
		07242165A	AR	4.1E+2	2.6E+3	7.6E+2	2.0E+0	9.8E+1	1.2E+3	7.4E+4	3.6E+3	4.0E+1	4.5E+2	5.5E+3	1.5E+1	3.3E+2	3.3E+0	4.9E+2	4.0E+1		9.9E+2	5.1E+1	1.2E+3	9.8E-1	7.3E+1
			800C	2.9E+2	1.6E+3	7.6E+2	4.1E+0	1.4E+2	1.3E+3	9.6E+2	2.5E+3	9.5E+0	2.5E+2	3.9E+3	2.3E+0	1.3E+2	1.9E+0	4.2E+2	1.3E+3		2.0E+3	3.0E+1	9.7E+2	7.0E+0	1.5E+1
			950C	1.5E+2	9.8E+2	3.5E+3	1.9E+0	1.1E+2	8.2E+2	2.4E+3	4.6E+2	9.7E+0	2.0E+2	3.4E+3	4.2E+0	4.0E+1	3.1E+0	6.2E+1	1.3E+3		1.4E+3	2.6E+2	1.5E+2	1.3E+1	1.3E+1
		07242201A (powder)	AR	1.3E+4	5.0E+4	2.0E+3	1.4E+1	1.3E+2	7.0E+3	1.3E+4	1.9E+3	1.2E+3	4.0E+2	9.9E+3	3.0E+2	1.3E+3	1.5E+1	1.6E+4	1.1E+4	7.5E+3	5.1E+2	1.6E+4	1.1E+3	6.3E+1	7.0E+2
			950C	3.9E+3	1.3E+4	5.6E+3	4.1E+0	3.9E+1	7.8E+3	1.6E+3	1.4E+3	2.4E+2	4.2E+2	7.8E+3	1.1E+2	2.3E+3	2.1E+1	1.2E+4	5.6E+3	1.5E+3	2.7E+3	7.9E+3	6.0E+2	1.0E+2	2.2E+2
		07242201A (chunks)	950C	5.2E+3	1.4E+4	1.9E+4	4.4E+0	1.4E+1	5.4E+3	5.0E+1	2.2E+2	9.6E+1	2.9E+2	1.3E+3	3.4E+1	1.1E+3	1.4E+1	2.2E+3	8.5E+3	5.7E+3	3.0E+3	1.7E+3	1.0E+1	4.0E+1	3.2E+1
		ARF-102-85-355	AR	6.1E+4	2.2E+4	1.8E+2	4.0E+0	9.0E+1	6.6E+2	6.7E+5	1.5E+3	5.2E+2	3.0E+2	3.2E+3	8.5E+1	8.0E+1	2.1E+1	2.6E+2	8.5E+2	8.7E+2	1.0E+3	8.0E+1	2.0E+1	2.5E+1	2.4E+2
			600C	4.1E+3	5.5E+3	4.2E+2	3.0E+0	6.0E+0	5.3E+2	1.1E+3	5.8E+3	5.3E+2	3.4E+2	1.8E+4	6.4E+1	8.8E+1	6.6E+1	1.9E+3	9.7E+2	8.3E+2	7.1E+2	2.1E+2	3.1E+1	3.9E+1	1.5E+2
			950C	7.4E+3	3.6E+3	3.6E+2	4.0E+0	9.0E+0	9.0E+2	4.8E+2	4.7E+3	1.7E+2	8.8E+2	3.7E+3	7.3E+0	1.7E+2	5.7E+1	3.5E+2	1.3E+2	9.2E+2	3.1E+2	8.0E+1	6.3E+1	2.8E+1	2.5E+2
		39-01153A	AR	1.5E+3	5.6E+2	7.6E+2	5.8E+0	8.0E+2	1.4E+3	5.8E+3	9.0E+2	5.2E+2	8.0E+1	1.3E+4	1.0E+2	2.1E+5	2.9E+1	4.2E+2	6.0E+3		1.7E+2	7.5E+2	5.0E+1	1.5E+1	5.7E+2
			800C	4.4E+4	1.8E+3	1.8E+3	1.5E+1	1.7E+3	8.5E+3	1.2E+3	1.9E+3	7.7E+2	2.6E+2	2.3E+4	8.0E+1	3.7E+5	7.0E+1	8.3E+2	2.7E+5		1.3E+5	7.1E+3	3.0E+0	2.7E+2	1.3E+3
			950C	1.0E+4	2.0E+3	1.7E+3	1.5E+1	1.8E+3	5.5E+3	1.3E+3	2.1E+3	4.6E+2	3.0E+2	2.5E+4	4.5E+1	4.1E+5	7.3E+1	1.1E+3	7.6E+3		4.9E+3	6.0E+3	2.0E+0	4.0E+2	6.8E+2
		63-88-06-121	AR	9.1E+3	1.8E+4	2.3E+4	2.1E+1	3.9E+1	2.0E+4		7.1E+3	1.8E+2	2.4E+2	5.4E+4	2.8E+2	4.1E+4	6.0E+2	3.8E+3	4.9E+3	3.1E+3	5.6E+4			3.3E+2	1.7E+3
			950C	1.9E+3	1.3E+4	4.1E+4	2.0E+1	5.4E+1	1.8E+4		1.1E+4	3.1E+2	2.7E+2	6.3E+4	2.1E+2	4.1E+4	2.6E+3	1.5E+4	6.1E+3	2.3E+2	6.3E+4			1.7E+3	1.4E+3
		66-00-11-355	AR	2.0E+1	2.0E+1		4.0E-1	2.6E+2		2.6E+2			1.7E+1	5.0E+0	2.2E+2		1.0E+3				8.3E+3		1.3E+1	1.1E+2	
			950C	9.0E+1	1.2E+3	7.5E+3	1.3E+1	3.7E+2	3.0E+3		4.7E+3	7.5E+2	1.7E+1		2.3E+2	3.1E+5	9.6E+2	5.4E+3	1.4E+3	1.6E+3	8.5E+3			1.0E+2	2.4E+3
		66-01-01-439	AR	3.0E+1	2.0E+1		4.0E-1	7.3E+0		3.2E+2	4.3E+2	3.1E+1	1.1E+0		1.2E+1		1.2E+2		6.3E+1	2.4E+2	1.8E+1	1.4E+2	3.5E+0	8.3E+0	6.4E+1
Metal Oxida-tion	Product	011589A	AR	1.6E+4	2.6E+3	4.7E+3	1.3E+1	3.5E+1	3.9E+3	7.4E+3	3.9E+2	1.3E+3	3.5E+3	3.7E+3	1.7E+2	2.3E+4	1.6E+1	2.0E+2	7.0E+3	7.9E+3	3.7E+3	2.1E+2	6.3E+2	6.0E+1	1.7E+2
			950C	1.0E+4	2.9E+3	4.7E+3	7.8E+1	3.9E+1	3.8E+3	2.1E+2	1.4E+2	4.9E+2	3.6E+3	3.6E+3	2.2E+1	2.3E+4	1.3E+1	1.7E+2	3.1E+3	7.8E+3	2.6E+3	4.4E+2	9.0E+2	4.2E+1	1.3E+1
		011608	AR	2.0E+3	2.7E+3		5.2E+0	8.5E+1	2.6E+3	4.2E+3	5.0E+0	2.9E+2	5.4E+3	5.9E+2	1.4E+1	7.2E+2	2.9E+1	1.0E+2	1.3E+3	8.0E+0	2.0E+3	4.0E+1	1.1E+3	7.1E+0	3.9E+1
			800C	9.1E+3	3.1E+3		7.7E+0	2.6E+2	5.4E+3	1.8E+2	3.2E+2	3.0E+2	5.1E+3	9.0E+2	9.3E+0	7.6E+2	8.7E+1	1.8E+2	2.8E+3	1.4E+3	3.9E+3	4.2E+2	2.1E+2	3.5E+1	4.6E+1
			950C	2.2E+3	2.6E+3		6.7E+0	6.4E+1	4.0E+3	2.5E+3	3.5E+2	1.6E+2	4.4E+3	9.1E+2	2.0E+1	9.1E+2	7.3E+1	2.3E+2	1.2E+3	8.0E+0	2.5E+3	4.6E+2	1.9E+3	2.0E+1	3.2E+1
		07221730	AR	1.0E+3	1.6E+3	8.1E+1	4.0E+0	5.0E+1	3.6E+3	3.5E+2	6.0E+1	2.2E+1	8.3E+1	3.9E+2	5.0E+0	3.5E+3	1.1E+0	9.3E+1	5.0E+1		4.0E+1	4.6E+1	1.9E+1	1.0E+1	8.8E+0
			950C	1.2E+3	1.8E+3	2.0E+2	4.0E+0	6.0E+1	4.1E+3	1.6E+2	1.2E+2	3.3E+1	1.0E+2	4.5E+2	4.0E+0	3.4E+3	9.0E+0	1.3E+2	9.8E+1		3.4E+2	5.5E+2	4.0E+1	9.0E+1	1.2E+1
		MT1490	AR	8.0E+1	5.0E+1	6.7E+2	7.4E+0	1.2E+2	1.9E+2	5.1E+2	3.0E+2	6.2E+2	5.1E+2	4.4E+3	1.8E+1	1.2E+2	4.7E+0	1.7E+2	4.0E+1		5.8E+1	3.0E+1	1.0E+2	1.7E+0	3.7E+1
			800C	8.8E+2	1.9E+2	4.5E+2	4.6E+0	9.2E+1	1.7E+2	1.1E+2	8.9E+2	2.0E+2	4.3E+2	8.0E+3	1.2E+1	9.2E+1	1.6E+1	4.9E+2	6.5E+1		5.5E+2	4.3E+1	5.6E+1	1.1E+1	1.8E+1
			950C	1.7E+2	1.0E+2	5.5E+2	5.1E+0	9.9E+1	3.0E+2	1.3E+2	3.5E+2	6.5E+1	4.1E+2	4.7E+3	5.8E+0	1.4E+2	7.1E+0	1.9E+2	2.3E+2		3.6E+2	3.2E+2	8.6E+1	2.1E+1	1.0E+1
		TS707001	AR	1.9E+3	1.4E+3	7.7E+1	1.0E+1	1.0E+1	1.3E+2	6.9E+2	1.6E+2	1.2E+2	7.0E+2	2.3E+2	3.4E+1	8.3E+1	1.0E+1	1.7E+2	1.0E+2	5.7E+2	1.0E+2		6.5E+1	3.6E+1	2.2E+1
			950C	1.4E+2	8.5E+2	9.6E+1	1.0E+1	1.0E+1	1.0E+2	8.0E+1	1.5E+2	3.2E+1	6.0E+2	2.4E+2	1.6E+1	9.6E+1	1.0E+1	1.0E+2	1.0E+2	5.5E+2	1.0E+2		1.1E+2	4.1E+1	1.2E+1
		UPOPLOT0003	950C	3.0E+1	3.0E+1	5.4E+1	1.3E+0	6.3E+0	1.0E+1	3.0E+1	4.1E+2	3.9E+1	6.5E+3	1.3E+3	1.8E+1	1.2E+1	1.9E+1	5.1E+3	1.0E+1	7.6E+1	7.2E+1	1.0E+1	1.3E+1	7.1E+0	1.9E+1
	By-product	ARF-102-85-114-1	AR	1.4E+2	3.0E+1	8.0E+0	2.1E+3	4.9E+1	1.8E+2	3.2E+2	2.6E+1	2.3E+1	4.1E+3	2.9E+2	6.2E+0	6.0E+1	3.7E+0	4.9E+1	5.0E+2	1.6E+3	8.9E+2	5.6E+1	5.6E+0	1.7E+1	2.0E+1
			950C	1.4E+3	1.3E+2	2.7E+2	1.4E+3	4.8E+1	5.0E+2	2.0E+2	1.5E+2	1.3E+1	5.1E+3	3.9E+2	4.8E+0	2.6E+2	4.2E+1	1.9E+2	4.0E+2	1.6E+3	8.8E+2	4.0E+2	1.2E+1	6.5E+1	1.5E+1
		TS707013	AR	8.1E+4	2.2E+2	2.9E+3	3.9E+0	1.2E+1	1.8E+3	5.6E+2	5.0E+3	1.0E+3	3.9E+3	1.5E+4	9.3E+1	1.3E+4	5.8E+1	2.6E+3	1.5E+4	8.2E+2	1.0E+4	5.7E+1	4.7E+2	5.0E+0	3.1E+2
			950C	1.8E+4	4.9E+2	4.4E+3	1.0E+1	2.0E+1	1.7E+3	2.4E+2	5.8E+3	5.8E+2	4.7E+3	1.7E+4	7.1E+0	1.3E+4	3.3E+1	2.4E+3	9.0E+3	8.5E+2	7.8E+3	5.6E+2	2.1E+2	6.9E+0	5.9E+1

Table A9-2. Representative Samples: Trace Element Analysis Data Arranged by Process Category and MIS Sample for All Conditions *(continued)*

Process Category	Process Sub-category	MIS Sample	Cond	Cl ppm	F ppm	Al ppm	Be ppm	B ppm	Ca ppm	C ppm	Cr ppm	Cu ppm	Ga ppm	Fe ppm	Pb ppm	Mg ppm	Mo ppm	Ni ppm	K ppm	Si ppm	Na ppm	S ppm	Ta ppm	W ppm	Zn ppm
Misc.	Misc.	64-85-12-1858	AR	7.5E+2	4.4E+3	4.1E+3	1.4E+1	2.1E+2	2.1E+4		2.2E+3	1.1E+2	1.2E+1	3.3E+4	8.5E+2	9.1E+3	3.3E+1	1.3E+3	7.2E+3	2.5E+2	6.5E+3			6.7E+0	2.5E+2
			950C	2.9E+2	2.6E+3	3.6E+3	1.1E+1	1.8E+2	1.5E+4		1.8E+3	2.2E+2	3.3E+1	2.8E+4	5.1E+2	7.5E+3	3.3E+2	1.8E+3	6.6E+3	5.0E+3	5.5E+3			1.8E+1	1.9E+2
		PPSL-365	600C	6.6E+1		3.1E+2	2.0E+0	4.0E+2	1.5E+2	4.7E+2	9.6E+2	5.6E+2	7.0E+0	8.1E+3	5.0E+1	6.8E+2	5.7E+1	5.4E+2	6.1E+2	3.0E+3	2.5E+3		6.9E+1	2.0E+2	2.7E+2
		PuF4-1	AR	1.6E+4	2.3E+5					3.9E+2															
			950C	1.1E+4	1.9E+5	5.4E+2	4.7E+1	6.3E+1	1.6E+2		3.9E+4	2.1E+2	4.5E+2	8.0E+4	1.9E+2	1.3E+3	1.3E+3	3.3E+5	1.5E+4	9.9E+2	1.4E+3			5.4E+2	1.2E+3
Mixed Actinide Ops.	Product	5501579	AR	6.4E+2		2.5E+1	3.5E+0	1.3E+2	3.0E+1	2.1E+2	7.8E+1	3.9E+2	1.0E+4	1.0E+3	7.9E+0	8.0E+0	6.4E+0	5.1E+2	2.9E+2	4.6E+2	3.0E+1	5.0E+1	5.0E+2	3.0E+1	1.4E+1
			950C	7.5E+2		1.8E+2	8.0E-1	1.5E+1	3.7E+1	1.3E+2	5.7E+1	5.6E+1	9.0E+3	6.9E+2	4.0E+0	8.7E+1	2.6E+0	1.3E+2	3.0E+2	9.6E+2	3.0E+1	5.0E+1	2.4E+2	3.0E+1	1.0E+1
		SCP711-46	AR	2.3E+2	7.0E+1	9.0E+0	5.2E+0	2.4E+1	4.5E+2	1.1E+4	1.6E+2	4.0E+0	1.9E+1	6.2E+2	1.5E+2	1.2E+2	3.9E+1	6.1E+2	3.3E+2	4.0E+1	4.3E+2	4.0E+1	1.8E+1	1.2E+1	7.0E+1
			950C	2.0E+1	4.0E+1	9.0E+0	5.0E+0	5.1E+1	7.0E+1	2.3E+2	1.1E+2	5.0E+0	1.4E+1	4.0E+2	3.2E+1	1.3E+2	3.1E+1	5.3E+2	3.4E+2	6.3E+1	4.8E+2	5.0E+1	3.4E+0	4.8E+1	6.1E+1
		SCP711-56	AR	1.1E+2	2.0E+1	8.9E+2	1.5E+1	3.8E+2	9.2E+2	4.1E+3	6.7E+1	4.3E+1	3.3E+0	4.9E+2	8.9E+1	2.3E+2	1.4E+1	1.9E+3	3.7E+2	6.7E+3	2.8E+3	4.0E+1	1.0E+1	9.7E+0	7.9E+1
	Misc.	CXL1685	AR	3.6E+2	7.0E+1	1.2E+1	4.0E-1	1.4E+0	4.0E+0	1.2E+3	6.2E+0	6.7E+0	5.4E+0	3.8E+1	2.0E-1	5.4E+0	6.0E-1	8.8E+0	6.0E+1	2.4E+1	1.7E+1	7.3E+1	2.4E-1	9.1E+0	6.0E-1
			950C	2.5E+1	7.0E+1	3.5E+1	4.0E-1	6.5E+0	4.4E+1	1.5E+1	1.7E+1	4.3E+0	1.6E+1	5.5E+1	2.0E-1	8.3E+1	2.9E+0	1.5E+1	6.6E+1	6.5E+1	1.7E+1	4.6E+1	1.1E+0	1.6E+1	6.5E-1
	By-product	053038	AR	9.0E+4	6.5E+3	7.5E+3	3.0E+2	1.1E+2	2.7E+4	1.2E+3	1.5E+4	4.4E+3	4.0E+3	4.6E+4	1.7E+3	5.2E+3	4.3E+1	4.4E+3	1.0E+4	1.4E+3	1.1E+4	3.4E+3	2.4E+2	2.2E+1	1.5E+4
			800C	5.9E+4	6.0E+3	5.1E+3	2.5E+2	6.3E+1	2.6E+4	1.5E+2	5.5E+3	1.8E+3	2.5E+3	2.4E+4	2.3E+2	4.7E+3	8.5E+1	2.8E+3	1.1E+4	5.1E+3	8.8E+3	2.7E+3	8.9E+1	1.8E+1	5.8E+3
			950C	4.5E+4	5.7E+3	7.8E+3	3.4E+2	1.5E+2	3.2E+4	2.3E+2	1.3E+4	1.1E+3	4.0E+3	3.4E+4	3.8E+1	8.3E+3	1.4E+2	5.8E+3	1.4E+4	1.8E+3	1.1E+4	3.9E+3	6.9E+1	1.4E+1	4.0E+3
		5501407	AR	2.2E+3	5.2E+2	1.0E+2	5.6E+2	2.1E+2	8.1E+1	3.2E+2	1.6E+3	2.4E+2	7.6E+3	2.2E+3	1.6E+1	3.0E+0	7.0E+0	4.3E+4	3.4E+2	5.0E+1	7.0E+1	4.0E+4	1.0E+2	2.0E+2	7.0E+0
			950C	1.6E+3	1.9E+1	3.0E+1	3.7E+2	2.3E+2	1.1E+2	7.0E+1	1.0E+3	1.5E+2	7.7E+3	1.0E+3	1.3E+0	4.0E+0	5.8E+0	2.8E+4	3.3E+2	5.0E+1	8.0E+1	2.8E+1	4.4E+1	5.8E+2	1.7E+1
		62750	AR	8.7E+2	6.0E+1	4.0E+2	1.6E+1	4.0E+0	2.8E+2	4.1E+2	6.2E+2	2.7E+2	5.9E+3	2.1E+3	1.5E+2	7.3E+1	2.5E+3	4.8E+2	4.1E+2	9.0E+2	6.1E+2	6.0E+1	3.4E+1	1.6E+1	2.6E+1
			800C	5.1E+2	4.0E+1	2.5E+2	2.0E-1	1.6E+2	6.5E+2	2.1E+2	2.0E+2	1.1E+2	7.2E+3	1.2E+3	1.8E+1	2.0E+0	3.8E+3	4.0E+2	4.4E+2	1.3E+3	9.1E+2		1.9E+1	2.2E+1	9.4E+0
			950C	2.1E+2	9.5E+1	7.8E+2	3.7E+1	1.6E+2	3.8E+2	2.5E+2	8.2E+2	1.8E+2	5.6E+3	3.2E+2	1.6E+1	2.0E+2	6.2E+2	6.8E+2	2.9E+2		4.0E+2	2.6E+2	1.1E+2	3.1E+1	2.8E+1
		669194	AR	8.1E+2	3.9E+1	3.3E+2	3.0E+3	3.0E+0	6.6E+1	1.9E+2	4.7E+2	7.3E+1	8.8E+2	4.9E+3	2.7E+2	3.0E+0	5.0E+1	4.2E+2	8.0E+1	5.0E+1	7.0E+1	9.5E+1	1.2E+2	4.0E+1	2.0E+1
			950C	1.4E+2	4.8E+1	1.2E+3	1.8E+3	4.1E+1	5.9E+2	1.6E+2	2.0E+2	1.3E+2	2.8E+2	9.5E+2	4.0E+1	5.2E+2	1.4E+1	3.0E+2	1.3E+3	6.0E+1	1.9E+3	3.0E+1	1.2E+1	2.1E+1	7.9E+1
		CAN92	AR	2.5E+3		3.0E+1	3.7E+3	2.8E+2	7.4E+0	2.1E+2	6.6E+2	2.2E+3	3.6E+3	4.0E+3	2.0E+2	4.0E+0	2.6E+1	5.5E+2	1.8E+2	5.0E+1	8.0E+1	3.6E+1	1.7E+1	3.3E+1	3.3E+2
			950C	8.6E+2	6.0E+1	1.6E+3	3.1E+3	2.5E+2	1.5E+2	4.9E+2	1.3E+2	6.0E+1	8.0E+2	1.2E+3	5.0E+0	1.3E+2	6.0E+0	1.6E+2	1.8E+3	1.4E+2	7.0E+1	6.0E+1	2.1E+1	5.0E+1	8.0E+0
		PSU-84-06-05	AR	7.2E+1	4.4E+1	9.7E+1	1.0E+1	1.0E+1	1.7E+2	3.6E+2	3.1E+2	2.1E+1	6.0E+0	3.3E+2	1.6E+2	5.1E+1	7.0E+1	4.3E+2	1.0E+2	1.0E+2	6.9E+2		1.0E+1	1.0E+1	5.5E+1
			950C	1.2E+1	3.1E+1	1.6E+2	1.2E+1	1.0E+1	1.7E+2	1.6E+2	3.1E+2	3.0E+1	1.1E+1	4.0E+2	1.2E+2	1.0E+2	7.4E+1	4.5E+2	1.0E+2	1.0E+2	6.7E+2		1.0E+1	1.0E+1	7.2E+1
		PuUOXBC05	AR						1.6E+4	2.3E+2		6.0E+4		1.6E+3		5.2E+4		3.7E+2	2.5E+3		1.5E+3				5.7E+4
			600C						1.9E+4	1.2E+2		9.0E+4	1.1E-1	1.5E+3		6.1E+4		5.0E+2	2.6E+3		1.4E+3				6.1E+4
			950C						1.9E+4	9.0E+1		1.9E+4	1.2E+3	1.7E+3		4.7E+4		4.5E+2	1.0E+3		1.4E+3				5.4E+4
Molten Salt Ops.	By-product	07242141A	950C	4.0E+1	1.6E+3	8.1E+3	8.0E+0	2.0E+1	3.0E+3		5.2E+3	1.1E+3	4.4E+3	2.7E+4	7.9E+0	8.0E+2	2.0E+2	2.7E+3	3.5E+3	3.4E+2	1.0E+3			1.3E+1	3.4E+1
		ARF-102-85-223	AR	1.1E+5	1.7E+2	7.6E+1	4.0E+0	9.5E+1	1.8E+2	7.4E+2	2.7E+2	2.8E+1	8.9E+1	2.7E+2	4.0E+1	8.6E+3	9.0E-1	1.5E+3	6.6E+4		4.8E+4	1.1E+2	1.2E+2	5.2E+1	9.6E+0
			600C	1.2E+5	1.1E+2	6.2E+1	4.0E+0	8.6E+1	2.4E+2	1.8E+2	2.7E+2	6.8E+1	8.5E+1	2.1E+2	5.9E+2	6.3E+3	9.1E-1	2.7E+3	4.9E+4	1.6E+3	3.7E+4	7.4E+1	1.8E+0	3.0E+1	1.5E+1
			950C	5.5E+4	6.1E+2	1.3E+2	4.0E+0	8.0E+0	3.3E+2	1.5E+3	1.2E+3	6.8E+1	6.3E+2	6.3E+2	5.1E+1	5.4E+3	4.5E+1	2.7E+3	1.9E+4	7.2E+2	1.5E+4	2.8E+2	1.0E+2	9.4E+1	1.8E+1
		ARF-102-85-295	AR	2.0E+5		2.0E+3	2.0E-1	6.1E+1	1.2E+3	3.6E+3	3.5E+3	1.9E+3	4.0E+3	2.5E+4	1.3E+3	6.7E+4	9.5E+1	2.0E+4	5.4E+4	2.9E+3	3.6E+4	2.2E+2	3.0E+2	8.0E+1	3.2E+2
			600C	1.8E+5						7.0E+1															
			950C	7.7E+4		5.5E+3	1.6E+0	9.3E+1	9.4E+2	1.8E+2	1.3E+4	1.1E+3	7.0E+3	5.4E+4	1.5E+2	4.0E+4	1.3E+2	4.1E+4	2.3E+4	2.8E+3	2.4E+4	1.7E+2	1.3E+2	6.0E+1	3.4E+2
		ARF-102-85-365	AR	1.1E+4	1.7E+2	3.0E+1	2.8E+0	4.4E+1	1.1E+2	4.3E+3	1.7E+2	7.9E+1	1.0E+3	3.7E+2	2.4E+2	1.1E+4	1.5E+0	5.1E+2	5.8E+4	5.0E+2	4.4E+4	2.0E+2	3.0E+3	1.3E+2	2.2E+1
			950C	3.8E+4	1.1E+3	1.7E+2	2.6E+0	5.0E+0	2.5E+2	1.1E+3	3.8E+2	1.0E+2	1.5E+3	5.0E+2	8.0E+1	5.6E+3	3.8E+0	1.6E+3	2.2E+4	5.3E+2	1.6E+4	6.0E+2	4.5E+3	1.6E+3	1.6E+1
		520610020	AR	1.7E+5	3.0E+2	1.1E+4	2.0E-1	4.8E+1	1.1E+5	1.8E+3	4.4E+3	4.1E+2	6.0E+2	9.5E+3	2.1E+2	6.6E+4	4.1E+2	1.5E+4	1.8E+4	1.9E+3	1.3E+4	5.5E+2	2.4E+1	8.3E+0	3.1E+2
			800C	1.3E+5	1.4E+3	1.6E+4	2.0E-1	6.0E+1	9.4E+4	2.6E+2	7.1E+3	6.9E+2	1.1E+3	2.2E+4	3.4E+1	8.5E+4	6.4E+2	3.1E+4	1.8E+4	2.0E+3	1.3E+4	5.6E+2	2.1E+1	2.0E+0	5.0E+2
			950C	6.7E+4	9.1E+2	1.8E+4	1.4E+0	2.0E+2	8.6E+4	4.6E+2	8.0E+3	6.6E+2	1.2E+3	1.5E+4	6.0E+1	6.7E+4	8.4E+2	2.0E+4	1.6E+4	1.1E+3	1.0E+4	1.8E+3	7.2E+0	8.6E+0	5.2E+2
		ATL27960	AR			9.6E+2	2.0E+0	1.0E+1	1.0E+3	2.0E+4	6.1E+2			8.4E+2	3.8E+2	6.2E+2	8.0E+1	5.2E+2	1.4E+4	6.5E+1	4.4E+4		1.0E-1	3.0E-1	
			950C			1.0E+3	2.0E+0	1.3E+1	1.0E+3	1.0E+4	8.2E+2			8.0E+2	2.1E+2	4.7E+2	1.3E+2	6.2E+2	1.2E+4	4.4E+2	3.9E+4		1.0E-1	3.0E-1	
		C00024A	AR	5.7E+4	4.6E+2	4.9E+2	2.1E+3	3.8E+1	2.1E+2	2.0E+2	4.5E+3	1.1E+2	2.4E+4	7.2E+3	6.4E+1	1.1E+4	2.3E+1	4.7E+2	2.6E+4	1.0E+3	2.2E+4	3.1E+2	1.9E+2	3.1E+2	2.7E+2
			800C	1.9E+4	5.4E+2	2.2E+3	2.7E+3	2.7E+1	1.1E+3	1.2E+3	1.4E+3	6.9E+1	1.5E+4	3.5E+3	2.6E-1	8.8E+3	7.0E+1	3.7E+2	1.7E+4	1.3E+2	1.1E+4	3.4E+2	2.3E+1	9.4E+1	3.4E+1
			950C	8.2E+3	4.0E+2	5.1E+2	4.8E+3	4.2E+1	4.7E+2	3.2E+2	2.2E+3	5.9E+1	2.2E+4	2.3E+3	2.2E+1	9.3E+3	3.3E+1	5.2E+2	1.7E+4	4.4E+3	1.1E+4	6.2E+2	2.7E+2	8.2E+1	1.1E+2
		C00695	AR	9.0E+4	2.9E+2	1.7E+2	1.0E+1	2.0E+2	3.7E+2	8.0E+1	2.2E+2	2.2E+1	7.8E+1	8.2E+2	1.8E+1	2.0E+4	2.3E+0	2.7E+3	3.5E+4	1.6E+3	7.4E+4	7.7E+2	8.3E+0	3.8E+1	6.2E+0
			800C	7.0E+4	3.4E+2	3.0E+1	4.0E+0	5.3E+1	2.1E+2	1.6E+2	6.4E+2	2.8E+1	1.2E+2	6.5E+2	1.0E+1	9.4E+3	9.9E+0	7.1E+3	2.8E+4	1.6E+3	2.2E+4	3.3E+2	5.6E+0	1.2E+2	9.0E+0
			950C	5.5E+4	3.0E+2	6.2E+1	4.0E+0	7.1E+1	1.5E+3	1.4E+2	3.6E+2	1.8E+1	5.5E+1	2.5E+3	9.6E+0	7.2E+3	1.7E+1	3.9E+3	1.8E+4	1.6E+3	1.8E+4	1.8E+3	1.5E+1	8.8E+1	7.0E+0
		C06032A	AR	1.6E+5	8.6E+2	1.0E+2	1.0E+1	8.4E+1	2.5E+2	4.4E+2	1.7E+2	3.8E+1	1.7E+3	2.7E+2	4.0E+1	2.2E+4	1.0E+1	6.1E+2	4.1E+4	4.5E+3	2.3E+4		2.6E+2	5.2E+2	1.0E+1
			950C	5.7E+4	5.0E+2	1.1E+2	1.0E+1	1.1E+2	1.1E+2	2.7E+2	2.4E+2	4.5E+1	2.2E+3	3.0E+2	4.7E+1	2.4E+4	1.0E+1	6.0E+2	1.1E+5	2.7E+3	5.3E+4		5.0E+2	6.1E+2	1.4E+1
			950C	4.6E+4	7.2E+2	1.3E+4	1.0E+1	1.0E+2	2.1E+2	9.5E+1	5.7E+2	5.0E+1	5.8E+3	1.5E+3	1.2E+1	2.1E+4	1.1E+1	1.8E+3	2.8E+4	1.4E+4	1.3E+4		3.8E+2	4.4E+2	1.0E+1
		CLLANL025	AR	1.0E+5	2.1E+2	3.4E+3	5.0E+0	3.0E+1	6.1E+1	2.0E+2	1.9E+2	2.1E+1	3.6E+2	4.4E+2	2.2E+1	5.5E+3	1.0E+1	9.2E+2	5.1E+4	3.8E+3	2.0E+4	8.4E+1	4.8E+1	6.2E+2	1.0E+1
			950C	5.9E+4	1.5E+2	3.4E+3	5.0E+0	3.6E+1	6.4E+1	1.3E+2	1.8E+2	3.7E+1	3.5E+2	6.1E+2	1.4E+1	3.8E+3	1.0E+1	5.4E+2	2.9E+4	4.3E+3	1.0E+4	1.8E+2	9.2E+1	7.4E+2	1.0E+1
		PMAXBS	800C	5.2E+4	2.6E+2	2.8E+2	1.3E+1	1.9E+1	1.9E+2	6.2E+1	3.1E+2	7.5E+1	2.5E+4	3.4E+3	4.0E+1	2.4E+3	2.3E+1	3.5E+2	3.5E+4	4.0E+2	1.9E+4	2.4E+2	2.3E+2	1.8E+1	1.6E+1

Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

Table A9-3. Nonrepresentative Samples: Trace Element Analysis Data Arranged by Process Category and MIS Sample for All Conditions

Process Category	Process Subcategory	Item Id	Cond	Cl wt%	F wt%	Al wt%	Be wt%	B wt%	Ca wt%	C wt%	Cr wt%	Cu wt%	Ga wt%	Fe wt%	Pb wt%	Mg wt%	Mo wt%	Ni wt%	K wt%	Si wt%	Na wt%	S wt%	Ta wt%	W wt%	Zn wt%
Mixed Actinides	Misc	MISNE2	950C	0.09	0.02	0.02	0.00	0.08	0.60	0.01	0.02	0.00	0.15	0.073	0.00	0.10	0.02	0.01	0.01	0.02	0.04	0.36	0.00	0.00	0.00
Mixed Actinides	Misc	MISNE2	AR	0.02	0.11	0.02	0.00	0.11	0.78	0.05	0.01	0.00	0.16	0.074	0.00	0.09	0.02	0.01	0.01	0.02	0.04	0.51	0.07	0.04	0.00
Misc	Misc	MISNE4	950C			0.33	0.00	0.01	0.99		0.38	0.06	0.21	0.682	0.00	6.37	0.02	0.37	1.31	0.02	0.85	0.04	0.37	0.13	0.01

Note: Values reported as "below detectable limits" were entered as the detection limit. Empty cells indicate that a sample was not measured for a particular element.

Table A9-4. Representative Samples: Percent Change in Concentration for Selected Elements After Calcination at 950°C

Process Category	Process Subcategory	MIS Sample	Condition	Cl (wt%)	F (wt%)	Ca (wt%)	Mg (wt%)	Na (wt%)	K (wt%)
Aqueous Processing	Byproduct	07242201A	950C	0.39	1.27	0.78		0.27	0.56
		07242201A	AR	1.28	4.97	0.70		0.05	1.06
		% change		70%	74%	-12%		-438%	47%
		39-01153A	950C	1.01		0.55	40.70	0.49	0.76
		39-01153A	AR	0.15		0.14	20.80	0.02	0.60
		% change		-573%		-300%	-96%	-2800%	-26%
		63-88-06-121	950C	0.19	1.30	1.83	4.13	6.27	0.61
		63-88-06-121	AR	0.91	1.77	2.03	4.14	5.55	0.49
		% change		79%	27%	10%	0%	-13%	-25%
		ARF-102-85-355	950C	0.74	0.36				
		ARF-102-85-355	AR	6.10	2.15				
	Product	% change		88%	83%				
		07161856	950C		0.10				
		07161856	AR		0.50				
		% change			80%				
		1000089	950C	0.26			1.19		
		1000089	AR	0.34			0.55		
		% change		24%			-116%		

Note: % change = $(1 - wt\%_{950}/wt\%_{AR})$

Table A9-4. Representative Samples: Percent Change in Concentration for Selected Elements after Calcination at 950°C *(continued)*

Process Category	Process Subcategory	MIS Sample	Condition	Cl (wt%)	F (wt%)	Ca (wt%)	Mg (wt%)	Na (wt%)	K (wt%)
Metal Oxidation	Byproduct	TS707013	950C	1.82			1.32	0.78	0.90
		TS707013	AR	8.10			1.26	1.02	1.51
		% change		78%			-5%	24%	40%
	Product	011589A	950C	1.03	0.29	0.38	2.26	0.26	0.31
		011589A	AR	1.63	0.26	0.39	2.27	0.37	0.70
		% change		37%	-12%	2%	0%	30%	56%
		011608	950C		0.26	0.40		0.25	0.12
		011608	AR		0.27	0.26		0.20	0.13
		% change			4%	-54%		-25%	8%
		07221730	950C			0.41	0.34		
		07221730	AR			0.36	0.35		
		% change				-13%	1%		
Miscellaneous	Miscellaneous	64-85-12-1858	950C		0.26	1.50	0.75	0.55	0.66
		64-85-12-1858	AR		0.44	2.10	0.91	0.65	0.72
		% change			41%	29%	18%	15%	8%
		PuF4-1	950C	1.10	19.00				
		PuF4-1	AR	1.60	22.90				
		% change		31%	17%				
Mixed Actinide Operations	Byproduct	053038	950C	4.45	0.57	3.20	0.83	1.10	1.40
		053038	AR	9.00	0.65	2.70	0.52	1.10	1.00
		% change		51%	12%	-19%	-60%	0%	-40%
		PuUOXBC05	950C			1.90	4.70		0.10
		PuUOXBC05	AR			1.60	5.20		0.25
		% change				-19%	10%		60%

Table A9-4. Representative Samples: Percent Change in Concentration for Selected Elements in MIS Samples After Calcination at 950°C *(continued)*

Process Category	Process Subcategory	MIS Sample	Condition	Cl (wt%)	F (wt%)	Ca (wt%)	Mg (wt%)	Na (wt%)	K (wt%)
Molten Salt Operations	Byproduct	520610020	950C	6.70		8.64	6.73	1.03	1.65
		520610020	AR	16.85		11.07	6.62	1.35	1.75
		% change		60%		22%	-2%	23%	6%
		ARF-102-85-223	950C	5.50			0.54	1.47	1.87
		ARF-102-85-223	AR	11.20			0.86	4.78	6.62
		% change		51%			37%	69%	72%
		ARF-102-85-295	950C	7.70			4.04	2.36	2.33
		ARF-102-85-295	AR	20.40			6.75	3.65	5.44
		% change		62%			40%	35%	57%
		ARF-102-85-365	950C	3.81			0.56	1.63	2.17
		ARF-102-85-365	AR	1.13			1.09	4.43	5.77
		% change		-237%			49%	63%	62%
		ATL27960	950C					3.90	1.20
		ATL27960	AR					4.40	1.40
		% change						11%	14%
		C00024A	950C	0.82			0.93	1.06	1.72
		C00024A	AR	5.68			1.15	2.19	2.62
		% change		86%			19%	52%	34%
		C00695	950C	5.50			0.72	1.77	1.75
		C00695	AR	9.00			2.00	7.43	3.47
		% change		39%			64%	76%	50%
		C06032A	950C	5.73			2.44	5.27	10.59
		C06032A	AR	16.20			2.23	2.25	4.14
		% change		65%			-9%	-134%	-156%
		CLLANL025	950C	5.90			0.38	1.01	2.90
		CLLANL025	AR	10.00			0.55	1.96	5.12
		% change		41%			30%	49%	43%

Note: For samples with both "powder" and "chunk" portions, only data for "powder" portion is shown.

Appendix 10, Chloride Salt Mass Balance and Soluble Constituent Data

Table A10-1.Representative Samples: Results of "Free Chloride" Calculation for Select MIS Samples

Process Category	Process Subcategory	MIS Sample	Cl (wt%)	K (wt%)	Na (wt%)	Free Cl (mol%)	Free Cl (wt%)
Aqueous Processing	Byproduct	07032282A	0.60	0.12	0.25	0.003	0.11
		07242201A (powder)	0.39	0.56	0.27		0.00
		39-01153A	1.01	0.76	0.49		0.00
		ARF-102-85-355	0.74	0.01	0.03	0.019	0.68
	Product	1000089	0.26	0.06	0.10	0.001	0.05
		CXLOX091802	2.20	2.40	0.22		0.00
Metal Oxidation	Byproduct	ARF-102-85-114-1	0.14	0.04	0.09		0.00
		TS707013	1.82	0.90	0.78		0.00
	Product	011589A	1.03	0.31	0.26	0.010	0.35
Miscellaneous	Miscellaneous	PuF4-1	1.10				0.00
Mixed Actinide Operations	Byproduct	053038	4.45	1.40	1.10	0.042	1.50
Molten Salt Operations	Byproduct	520610020	6.70	1.65	1.03	0.102	3.60
		ARF-102-85-223	5.50	1.87	1.47	0.044	1.50
		ARF-102-85-295 (powder)	7.70	2.33	2.36	0.055	2.00
		ARF-102-85-365	3.81	2.17	1.63		0.00
		C00024A	0.82	1.72	1.06		0.00
		C00695	5.50	1.75	1.77	0.033	1.20
		C06032A (chunks)	4.63	2.78	1.34	0.001	0.04
		C06032A (powder)	5.73	10.59	5.27		0.00
		CLLANL025	5.90	2.90	1.01	0.048	1.70

Notes:
Included are representative MIS Samples with greater than 0.25 wt% chlorine.
It is assumed that potassium and sodium are present as KCl and NaCl only.
Free Chloride (mol%) = (Cl mol%) – (K mol%) – (Na mol%)
mol% = Wt%/Atomic Wt

Table A10-2. Representative Samples: Comparison of Trace Element Analysis Data from Leach and Dissolution of MIS Samples in Various Conditions

Gas Generation Behavior	MIS Sample	Analysis Type	Cond	Cl% (wt%)	Na (wt%)	K (wt%)	F (wt%)	Ca (wt%)	Cr (wt%)	Fe (wt%)	Ni (wt%)	Mg (wt%)	Mn (wt%)	Mo (wt%)
Cl Containing Materials (Hydrogen Generating Impure Oxides)	011589A	Leach	950C	1.02	0.261	0.326	0.021	0.251	0.000	0.001	0.000	0.086	0.000	0.000
	011589A	Total	950C	1.03	0.258	0.310	0.289	0.378	0.014	0.362	0.017	2.259	0.005	0.001
	011589A	(Leach/Total) %		99.34	101.035	105.282	7.384	66.288	N/A	0.276	N/A	3.815	N/A	N/A
	053038	Leach	950C	4.18	0.784	1.226	0.053	1.583	0.095	0.000	0.008	0.038	0.001	0.000
	053038	Total	950C	4.45	1.100	1.400	0.570	3.200	1.300	3.400	0.580	0.830	0.096	0.014
	053038	(Leach/Total) %		93.86	71.302	87.545	9.216	49.479	7.320	N/A	1.396	4.635	1.042	N/A
	520610020	Leach	750C	12.55	1.528	2.062	0.010	5.526	0.474	0.000	0.000	0.001	0.000	0.082
	520610020	Total	800C	13.40	1.300	1.800	0.140	9.400	0.710	2.200	3.100	8.500	0.060	0.064
	520610020	(Leach/Total) %		93.62	117.561	114.559	7.059	58.782	66.827	N/A	N/A	0.009	N/A	128.253
	ARF-102-85-223	Leach	750C	9.58	2.955	5.010	0.008	0.002	0.020	0.000	0.097	0.157	0.000	0.002
	ARF-102-85-223	Total	600C	12.00	3.735	4.900	0.011	0.024	0.027	0.021	0.270	0.630	0.001	0.000
	ARF-102-85-223	(Leach/Total) %		79.84	79.122	102.247	76.648	8.419	74.551	N/A	35.785	24.940	N/A	N/A
	ARF-102-85-295	Leach	750C	9.05	2.919	3.628	0.016	0.064	0.018	0.000	0.255	0.157	0.010	0.006
	ARF-102-85-295	Total	950C	7.70	2.358	2.327		0.094	1.330	5.403	4.090	4.038	0.118	0.013
	ARF-102-85-295	(Leach/Total) %		117.55	123.796	155.959	N/A	68.085	1.349	N/A	6.239	3.900	8.483	46.007
	ARF-102-85-365	Leach	750C	6.89	2.262	3.384	0.004	0.000	0.060	0.000	0.004	0.050	0.001	0.070
	ARF-102-85-365	Total	950C	3.81	1.632	2.169	0.108	0.025	0.038	0.050	0.160	0.556	0.004	0.000
	ARF-102-85-365	(Leach/Total) %		180.78	138.585	156.050	3.616	N/A	159.441	N/A	2.417	9.037	23.256	N/A
	C00695	Leach 800/950C Mix		6.40	2.074	2.074	2.702	0.008	0.014	0.006	0.000	0.034	0.174	0.000
	C00695	Total	800C	7.00	2.217	2.760	0.034	0.021	0.064	0.065	0.710	0.943	0.004	0.001
	C00695	(Leach/Total) %		91.49	93.583	97.894	24.168	67.297	9.246	N/A	4.723	18.408	N/A	101.010
	C06032A	Leach	950C	8.10	2.903	4.100	0.010	0.000	0.001	0.000	0.000	0.218	0.000	0.000
	C06032A	Total	950C	4.63	1.342	2.784	0.072	0.021	0.057	0.153	0.183	2.149	0.005	0.001
	C06032A	(Leach/Total) %		174.92	216.323	147.277	14.023	N/A	2.122	N/A	N/A	10.152	N/A	N/A
	CLLANL025	Leach	750C	5.25	1.910	2.620	0.002	0.000	0.392	0.000	0.001	0.062	0.000	0.354
	CLLANL025	Total	950C	5.90	1.010	2.901	0.015	0.006	0.018	0.061	0.054	0.381	0.001	0.001
	CLLANL025	(Leach/Total) %		88.97	189.171	90.342	12.888	N/A	N/A	N/A	1.838	16.211	N/A	N/A
	PMAXBS	Leach	750C	6.17	1.933	2.711	0.001	0.006	0.000	0.000	0.002	0.016	0.002	0.000
	PMAXBS	Total	800C	5.20	1.925	3.515	0.026	0.019	0.031	0.342	0.035	0.236	0.004	0.002
	PMAXBS	(Leach/Total) %		118.59	100.414	77.117	3.873	31.014	N/A	N/A	5.653	6.658	56.121	N/A
High Purity Oxide	TS707001	Leach	950C	0.04	0.006	0.008	0.011	0.024	0.000	0.001	0.000	0.002	0.001	0.000
	TS707001	Total	950C	0.01	0.010	0.010	0.085	0.010	0.015	0.024	0.010	0.010	0.002	0.001
	TS707001	(Leach/Total) %			N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Other Materials	07242201A	Leach	950C	0.27	0.257	0.344	0.095	0.274	0.004	0.002	0.013	0.023	0.006	0.000
	07242201A	Total	950C	0.39	0.274	0.561	1.270	0.778	0.141	0.776	1.212	0.229	0.019	0.002
	07242201A	(Leach/Total) %		69.90	93.846	61.375	7.443	35.173	2.711	0.247	1.105	10.047	29.904	N/A
Average		**(Leach/Total) %**		**110%**	**110%**	**110%**	**7%**	**48%**				**7%**		

Table A10-3. Mass Balance of Major Soluble Impurities Assuming That the Cations Are Present as Chlorides

Gas Generation Category	MIS Sample	Mg (mol)	Ca (mol)	Na (mol)	K (mol)	Cl (mol)	Cations (mol)	Cl Excess (mol)
Cl Containing Materials (Hydrogen Generating Impure Oxides)	011589A	3.5E-03	6.3E-03	1.1E-02	8.3E-03	0.029	0.029	-0.001
	053038	1.6E-03	4.0E-02	3.4E-02	3.1E-02	0.118	0.107	0.011
	520610020	3.3E-05	1.4E-01	6.6E-02	5.3E-02	0.354	0.257	0.097
	ARF-102-85-223	6.5E-03	5.0E-05	1.3E-01	1.3E-01	0.270	0.263	0.007
	ARF-102-85-295	6.5E-03	1.6E-03	1.3E-01	9.3E-02	0.255	0.228	0.027
	ARF-102-85-365	2.1E-03	0.0E+00	9.8E-02	8.7E-02	0.194	0.187	0.007
	C00695	7.1E-03	3.4E-04	9.0E-02	6.9E-02	0.181	0.167	0.014
	C06032A	9.0E-03	0.0E+00	1.3E-01	1.0E-01	0.228	0.240	-0.012
	CLLANL025	2.5E-03	0.0E+00	8.3E-02	6.7E-02	0.148	0.153	-0.005
	PMAXBS	6.5E-04	1.5E-04	8.4E-02	6.9E-02	0.174	0.154	0.020
High Purity Oxide	TS707001	6.5E-05	5.9E-04	2.5E-04	2.0E-04	0.001	0.001	0.000
Other Materials	07242201A	9.4E-04	6.8E-03	1.1E-02	8.8E-03	0.008	0.028	-0.020

Note: The moles of cations were present assuming that the major soluble cations (Mg, Ca, Na and K) were present as chloride salts. Moles of excess chlorine is the moles of soluble chloride present in the solution minus the number of moles cations. The negative values may indicate a limited amount of a nonchloride compounds, such as CaO, may also have dissolved.

Appendix 11 Shelf-life Surveillance Representative Samples

MIS Sample	Source Site	Gas Generation Category*	Surveillance Data Reference
011589A	RFETS	Cl Containing Materials	LA-UR-09-07151
	RFETS	Cl Containing Materials	LA-UR-09-07152
053038	RFETS	Cl Containing Materials	LA-UR-09-07153
07032282A	RFETS	Cl Containing Materials	LA-UR-09-07169
520610020	RFETS	Cl Containing Materials	LA-UR-09-07154
ARF-102-85-223	HANFORD	Cl Containing Materials	LA-UR-09-07144
ARF-102-85-295	HANFORD	Cl Containing Materials	LA-UR-09-07174
	HANFORD	Cl Containing Materials	LA-UR-09-07175
ARF-102-85-355	HANFORD	Cl Containing Materials	LA-UR-09-07158
ARF-102-85-365	HANFORD	Cl Containing Materials	LA-UR-09-07159
C00024A	RFETS	Cl Containing Materials	LA-UR-09-07163
C00695	RFETS	Cl Containing Materials	LA-UR-09-07166
C06032A	RFETS	Cl Containing Materials	LA-UR-09-07143
CLLANL025	RFETS	Cl Containing Materials	LA-UR-09-07139
PMAXBS	LANL	Cl Containing Materials	LA-UR-09-07177
TS707013	RFETS	Cl Containing Materials	LA-UR-09-07167
5501579	RFETS	High Purity Oxide	LA-UR-09-07136
ARF-102-85-114-1	HANFORD	High Purity Oxide	LA-UR-09-07173
BLO-39-11-14-004	HANFORD	High Purity Oxide	LA-UR-09-07146
MISSTD-1	LANL	High Purity Oxide	LA-UR-09-07180
PBO-47-09-012-023	HANFORD	High Purity Oxide	LA-UR-09-07157
PEOF1	LANL	High Purity Oxide	LA-UR-09-07178
TS707001	RFETS	High Purity Oxide	LA-UR-09-07135
07242141A	RFETS	Other Materials	LA-UR-09-07156
07242165A	RFETS	Other Materials	LA-UR-09-07155
07242201A	RFETS	Other Materials	LA-UR-09-07140
1000089	RFETS	Other Materials	LA-UR-09-07150
41-85-08-1379B	Hanford	Other Materials	LA-UR-09-07170
5501407	RFETS	Other Materials	LA-UR-09-07141
63-88-06-121	Hanford	Other Materials	NA
64-85-12-1858	HANFORD	Other Materials	LA-UR-09-07171
66-00-11-355	HANFORD	Other Materials	LA-UR-09-07161
66-01-01-439	HANFORD	Other Materials	LA-UR-09-07148
669194	RFETS	Other Materials	LA-UR-09-07138
07161856	RFETS	Other Materials	LA-UR-09-07134
CAN92	RFETS	Other Materials	LA-UR-09-07162
MT-1490	RFETS	Other Materials	LA-UR-09-07137
PSU-84-06-05	HANFORD	Other Materials	LA-UR-09-07147