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LANL Phase 3 Container Loading in Support of the Fire-Induced Pressure Response and Failure Characterization of PCV/SCV/3013 Containers

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1.0 Introduction

The PCV/SCV/3013 Thermal Test Program is intended to determine the pressure response of packaged 3013 containers when exposed to a facility fire in the K-Area Complex (KAC) at the Savannah River Site. The fire tests support the KAC safety basis and must be bounding of the maximum pressure that would result from any 3013 container exposed to a KAC facility fire. The testing program has the following objectives [1, 2]:

1. "To increase understanding of the pressure and temperature conditions at which failure occurs
2. To determine the allowable moisture for which failure will not occur under specified fire conditions, and
3. To increase understanding regarding the effects of salts on the pressurization, corrosion, and potential failure of the container."

The most important parameters affecting the pressure response are the free gas volume within the packaged 3013 container and the amount of moisture adsorbed on the packaged materials[3]. Control of these parameters in the preparation of test containers is important for understanding the results that are obtained. In addition, the payload materials must have similar thermal characteristics as actual materials and composition in terms of the major impurities present. Many of the 3013 containers in the storage population have percent level concentrations of chloride salt impurities in the oxide material matrix. Having those impurities present in the payload materials for the thermal testing ensures that effects due to high-temperature reactions and corrosion are assessed.

A set of twelve welded 3013 containers was prepared at Los Alamos National Laboratory for thermal testing in the PCV/SCV/3013 Thermal Test Program. Aluminum oxide was selected as the surrogate for plutonium oxide based on its thermal characteristics. However, aluminum oxide has a lower packing fraction than plutonium oxide (ratio of material volume to total volume; 0.38 for aluminum oxide versus 0.5 for typical 3013 packaged material); therefore, stainless steel filler material was used to occupy some of the gas volume. Chloride salts were added to some of the test containers to determine whether these components affect the container or pressure response. The loading conditions for the test containers were determined by a calculation and included in the loading test plan approved by the Materials Identification and Surveillance (MIS) Working Group [3, 4]. A reasonable estimate for the minimum free gas volume was obtained based on DOE-STD-3013-2018 assuming a packing fraction of 0.5 [3, 5]. An analysis of the packaging data in the Integrated Surveillance Program database and results from the destructive evaluation program shows that the calculated minimum free gas volumes are reasonable. The loading conditions (aluminum oxide payload, filler, water, and salt) are specified for each test container based on the minimum free gas volume calculated for the storage population [3, 4].

This report documents the materials used to load containers for the PCV/SCV/3013 Thermal Test Program and the methods for preparation, handling, characterization, and storage. The actual loading conditions for each container are presented and compared with those specified in the calculation and the test plan[4].

2.0 Materials and Methods

The test containers prepared for the 3013 thermal tests were designed to evaluate a 3013 container at the minimum free gas volume with a maximum water content that bounds the current inventory and intermediate water contents that may result in no failure. The test containers were prepared using actual 3013 container components loaded with a surrogate payload. The loading conditions were documented in a test plan and approved by the MIS working group. Two versions of 3013 containers are being tested: a Machined version and a Flowform version. Aluminum oxide was chosen for use as the payload material. This material was provided by the manufacturer in a similar mesh size to plutonium oxide. The test containers each had moisture added to the payload. The specifications for the mass of moisture include a conservative 20% margin to the best moisture values for the population of 3013 containers in KAC in order to account for uncertainties in sampling and in the measurement itself. This results in moisture levels of 6, 12, and 18 grams, which bound the best moisture measurements of 5, 10, and 15 grams. Salts are added to two containers in each of the Machined and Flowform container sets to investigate high-temperature corrosion and to determine whether the presence of salts affects the performance of a container in a fire scenario. The salt used in the test is a mixture of sodium chloride, potassium chloride, and calcium chloride. The ratio of salts by mass is 0.48:0.48:0.04. Because aluminum oxide has a lower packing fraction than plutonium oxide, stainless steel filler material in the form of machined disks was added to the containers to achieve the desired minimum free gas volume. The resulting twelve 3013 container loading configurations selected for the test matrix are given in Table 1.

Table 1. Loading Specifications for Fire Test Containers [4]

	Configuration Outer/Inner/ CC	Mass Al_2O_3 (g)	Mass SS Disk 1 (Outside CC) (g)	Mass SS Disk 2 (Inside CC) (g)	Mass Salt (g)	Mass Water (g) Volume Water (ml)	Total Mass (g)
1*	EPD/BTIC/BTCC	1,572	0	3,445	0	0	12,261
2	EPD/BTIC/BTCC	1,572	0	3,445	0	12	12,273
3	EPD/BTIC/BTCC	1,572	0	3,445	0	18	12,279
4	EPD/BTIC/BTCC	1,572	0	3,445	0	6	12,267
5	EPD/BTIC/BTCC	1,107	0	2,608	462	6	11,427
6	EPD/BTIC/BTCC	642	0	1,772	925	12	10,595
7*	ARIES/ARIES/Cogema	1,958	1,029	1,089	0	0	9,673
8	ARIES/ARIES/Cogema	1,958	1,029	1,089	0	6	9,679
9	ARIES/ARIES/Cogema	1,958	1,029	1,089	0	6	9,679
10	ARIES/ARIES/Cogema	1,958	1,029	1,089	0	12	9,685
11	ARIES/ARIES/Cogema	1,493	1,029	253	462	6	8,840
12	ARIES/ARIES/Cogema	734	1,029	0	925	12	8,298

* Indicates containers without added moisture for equipment testing and validation

2.1 Storage Containers

The 3013 containers in storage at KAC originated at five different sites: Rocky Flats (RFETS), Lawrence Livermore National Laboratory (LLNL), Hanford Site, Savannah River Site (SRS) and

Los Alamos National Laboratory (LANL). Variations exist in the packaging configurations used by the sites as shown in Table 2. Two versions of the 3013 outer container were used to package materials in storage at KAC: a machined outer 3013 container produced by Westinghouse Engineered Products Division (EPD) and a flowform outer container produced by Dynamic Flowform. Both types of outer 3013 containers have the same dimensions, tolerances and minimum inner volume $V_{OC,I}$, but the containers manufactured by the Flowform process have higher residual stresses and tensile strengths due to cold work from forming. The minimum unoccupied volume $V'_{OC,U}$ within each container set (from Table B-2 of DOE-STD-3013-2018) varies based on the geometry[3, 5]. Both outer container types were included in the tests. Based on the available container parts, five Hanford Machined container sets and five LANL Flowform containers sets were prepared for testing. One additional container of each type was prepared for equipment testing and validation. Drawings of the container sets are provided in Appendix 1. The container sets are inverted in the drawing to show the axial gap for the drilling process.

Table 2. Components of the 3013 Container Sets, Interior Volume of the Outer Container ($V_{OC,I}$), and Minimum Unoccupied Volume of the Outer Container ($V'_{OC,U}$) [3, 5]

Packaging Site	Outer Container	Inner Container	Convenience Container	$V_{OC,I}$ (cm^3)	$V'_{OC,U}$ (cm^3)
RFETS	Machined	BNFL	BNFL	2596	2168
LLNL	Machined	BNFL	BNFL	2596	2168
Hanford	Machined	BTIC	Hanford BTCC	2596	2155
SRS	Machined	BTIC	SRS BTCC	2596	2248
LANL	Flowform	ARIES IC	Food pack can	2596	2432
LANL-ARIES	Flowform	ARIES IC	Cogema Short Cogema	2596	2382 2385*

*Calculated in ref. [3]

The Flowform container set prepared for the 3013 thermal tests used a LANL outer container, an ARIES inner container, and a short version of the Cogema convenience container. The short Cogema containers are surplus and were used to minimize the impact to the ongoing LANL 3013 packaging program. The fabrication of the short Cogema containers was the same as the standard Cogema containers except that the height was 1-cm less. A stainless steel disk is placed between the inner container and the convenience container. The original design had all three containers in the same orientation. However, the manual can crimper used to close the convenience containers produced a crimp that was higher than in the original drawings, and the design was later changed to have the Cogema container inverted inside the inner container to better accommodate the disk. An additional stainless steel disk was placed inside the convenience container.

The Machined container set prepared for the 3013 thermal tests used a Hanford outer container, a Hanford BTIC inner container, and a Hanford BTCC convenience container. Each container in this set had a stainless steel disk placed inside the convenience container.

2.2 Payload Materials

The surrogate materials including the aluminum oxide, sodium chloride, potassium chloride, and calcium chloride powders were obtained through a level 3 procurement. The procurement specifications required receipt inspection and verification of supplier documentation in the form of a signed certificate of analysis for the product materials. The certificates of analyses for the aluminum oxide, sodium chloride, potassium chloride, and calcium chloride are provided in Appendix 2. The aluminum oxide is a high purity grade polishing compound containing greater than 99% Al_2O_3. The chloride salts are ACS grade requiring greater than 99.0% assays for the sodium and potassium chlorides and greater than 96.0% assay for the $CaCl_2$.

2.3 Characterization

Previous characterization by SRNL performed on a separate lot of aluminum oxide procured for the Phase 1/2 tests had already confirmed that the aluminum oxide is high purity and its composition is consistent with the supplier's specifications for particle size, which are similar to that of plutonium oxide[6, 7]. Additionally, measured pycnometer density of 3.9694 g/cm^3 is consistent with the theoretical density of aluminum oxide 3.974 g/cm^3.

The total amount of water in the sealed fire test containers is a critical parameter. The presence of water and other adsorbed species on the powders could result in higher than expected pressures during thermal cycling; therefore, the payload materials were characterized by thermogravimetric analysis with mass spectrometry (TGA-MS) to determine the adsorbed species on the materials. A TGA-MS measurement performed by SRNL indicated that aluminum oxide stored in room air has a moisture content of 0.191 wt%. This result shows that the amount of moisture on the materials as received from the supplier is significant, and it was necessary to remove the moisture prior to loading the convenience containers.

LANL performed a series of TGA measurements as part of the initial characterization of the payload materials. The measurements were intended to identify and determine the amounts of adsorbed species on all of the materials as they were received from the various suppliers. This information also aided the development of a treatment plan for the payload materials prior to loading in order to reduce the amounts of adsorbed species to the largest extent possible. An initial set of loss on heating (LOH) or loss on ignition (LOI) measurements were also performed on all of the payload materials. Because performing TGA on the payload materials at the time of loading was not feasible, these LOH and LOI measurements were intended to provide a reasonable estimate of the adsorbed moisture content of the payload materials at the time of loading based on the mass loss curves from TGA-MS.

TGA measurements were performed at LANL using a Netzsch STA 409 PC Luxx interfaced to a Pfeiffer ThermoStar mass spectrometer (MS). The LANL TGA-MS system is located in a dry air glovebox in the plutonium facility. The instrument is calibrated for determining water from the MS signal. The MS also monitors other volatile species CO_2, SO_2, NO_2, Cl_2, HCl, and other hydrocarbons, but the results are not quantitative.

Loss on ignition and loss on heating measurements were performed inside a helium-filled atmosphere controlled glovebox (ACB). Samples were weighed on a balance, placed inside a muffle furnace and heated to the specified temperature for two hours. After heating, the samples were allowed to cool and then weighed to determine the weight loss. Loss on ignition measurements were used to determine the weight loss of 10 g samples of aluminum oxide held at 1,000 ºC for two hours. Loss on heating measurements were used to measure the weight loss of the salt components at a specified temperature below the melting temperature of the salt for a two hour period. The LOH measurements were performed on 5 g samples of calcium chloride heated to 200 ºC and on 10 g samples of potassium and sodium chloride samples heated to 650 ºC.

2.3.1 Initial TGA and LOI Measurements on Aluminum Oxide Powder

Samples of the LANL aluminum oxide were removed from their original containers and loaded into CF containers under a dry helium atmosphere (a CF container is a stainless steel container hermetically sealed with a CF flange aka ConFlat® flange). A portion of this material was placed in a ceramic crucible, heated to 400 ºC in room air for 4 hours, and then returned to the ACB. Samples (approximately 3 g) of the heated and unheated aluminum oxide were loaded into CF containers under dry helium and transported to the TGA instrument. Additionally, a 3 g sample of the SRNL aluminum oxide previously shipped to LANL was loaded into a CF container and transported to the TGA instrument. Inside the dry air glovebox for each sample, the CF container was opened and a 1 g sample was loaded into the TGA crucible, which was then loaded into the TGA-MS instrument. The samples were run from room temperature to 750 ºC at a ramp rate of approximately 10 ºC/min. One sample was run to 1100 ºC for comparison.

The TGA results for the aluminum oxide materials are summarized in Table 3. The TGA and MS traces for each of the samples are given in Appendix 3. Moisture and CO_2 were the only species observed by the mass spectrometer (MS). Both species are evolved simultaneously up to 600 ºC. Because only two species were detected in the MS, the amount of adsorbed CO_2 was determined by subtracting the measured water from the total weight loss. Almost complete removal of moisture and CO_2 occurs by 600 ºC, and increasing the temperature to 750 ºC did not remove a significant amount of additional adsorbed species.

Comparing the heated and unheated samples, the TGA results for the LANL heated material show that heating in air to 400 ºC for 4 hours reduces the total amount of adsorbed water and CO_2 to less than 0.1 wt%. Additionally, the results suggested that heating to a higher temperature would be necessary to reduce the overall weight loss to below 0.03 wt% as recommended by the MIS Working Group. Comparing the results of the unheated LANL and SRNL samples, the moisture content of the SRNL material as determined by LANL was consistent with the previous SRNL measurement. The materials received at LANL had a smaller overall weight loss and less adsorbed moisture.

Table 3. Initial TGA Results for the Aluminum Oxide

Sample	TGA Total Mass Loss (wt%)	H_2O (wt%)	CO_2 (wt%)	Ratio H_2O / Total
SRNL Unheated Run A[1]	0.32	0.21	0.11	0.66
SRNL Unheated Run B	0.34	0.19	0.15	0.55
LANL Unheated	0.17	0.13	0.04	0.76
LANL Heated 400ºC for 4 hr. Run A	0.034	0.02	0.01	0.62
LANL Heated 400ºC for 4 hr. Run B	0.089	0.06	0.02	0.72

[1]Sample was heated to 1100 ºC. All other samples were heated to 750 ºC.

The TGA results were further analyzed to determine the proportion of the total weight loss that is water and whether that ratio changes with heating. The ratio of the amount of water to the total weight loss was computed for each sample. The ratios of the unheated samples range from 0.55 to 0.76. This range completely overlaps the range of the ratios for the heated samples (0.62 to 0.72). Therefore, it was concluded that this ratio would be unaffected by heating, and the average ratio of 0.66 could be used to estimate the amount of water from a LOI measurement.

Loss on ignition measurements were performed on samples of the SRNL unheated, LANL unheated, and LANL heated aluminum oxide. The results in Table 4 show good agreement between the total mass losses and the moisture determined by both methods.

Table 4. Comparison of Total Mass Losses and Moisture Based on LOI and TGA

	LOI Mass Loss (wt%)	**Average TGA Mass Loss (wt%)**	**Water (based on ratio of H_2O/Total) (wt%)**	**Average TGA H_2O (wt%)**
SRNL Unheated	0.36	0.33	0.24	0.20
LANL Unheated	0.19	0.17	0.13	0.13
LANL Heated 400ºC for 4 hr.	0.03	0.06	0.02	0.04

2.3.2 Initial TGA and LOH Measurements on Salt Materials

Samples of the salt materials were removed from their original containers and loaded into CF containers under a dry helium atmosphere and transported to the TGA instrument. Inside the dry air glovebox, the CF container was opened and a 1 g sample was loaded into the TGA crucible, which was then loaded into the TGA-MS instrument. The samples were heated from room temperature to 600 ºC at a ramp rate of approximately 10 ºC/min.

The TGA results for the salt materials are summarized in Table 5. The TGA and MS traces for each of the samples are given in Appendix 4. The calcium chloride had the largest mass loss of 8.31 wt%. All of the weight loss was attributed to adsorbed moisture. The moisture was removed by 325 ºC. Trace amounts of CO_2 and NO were observed by the MS. The potassium chloride had a total mass loss of 0.07 wt%, and the measured water was 0.03 wt% based on the MS. The sodium chloride had a total mass loss of 0.25 wt%, and the measured water was 0.08 wt% based on the MS. The mass loss curves for both the potassium and sodium chlorides show steep drops (at 392 ºC for KCl and 418 ºC for NaCl). These weight losses were both followed by a water signal in the MS; however, the measured water only explains a fraction of the weight loss. The identity of other species being evolved could not be determined from the MS result.

Table 5. Initial TGA Results for the Salt Materials

Sample	TGA Total Mass Loss (wt%)	H_2O (wt%)	Other Species
$CaCl_2$	8.31	See Note 2	Trace CO_2, NO
KCl	0.07	0.03	Trace CO_2
NaCl	0.25	0.08	Trace CO_2, NO, HCl

1. Samples were heated to 600 ºC to avoid melting the salts.
2. The amount of water loss is outside calibrated range for mass spectrometer. Entire mass loss is attributed to water.

Loss on heating measurements were performed on samples of each of the calcium, potassium, and sodium chloride salt materials. The results in Table 6 show that both methods give consistent results. For the calcium chloride sample, most of the moisture is removed by 200 ºC, and only small losses occur at 500 ºC and 650 ºC. The potassium and sodium chlorides have only small losses at 200 ºC and most of the weight loss occurs by 500 ºC.

Table 6. Comparison of Mass Losses by LOI and TGA for the Salt Materials

Sample	LOH Mass Loss to 200 ºC (wt%)	LOH Mass Loss to 500 ºC (wt%)	LOH Mass Loss to 650 ºC (wt%)	TGA Total Mass Loss to 650 ºC (wt%)
$CaCl_2$	8.21	8.41	8.44	8.31
KCl	0.01	0.07	0.08	0.07
NaCl	0.01	0.23	0.25	0.25

2.4 Material Preparation and Loading

Convenience containers were loaded with payload materials, water, and stainless steel filler materials as specified by the test plan. The measuring and test equipment used in the loading process is listed in Table 7. Loading was performed inside an ACB in TA55 PF3. The payload materials were weighed on a calibrated balance with a 64 kg range and 0.1 g accuracy. Moisture samples (approximately 10 g) were weighed on a calibrated balance with a 200 g range and 0.0001 g accuracy. Balances were checked daily with check weights prior to use. Data collection for the convenience container loading was captured on datasheets included in the test plan.

Table 7. Measuring and Test Equipment

Description	File Number	Range	Accuracy	Calibration Category
Balance Mettler Toledo	111167	0-64000 g	0.1 g	456 days
Balance Mettler Toledo	026984	0-200 g	0.0001 g	335 days
Check Weight Mettler Toledo	111539	2 kg		
Check Weight Mettler Toledo	021612	10 kg		
Check Weight Mettler Toledo	111536	20 kg		
Check Weight Mettler Toledo	111537	20 kg		
Check Weight Mettler Toledo	111538	20 kg		

Material preparation and container loading was completed according to a two-day or four-day loading plan depending on the payload composition as shown in Table 8. The loading plans were intended to pretreat the materials by heating and minimize the time that the materials could adsorb moisture or other gases from the atmosphere.

Table 8. Loading Plans for Test Containers

Two-Day Loading Plan Containers with Aluminum Oxide Only	
Containers	1, 2, 3, 4, 7, 8, 9, 10
Day 1	• Begin furnace run for Al_2O_3
Day 2	• Remove Al_2O_3 from furnace • Perform LOI: Al_2O_3 • Load container with Al_2O_3 and water

Four-Day Loading Plan Containers with Aluminum Oxide and Chloride Salts	
Containers	5, 6, 11, 12
Day 1	• Perform LOH-200 ºC: CaC_2 • Begin furnace run for NaCl
Day 2	• Remove NaCl from furnace • Begin furnace run for KCl
Day 3	• Remove KCl from furnace • Perform LOH-650 ºC: NaCl and KCl • Begin furnace run for Al_2O_3
Day 4	• Perform LOI: Al_2O_3 • Load container with Al_2O_3, $CaCl_2$, NaCl, KCl, and water

The furnace runs were performed by weighing out the material into a large ceramic crucible inside the ACB. The crucible holding the material was removed from the ACB and placed into the furnace inside a hood in the laboratory room. The material was heated in the furnace overnight. The furnace profiles for the aluminum oxide and salt each have three set points shown in Table 9. A 2 ºC/min ramp rate was used to reach each set point. The final set point was intended to keep the material warm until it was removed from the furnace to minimize the potential for moisture readsorption. Each material was removed from the furnace on and returned to the ACB where it was weighed and sealed in a container.

Table 9. Furnace Profiles for the Payload Materials

Aluminum Oxide Furnace Profile	
Set Point	**Time at Temperature**
400 ºC	60 min
600 ºC	240 min
200 ºC	Until unload

Chloride Salt Furnace Profile	
Set Point	**Time at Temperature**
200 ºC	60 min
500 ºC	240 min
200 ºC	Until unload

A moisture analysis was performed on each of the materials loaded in the container. The aluminum oxide, potassium chloride, and sodium chloride each had to meet an acceptance criterion of less than 0.03 wt% loss by LOH or LOI after heating. The calcium chloride was not heated prior to use due to the concern that heating may chemically alter the material. Instead, a LOH-200 ºC was performed to determine the moisture content, and the total weight loss from the LOH was assumed to be water. The sodium and potassium chlorides each had a LOH-650 ºC measurement after heating, and the total weight loss from the LOH was assumed to be water. The aluminum oxide had a LOI (1000 ºC) measurement performed after heating, and the mass of adsorbed water was calculated from the LOI result (0.66 × wt% loss).

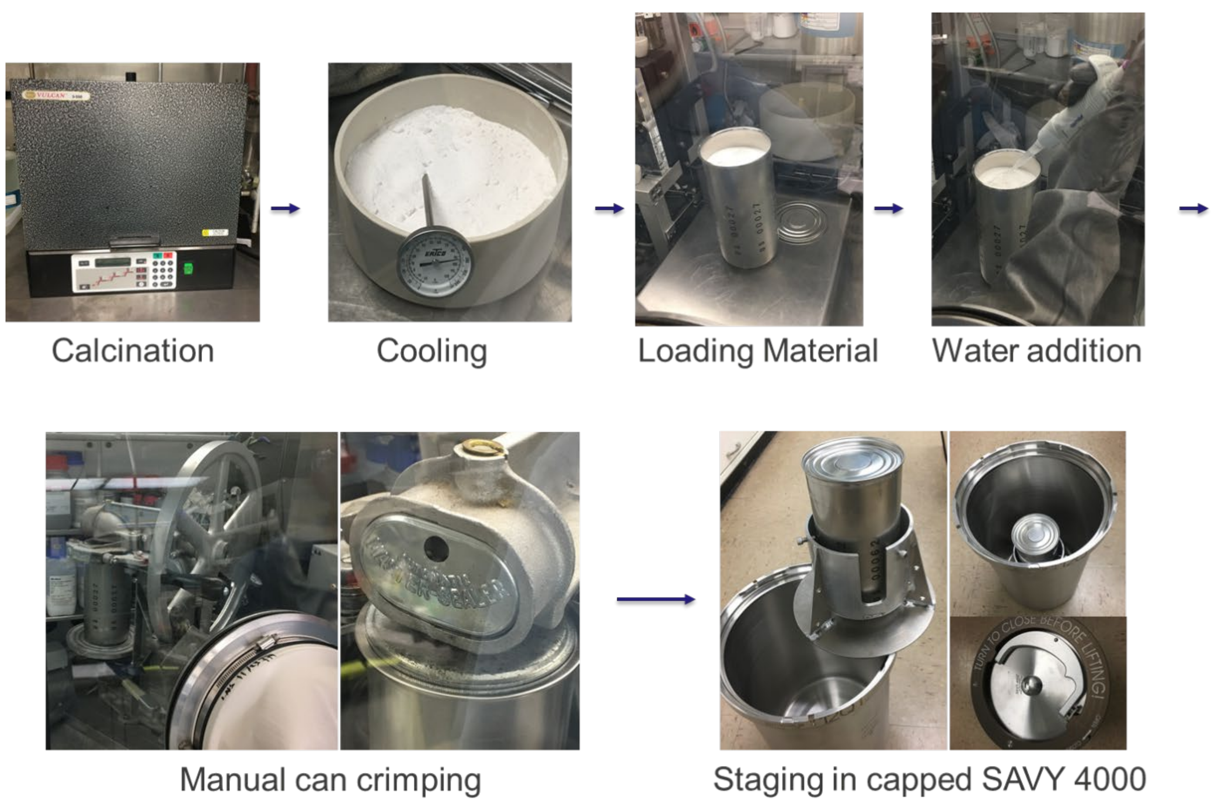

Figure 1. Container loading process.

For the containers with chloride salt components, each of the chloride salts was weighed out, and the salts were then mixed together by shaking for two minutes in a sealed can. The salt mixture was then added to the aluminum oxide and the mixture was mixed again by shaking for two minutes in a sealed can.

Liquid water was added to the containers by micropipette. The mass of liquid water to add to each container was calculated by subtracting the mass of adsorbed water remaining on the materials based on the results of the moisture analyses from the total mass of water specified in the test plan. Immediately following moisture addition, the weight of the loaded convenience container was measured, and the lid was installed. The final weight of the loaded convenience container was then obtained after installation of the lid.

The Cogema convenience containers had lids that required crimping. Crimping was done with the manual can crimper in the ACB immediately after loading. The crimped-sealed container was then inspected for proper closure. The Hanford convenience containers in the Machined container set are screw-top containers and did not require additional tools for closure.

The loaded convenience containers were placed inside a hermetically sealed overpack container while inside the ACB. The overpack container consisted of a capped SAVY-4000, which is a SAVY-4000 container modified by the installation of a filter cover with an O-ring seal that prevents gas exchange. The overpack container was then removed from the ACB for staging and transfer to the welding location.

2.5 Welding Processes

Welding of the container components was performed at LANL according to approved procedures. Each of the welds was inspected by a certified weld inspector and helium leak testing was performed to verify weld integrity. Inner 3013 containers for the packaging of plutonium are Pu-contaminated on the inside, and the welding of 3013 inner containers is performed in radiological gloveboxes with Pu-contaminated equipment. The 3013 fire test containers were required to be radiologically clean, and the welding of the 3013 inner containers for the fire test containers had to be performed using radiologically clean equipment. This required the set up or installation of welding equipment in cold areas that is identical to equipment in use or what had been used in the case of Hanford. The 3013 outer containers were welded using the existing 3013 outer container welder, which operates in a non-radiological helium filled glovebox located inside TA55 PF4.

2.5.1 Inner Container Welding: BTIC

Bagless Transfer Inner Containers were welded at TA03 SM39. Welds were made using an AMI 9-7500 orbital weld head powered by an AMI model 307 power supply. The welder uses 3/32 inch ceriated tungsten electrode set to an arc gap of 0.063 inches. BTCC convenience containers were transported to the welding location inside a hermetically sealed overpack container. Once the set up and preparation of the welder was complete, the overpack container was opened, and the convenience container was placed inside the BTIC body. The BTIC lid was placed on the top of the body, and helium was allowed to flow inside the container for two minutes to produce welds representative of welds produced at Hanford in 50% helium. The BTIC lid was then inserted to the stop in the BTIC body, and the welding program was started. The weld schedule makes three tack welds 90 degrees apart and begins the weld at the fourth 90-degree position. The thin can wall is welded to the thick plug wall with stepped travel synchronized with pulsed current. Upon completion of the weld, a visual inspection was done to verify that the welds meet the visual inspection criteria of the Fluor Hanford Nuclear Material Stabilization Project Welding Manual [8]. The excess container body above the weld was removed by cutting at 9.03 inches from the container bottom. Helium leak testing was then performed on the BTIC to ensure the welded container meets the acceptance criterion of 5×10^{-8} std-cc/s.

Figure 2. BTIC Welding.

2.5.2 Inner Container Welding :ARIES IC

The ARIES Inner 3013 containers were welded at TA55 PF3 inside a helium-filled ACB. ARIES Cogema convenience containers were transported to the welding location inside a hermetically sealed overpack container. The convenience containers were moved into the welding glovebox without a pump down, and the atmosphere of the glovebox was adjusted to less than 100 ppm oxygen. The overpack container was then opened, and the convenience container was inspected for proper closure. The container was then inverted and placed inside the ARIES Inner 3013 body on top of the stainless steel disk. Welding was performed using the Gas Tungsten Arc Welder. Upon completion of the weld, a visual inspection was done to verify that the welds meet the visual inspection criteria of the welding procedure. Helium leak testing was then performed on the ARIES Inner 3013 container to ensure the welded container meets the acceptance criterion of 5×10^{-8} std-cc/s.

Figure 3. ARIES inner container welding.

2.5.3 Outer Container Welding

The ARIES Flowform Outer 3013 containers and the Hanford Machined Outer 3013 containers were welded at TA55 PF4 inside a helium-filled ACB. The inner containers were moved into the welding glovebox, and the atmosphere of the glovebox was adjusted to less than 100 ppm oxygen. The inner container was then placed inside the outer container, and the outer container was welded closed. Upon completion of the weld, a visual inspection was done to verify that the welds meet the visual inspection criteria of the welding procedure. Helium leak testing was then performed on the Outer 3013 container to ensure the welded container meets the acceptance criterion of 5×10^{-8} std-cc/s.

2.6 Container Reworks

Any container not meeting the loading criteria as specified in the test plan or any acceptance criteria for proper closure was subject to reworking. A list of reworked containers is given in Table 10. A total of seven issues were identified with containers not meeting acceptance criteria. Two issues were related to the material loading parameters and were found to be acceptable, and the remaining five required reworking.

Table 10. List of Reworks for Each Test Container

	Configuration Outer/Inner/ CC	Rework Details
1*	EPD/BTIC/BTCC	**Acceptable**: aluminum oxide pretreated at 400 ºC rather than 600 ºC. LOI result > 0.03. Moisture 0.048 wt%. **Rework 1-1**: bad weld; replaced IC
2	EPD/BTIC/BTCC	None
3	EPD/BTIC/BTCC	None
4	EPD/BTIC/BTCC	None
5	EPD/BTIC/BTCC	None
6	EPD/BTIC/BTCC	None
7*	ARIES/ARIES/Cogema	**Acceptable**: aluminum oxide pretreated at 400 ºC rather than 600 ºC. LOI result > 0.03. Moisture 0.023 wt%.
8	ARIES/ARIES/Cogema	**Rework 8-1**: bad crimp; removed and replaced lid **Rework 8-2**: bad weld; replaced IC
9	ARIES/ARIES/Cogema	None
10	ARIES/ARIES/Cogema	**Rework 10-1**: bad crimp; reworked entire batch into new can **Rework 10-2**: bad weld; replaced IC
11	ARIES/ARIES/Cogema	None
12	ARIES/ARIES/Cogema	None

* Indicates containers without added moisture for equipment testing and validation

The two containers intended for equipment testing and validation (Containers 1 and 7) were loaded first. Initially, it was decided that the aluminum oxide would be heated to only 400 ºC for four hours to remove the adsorbed species. However, the material failed the LOI criterion of less than 0.03 wt% after heating. Because these containers are not official test containers, the conditions were deemed acceptable upon review by the MIS Working Group chair. This issue was resolved for all other containers by heating the material to 600 ºC for 4 hours rather than 400 ºC.

Rework 1-1 was performed to a weld blow-out on the BTIC. The BTIC was cut open and the loaded convenience container was welded into another BTIC. The weld issue was resolved by removing the backfill/purging lid above the BTIC lid prior to welding.

Reworks 8-1 and 10-1 were performed due to the improper closure of the Cogema container lid that occurred when the Cogema lid moved slightly off-center during crimping. The Cogema lid issue for Rework 8-1 was able to be resolved by removing the improperly closed lid, flattening out the lip on the Cogema container body, and installing a new lid. The Cogema lid issue for Rework 10-1 was unable to be resolved by replacing the lid, and a new batch of material was prepared and loaded into a new Cogema container. The issue with the crimper was resolved by placing additional spacers under the turn table that supports the can body to better hold the lid in place.

Reworks 8-2 and 10-2 were performed due to the improper closure of the ARIES inner containers. The issue was found to be a faulty electrical connection with the welder. Both containers were cut open and the loaded Cogema convenience containers were welded inside new inner containers.

3.0 Loading Results

Twelve 3013 containers were prepared for thermal testing. The mass of water and the free gas volume are critical parameters for the tests and were carefully controlled in the loading. The mass of water was controlled by pretreatment of the payload materials to reduce the amount of adsorbed species, storing the payload materials under inert atmosphere, and by performing LOI or LOH to determine the moisture content prior to loading. The free gas volume was controlled by loading the containers according to the specifications in the loading test plan.

The results for the water content of the payload materials and the free gas volume in each container are presented in this section. The containers are identified by a unique ID in the tables and figures. The first character is the container number (1-12) from Table 1. The second character describes the outer container version (E=EPD and A=ARIES). The third character gives the water content in grams or is shown as “D” to indicate the containers used for equipment testing and referred to by the program as the “dummy containers”. The fourth character “S”, if present, indicates the containers containing chloride salts.

3.1 Water Content of Payload Materials

Pretreatment of the payload materials including the aluminum oxide, potassium chloride, and sodium chloride reduced the amount of adsorbed species. The weight losses due to heating and the LOI measurements for the aluminum oxide are shown in Figure 4. The average weight loss due to heating was 0.20 wt%, which is in agreement with the initial TGA analysis for the LANL unheated material of 0.17 wt%. The LOI results for the aluminum oxide materials after heating are shown on the right. The LOI results range from 0.01 to 0.07 wt%. The two highest LOI results were for aluminum oxide that was heated to 400 ºC rather than 600 ºC and loaded in to the 3013 containers for equipment testing and validation. The average LOI result for aluminum oxide heated to 600 ºC was 0.02 wt%, and the average estimated moisture was 0.013 wt%.

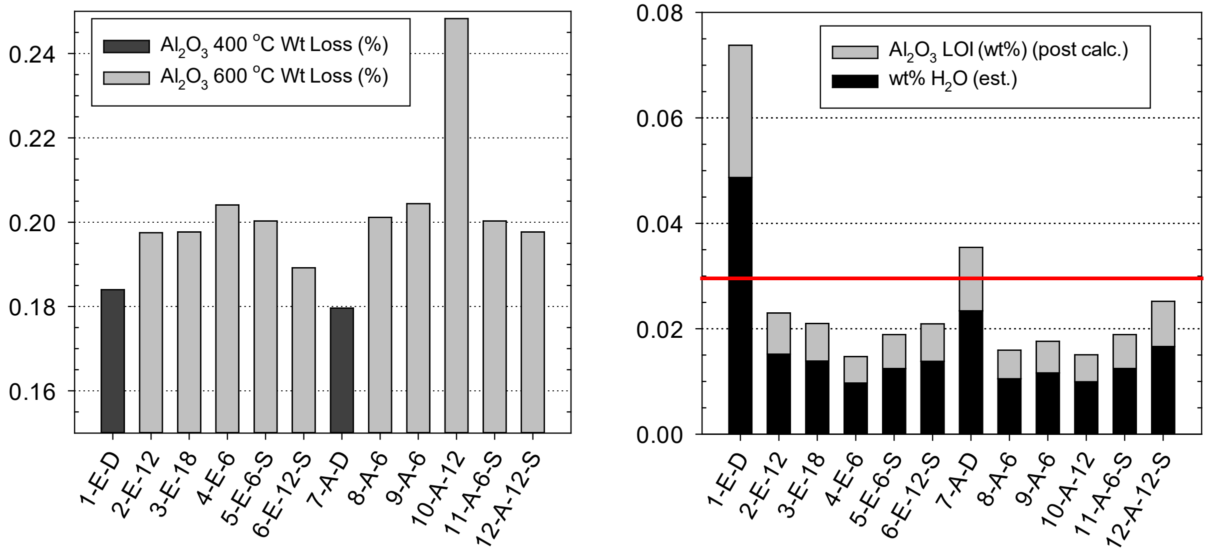

Figure 4. Aluminum oxide weight loss due to heating (left) and LOI results (right).

The weight losses due to heating and the LOH-650 measurements for the sodium chloride are shown in Figure 5. The average weight loss due to heating to 500 ºC was 0.16 wt%, which is lower than initial TGA and LOH-650 results (0.25 wt% for both). However, the average LOH-650 result for sodium chloride heated to 500 ºC was 0.025 wt%, which passed the 0.03 wt% acceptance criterion.

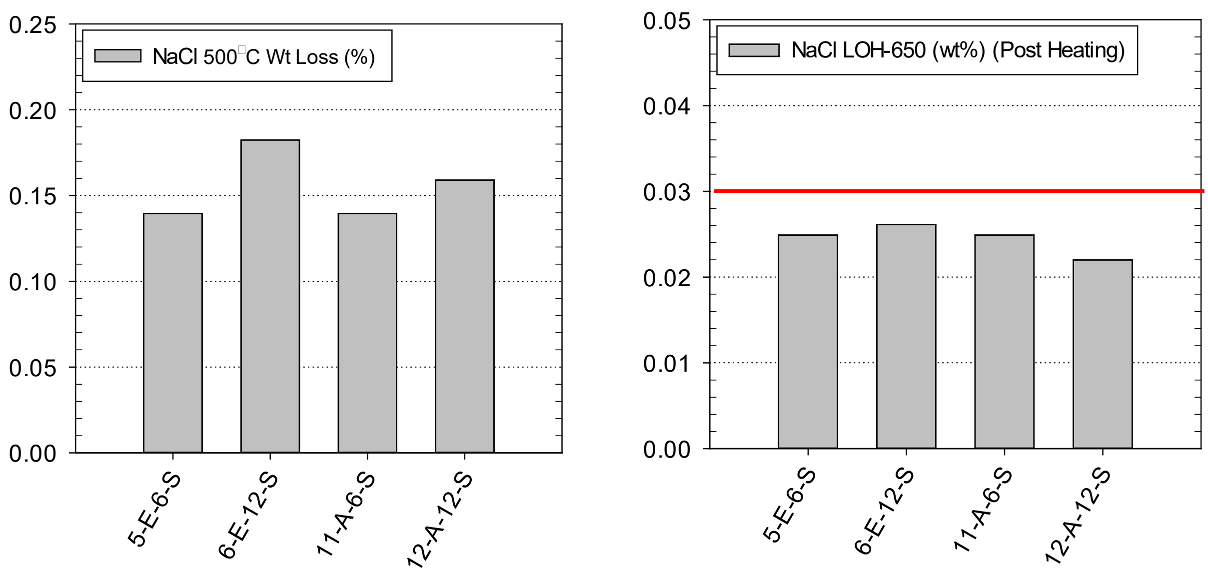

Figure 5. Sodium chloride weight loss due to heating (left) and LOH-650 results (right).

The weight losses due to heating and the LOH-650 measurements for the potassium chloride are shown in Figure 6. The average weight loss due to heating to 500 ºC was 0.03 wt%, which is lower than initial TGA and LOH-650 results of 0.07 and 0.08 wt%, respectively. However, the average LOH-650 result for sodium chloride heated to 500 ºC was 0.018 wt%, which passed the 0.03 wt% acceptance criterion.

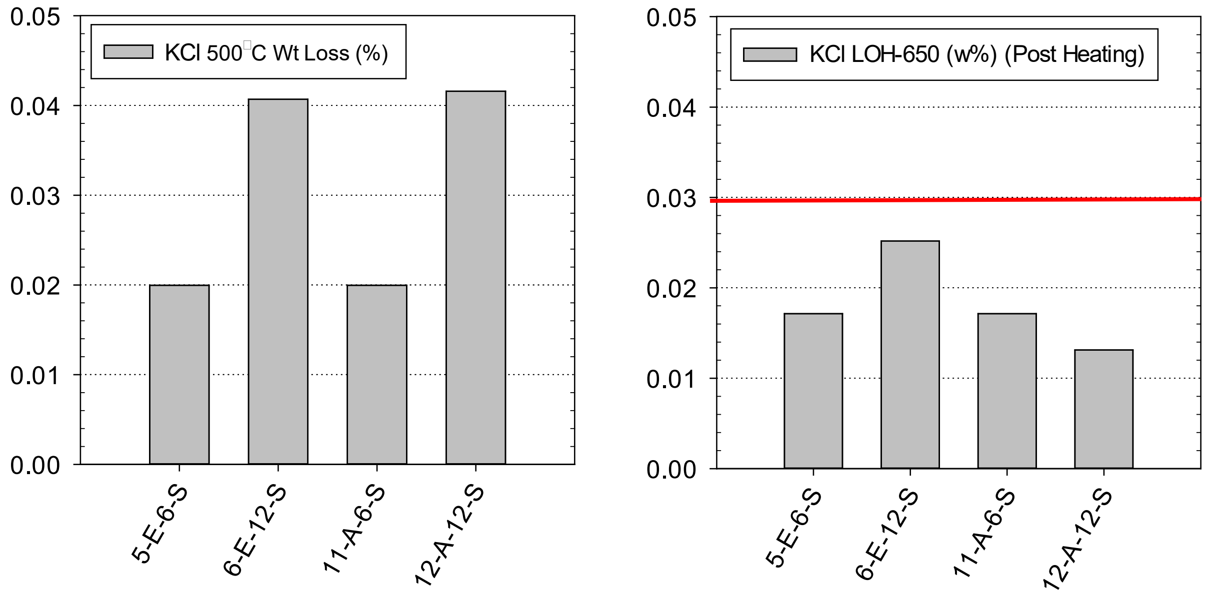

Figure 6. Potassium chloride weight loss due to heating (left) and LOH-650 results (right).

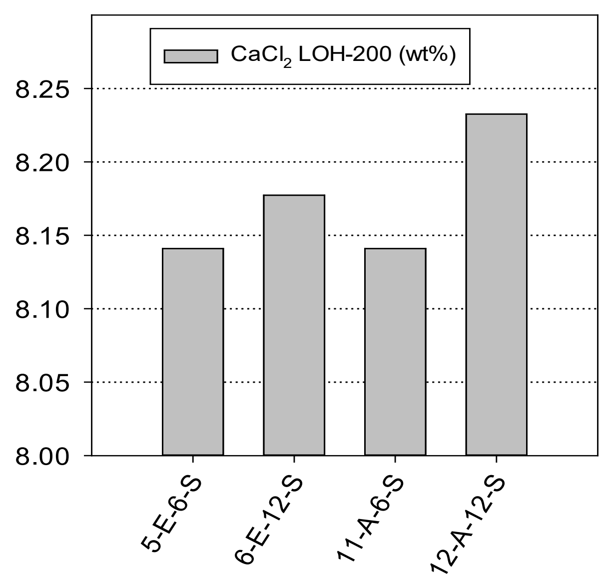

Figure 7. Calcium chloride LOH-200 results.

The calcium chloride was not heated prior to use. Instead, the water content was determined from a LOH-200 measurement. The LOH-200 measurements are summarized in Figure 7. The LOH-200 result was 8.17 wt%, which is consistent with the initial LOH-200 and slightly lower than initial TGA result of 8.31 wt%. The difference between the LOH-200 and the TGA results was likely due to the TGA being run to 650 ºC rather than 200 ºC.

The weights of the payload materials were adjusted for the water content based on the LOI and LOH results. Likewise, the mass of water required was reduced based on the amount of adsorbed water on each of the species. As shown in Table 11, the aluminum oxide contributed 0.1 to 0.2 g of adsorbed water after heating to 600 ºC. The largest contribution of adsorbed water (1.6 to 3.3 g) came from the calcium chloride that was added to the four containers with chloride salts. The total water on the material is given in the last column. Differences between the total water and the mass of water required may be explained by uncertainties in weighing throughout the loading process.

Table 11. Adsorbed Water and Water Added to Payload Materials

Container	Mass (g) Water on Al_2O_3	Mass (g) Water on $CaCl_2$	Mass (g) Water on KCl	Mass (g) Water on NaCl	Total Adsorbed Water (g)	Mass (g) Liquid Water Added	Mass (g) of Water Required	Total Water (g)
1-E-D	0.8	n.a.	n.a.	n.a.	0.8	0.0	0	**0.8**
2-E-12	0.2	n.a.	n.a.	n.a.	0.2	11.8	12	**12.0**
3-E-18	0.2	n.a.	n.a.	n.a.	0.2	17.8	18	**18.0**
4-E-6	0.2	n.a.	n.a.	n.a.	0.2	5.8	6	**6.0**
5-E-6-S	0.1	1.6	0.0	0.1	1.8	4.1	6	**5.9**
6-E-12-S	0.1	3.3	0.1	0.1	3.6	8.4	12	**12.0**
7-A-D	0.5	n.a.	n.a.	n.a.	0.5	0.0	0	**0.5**
8-A-6	0.2	n.a.	n.a.	n.a.	0.2	5.7	6	**6.1**
9-A-6	0.2	n.a.	n.a.	n.a.	0.2	5.7	6	**5.9**
10-A-12	0.2	n.a.	n.a.	n.a.	0.2	11.8	12	**12.0**
11-A-6-S	0.2	1.6	0.0	0.1	1.9	4.1	6	**6.0**
12-A-12-S	0.1	3.3	0.1	0.1	3.6	8.3	12	**11.9**

3.2 Free Gas Volume

Loading conditions in the test plan specified a payload in the test containers that would result in a free gas volume that is as close as possible to the minimum free gas volume that exists in the storage population of containers. The free gas volume was controlled by carefully controlling masses of the payload materials including the aluminum oxide, salt components, and the stainless steel filler.

The actual free gas volume V_g in each of the test containers was calculated from the minimum unoccupied volume inside of a 3013 outer container $V_{OC,U}$, the volume of the material V_M and the volume of water V_W shown in Eq.(1).

$$V_g = V_{OC,U} - V_M - V_W \qquad (1)$$

The actual volume of the material was calculated from the mass of the payload materials measured during loading and the particle density of each component as shown in Eq. (2).

$$V_M = \frac{m_{Al_2O_3}}{3.97\frac{g}{cm^3}} + \frac{m_{NaCl}}{2.17\frac{g}{cm^3}} + \frac{m_{KCl}}{1.98\frac{g}{cm^3}} + \frac{m_{CaCl_2}}{2.15\frac{g}{cm^3}} + \frac{m_{SS\_filler}}{7.9\frac{g}{cm^3}} \tag{2}$$

The unoccupied volume of the 3013 outer container was calculated from the minimum interior volume of the 3013 outer container, the volume occupied by the 3013 inner container, and the volume occupied by the convenience container as shown in Eq. (3).

$$V_{OC,U} = V_{OC,I} - V_{IC} - V_{CC} \tag{3}$$

The volume occupied by each of the 3013 inner and convenience containers was calculated from their respective masses measured during loading and the bulk density of stainless steel (7.9 g/cm^3). The interior volume of the outer container was assumed to be 2596 cm^3 from Ref [3].

Substituting $V_{OC,U}$ and V_M into Eq. (1) gives the actual free gas volume. Values of the actual free gas volumes and the free gas volumes specified in the calculation and test plan are given in Table 12. The specified free gas volume V'_g was calculated in Ref [3] based on the interior volume of the outer container $V_{OC,I}$ the maximum volume occupied by the inner container, the maximum volume of the convenience container for each container set, and the specified volume occupied by the payload material and the water.

Table 12. Comparison of Specified and Actual Material and Free Gas Volumes

Container	Specified V'_m (cm^3)	Actual V_m (cm^3)	Actual V_{IC} (cm^3)	Actual V_{CC} (cm^3)	Actual V_W (cm^3)	Specified V'_g, (cm^3)	Actual V_g (cm^3)
1-E-D	832	831.4	220.3	189.8	0.8	1323	1353.7
2-E-12	832	831.6	217.2	189.6	12.0	1311	1345.6
3-E-18	832	831.9	212.2	190.0	18.0	1305	1343.9
4-E-6	832	831.8	216.2	190.5	6.0	1317	1351.5
5-E-6-S	832	831.2	219.7	189.4	6.0	1317	1349.7
6-E-12-S	832	831.5	214.4	190.4	12.0	1311	1347.7
7-A-D	761	761.9	149.3	66.5	0.5	1624	1617.8
8-A-6	761	761.6	149.2	66.1	5.9	1618	1613.2
9-A-6	761	761.5	149.1	65.4	5.9	1618	1614.0
10-A-12	761	761.4	149.0	66.5	12.0	1612	1607.1
11-A-6-S	761	760.8	149.4	65.3	6.0	1618	1614.5
12-A-12-S	761	760.8	149.0	65.8	11.9	1612	1608.5

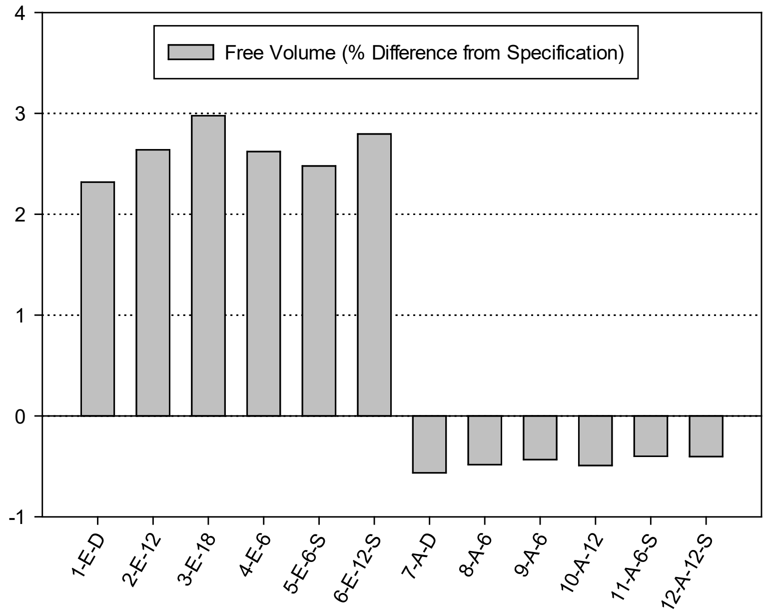

Figure 8. Percent difference between the specified and the actual free gas volumes.

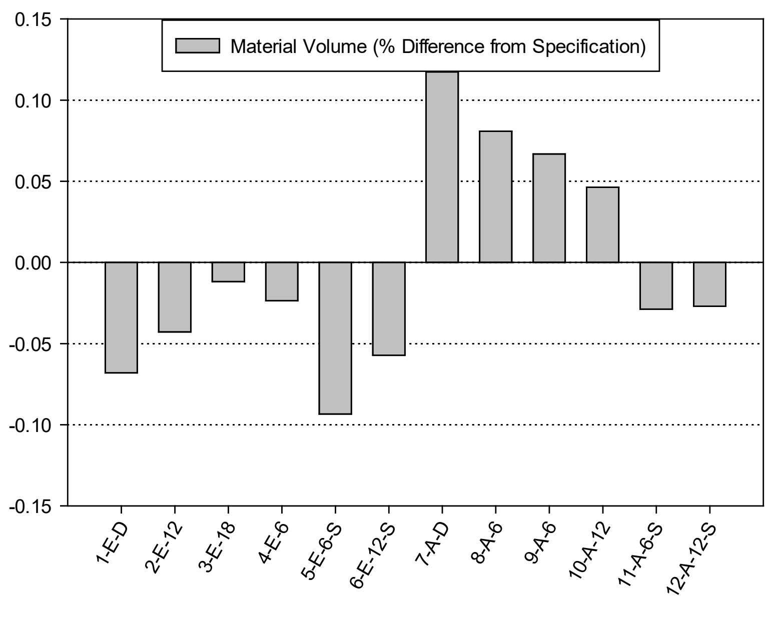

Figure 9. Percent difference between the specified and the actual material volumes.

The differences between the specified and actual free gas volumes are shown in Figure 8. The Machined container set has a +2.6% difference from the specified free gas volumes, and the Flowform container set has a -0.5% difference from the specified free gas volumes. The differences between the actual and specified material volumes are shown in Figure 9. The differences average to -0.04. Thus, the differences in the free gas volume are not due to the material loading.

The percent differences between the actual volume occupied by the 3013 inner container and the inner container volumes from Ref [3] are shown in Figure 10. The average BTIC volume of 217 cm^3 lies close to the average BTIC volume of 216 cm^3. The actual ARIES inner container volumes all exceed the upper tolerance limit by 8 cm^3.

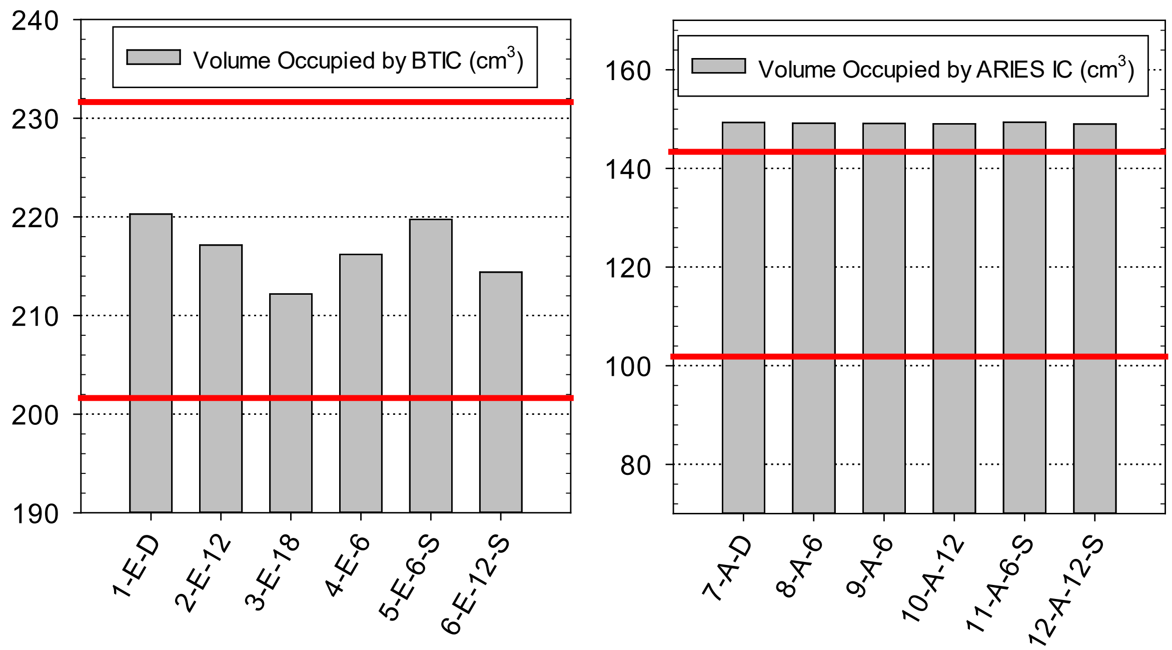

Figure 10. Comparison of the 3013 inner container volumes with the specifications based on the tolerances in the 3013 Standard.

The percent differences between the actual and specified convenience container volumes are shown in Figure 11. The average BTIC volume of 190 cm^3 lies close to the average convenience container volume of 191 cm^3. However, the average ARIES inner container volume of 70 cm^3 is close to but does not exceed the upper tolerance limit of 67 cm^3.

The results in Figure 10 and Figure 11 further illustrate that the differences in volume are due to the containers themselves. The calculations for the loading specifications in the test plan assumed values for the 3013 inner container and 3013 convenience container volumes would be at the upper tolerance limit. The actual volumes of the 3013 inner containers and 3013 convenience containers in the Flowform container set were closer to the upper tolerance limit and thus have smaller percent differences between the actual and specified free gas volumes. The actual volumes of the 3013 inner containers and 3013 convenience containers in the Machined container set were further from the upper tolerance limit and thus have larger percent differences between the actual and specified free gas volumes.

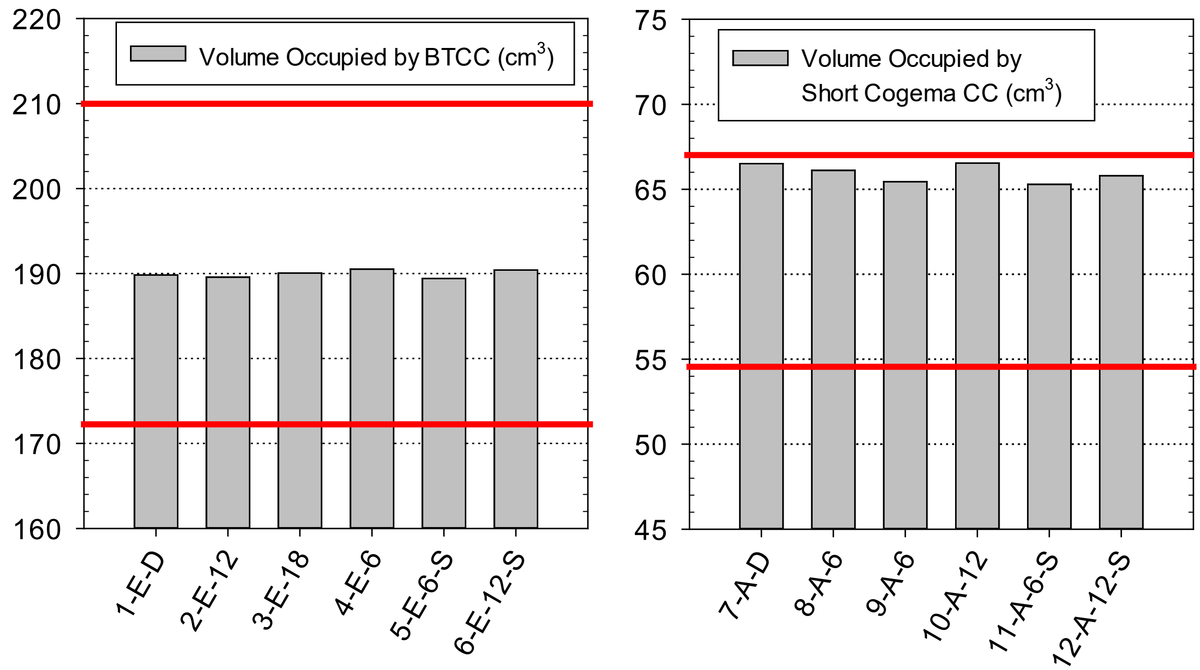

Figure 11. Comparison of the 3013 convenience container volumes with the specifications based on the tolerances in the 3013 Standard.

4.0 Conclusions

A set of twelve welded 3013 containers was prepared at Los Alamos National Laboratory for thermal testing in the PCV/SCV/3013 Thermal Test Program to determine the pressure response of packaged 3013 containers when exposed to a facility fire in the K-Area Complex (KAC) at the Savannah River Site. The set of test containers included five Machined 3013 containers and five Flowform 3013 containers. One additional container of each version was prepared for equipment testing and validation. The containers were loaded and welded, and each of the 3013 inner and outer containers successfully passed helium leak testing.

The critical parameters associated with loading containers are the amount of water in the payload and the free gas volume inside the 3013 outer containers. The water was controlled by pretreating the payload materials prior to loading by heating and storing the material in an inert environment. Pretreatment also removed other adsorbed species such as CO_2. Water remaining on the materials at the time of loading was determined by LOH or LOI measurements. The amount of water in the payload of the ten containers to be used for testing is within 0.1g of the specification in the test plan. The free gas volumes inside the loaded 3013 outer containers were within 3% of the specification. The differences between the actual and specified free gas volumes were due to the volumes occupied by the inner and convenience containers.

5.0 References

[1] C. McClard, B. Hackney, D. Riley, T. Venetz, and L. Worl, "Fire Testing of 3013 Containers," M. Maxted, ed., 2019.

[2] S. Hensel, and R. A. Sprankle, "Recommended 3013 Loadings and Configurations For Fire Testing," 2019.

[3] D. Veirs, C. McClard, and S. Hensel, *Calculation of fill and filler masses and volumes for 3013 fire test,* LA-UR-19-22902, Los Alamos National Laboratory, Los Alamos, NM, 2019.

[4] J. Narlesky, C. Emms, D. Tung, J. Stritzinger, L. Simms, D. Gregory, and L. Worl, *LANL Phase 3 Test Plan For Container Loading to Support the Fire-Induced Pressure Response and Failure Characterization of PCV/SCV/3013 Containers,* LA-UR-19-28746, Los Alamos National Laboratory, Los Alamos, NM, 2019.

[5] U. S. Department of Energy, *Stabilization, Packaging, and Storage of Plutonium-Bearing Materials,* DOE-STD-3013-2018, Washingon, D.C., 2018.

[6] H. W. Eldridge, and J. H. Scogin, *Payload Production for the Fire Testing of Primary Containment Vessels,* SRNL-STI-2018-00418, Savannah River National Laboratory, Aiken, SC, 2018.

[7] H. W. Eldridge, J. H. Scogin, and J. M. Duffey, *Criteria and Statistical Tolerances of Payload Production for Fire Testing,* SRNL-TR-2018-00009, Savannah River National Laboratory, Aiken, SC, 2018.

[8] J. R. Berkey, *Fluor Hanford Nuclear Material Stabilization Project Welding Manual,* HNF-7082, Fluor Hanford, Richland, WA, 2000.

5.1 Operating Procedures

Document No.	Title
PMT4-DOP-037	*Canning Material Using the Manual Convenience Canner (U)*
IWD-0013734	*Use of Welding Processes*
PMT4-DOP-130	*Gas Tungsten Arc Welding*
PMT4-DOP-139	*Helium Leak Testing a 3013 Container*
PMT4-DOP-043	*Plutonium Packaging for Storage*
PMT4-DOP-044	*Outer Container Welding for Long-Term Storage*

Appendix 1.Container Set Drawings

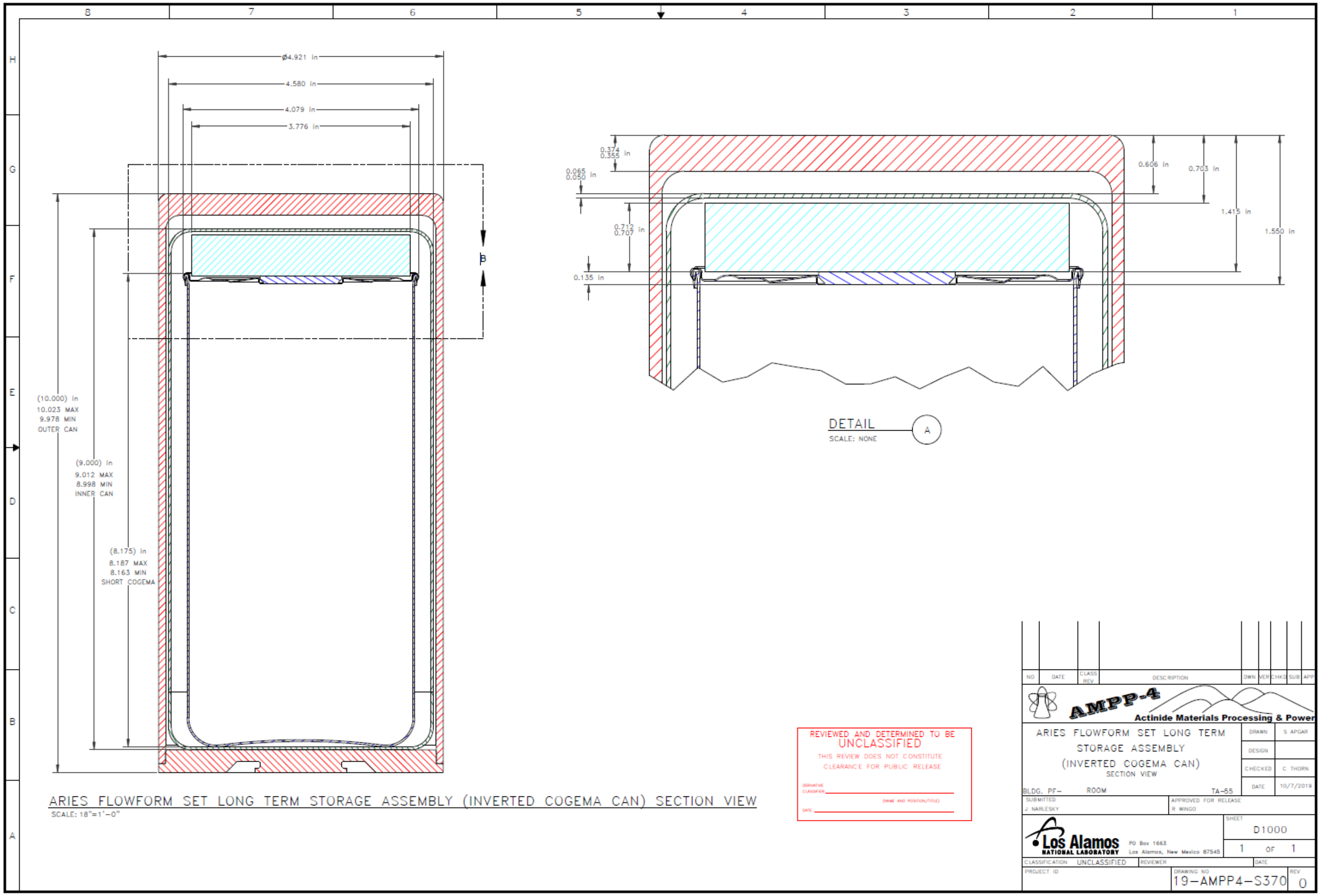

Figure 12. Flowform container set drawing.

Appendix 1. Container Set Drawings (cont.)

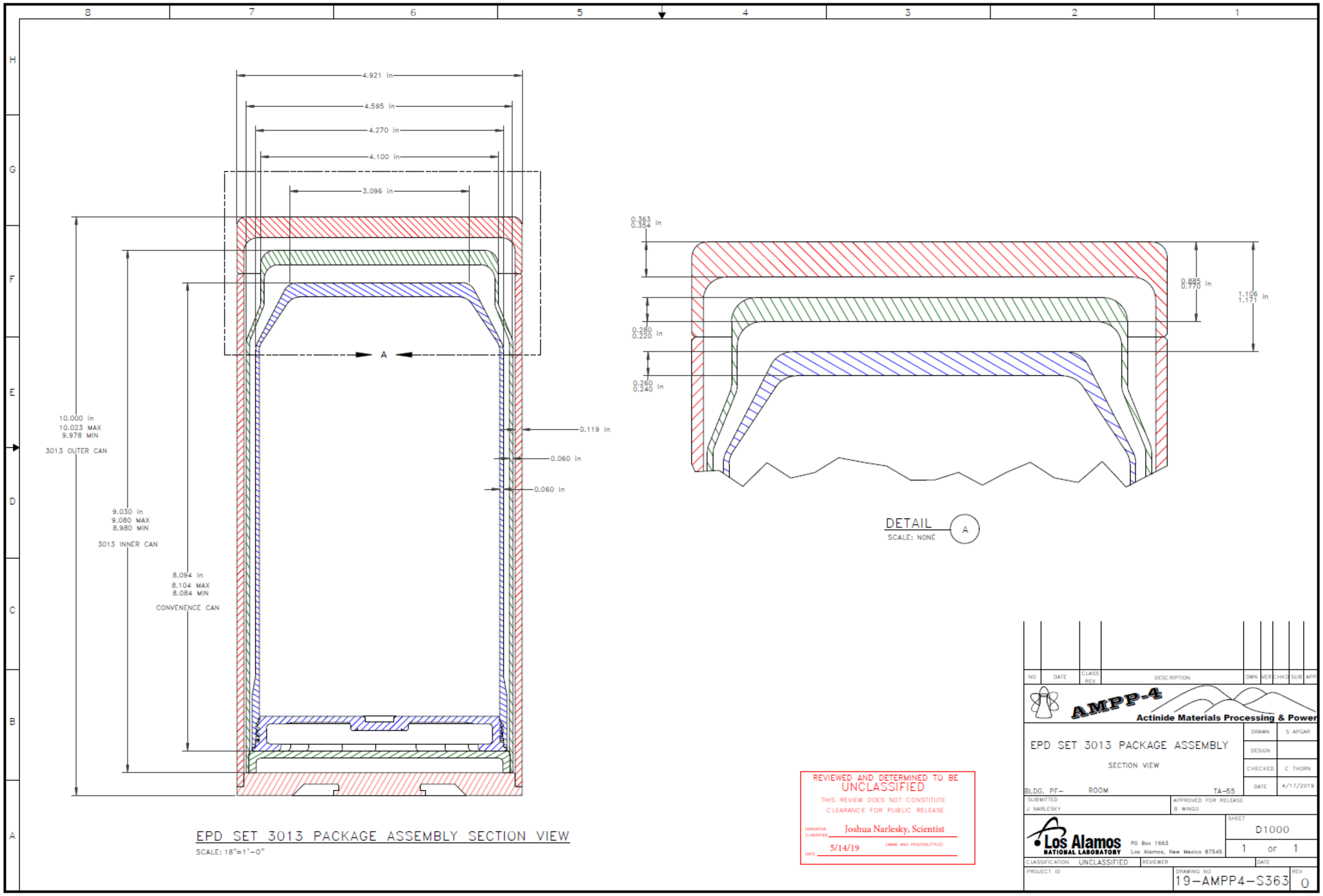

Figure 13. Machined (EPD) container set drawing.

Appendix 2. Certificates of Analyses

Kramer Industries, Inc.

140 Ethel Road West
Unit U
Piscataway, NJ 08854

phone: 888-515-9443
fax: 732-650-0556
www.KramerIndustriesOnline.com

Right From The Start

Certificate of Analysis

White Aluminum Oxide Grit - 400 Mesh

Lot #: J4904515708

Mfg. Date: 04/2019 Shelf Life: 2 Years Exp. Date: 04/2021

-APPROVED-

Chemical Analysis:

Al_2O_3	99.36 %	**CaO**	0.03 %
TiO_2	0.01 %	**MgO**	0.05 %
SiO_2	0.01 %	**Na_2O**	0.49 %
Fe_2O_3	0.05 %	**K_2O**	0 %

Particle Size Distribution:

Screen Size	Lower Limit	Upper Limit	% Retention
Ds 3	0 %	32 %	23.44 %
Ds 50	16.3 %	18.3 %	17.62%
Ds 94	8 %	100 %	12.64 %

Notes: FEPA-42-2: 2006; B. Density: FEPA-44-1: 2006 (Macrogrits)

PO #547568
Quantity: 50 pounds

Steven Schneider, QC Manager

1 Results obtained using Sedigraph settling test.
2 Results of a recent batch.
3 DS 3 result indicates that 3% of distribution is the result and larger.
4 DS 50 result is median of particle size distribution.
5 DS 94 result indicates that 94% of distribution is the result and larger.

Appendix 2. Certificates of Analyses (cont.)

Certificate of Analysis Page 1 of 2

ThermoFisher SCIENTIFIC

Certificate of Analysis

1 Reagent Lane
Fair Lawn, NJ 07410
201.796.7100 tel
✓ 201.796.1329 fax

ThermoFisher Scientific's Quality System has been found to conform to Quality Management System Standard ISO9001:2015 by SAI Global Certificate Number CERT - 0120632

This is to certify that units of the lot number below were tested and found to comply with the specifications of the grade listed. Certain data have been supplied by third parties. ThermoFisher Scientific expressly disclaims all warranties, expressed or implied, including the implied warranties of merchantability and fitness for a particular purpose. Products are for research use or further manufacturing. Not for direct administration to humans or animals. It is the responsibility of the manufacturer to determine suitability based upon the intended use of the end product. Products are tested to meet the analytical requirements of the noted grade. The following information is the actual analytical results obtained.

Catalog Number	S271	Quality Test / Release Date	11/26/2018
Lot Number	188345		
Description	SODIUM CHLORIDE, CERTIFIED A.C.S.		
Country of Origin	United States	Suggested Retest Date	Nov/2023
Chemical Origin	Inorganic-non animal		
BSE/TSE Comment	No animal products are used as starting raw material ingredients, or used in processing, including lubricants, processing aids, or any other material that might migrate to the finished product.		
Chemical Comment	This material does not contain any BPA (Bisphenol A), phthalates/phthalate esters or animal derived substances.		

N/A

Result Name	Units	Specifications	Test Value
APPEARANCE		REPORT	Small white crystals
ASSAY	%	>= 99.0	99.6
SULFATE (SO4)	%	<= 0.004	<0.004
BROMIDE	%	<= 0.01	<0.01
CALCIUM	%	<= 0.002	0.001
CHLORATE & NITRATE	%	<= 0.003	<0.003
HEAVY METALS BY ICP-OES	ppm	<= 5	<5
IDENTIFICATION	PASS/FAIL	= PASS TEST	PASS TEST
INSOLUBLE MATTER	%	<= 0.005	0.001
IODIDE	%	<= 0.002	<0.002
IRON (Fe)	ppm	<= 2	<2
MAGNESIUM	%	<= 0.001	<0.001
PH 5% SOLUTION @ 25 DEG C		Inclusive Between 5.0 - 9.0	6.6
PHOSPHATE (PO4)	ppm	<= 5	<5
POTASSIUM (K)	%	<= 0.005	0.003
BARIUM (Ba)	PASS/FAIL	= PASS TEST	PASS TEST

Residual Solvents	

Note: The data listed is valid for all package sizes of this lot of this product, expressed as an extension of this catalog number listed above.
If there are any questions with this certificate, please call at (800) 227-6701.
*Based on suggested storage condition.

Appendix 2. Certificates of Analyses (cont.)

Certificate of Analysis

ThermoFisher SCIENTIFIC

Certificate of Analysis

1 Reagent Lane
Fair Lawn, NJ 07410
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Catalog Number	P217	Quality Test / Release Date	04/25/2019
Lot Number	187537		
Description	POTASSIUM CHLORIDE, A.C.S.		
Country of Origin	United States	Suggested Retest Date	Apr/2024
Chemical Origin	Inorganic-non animal		
BSE/TSE Comment	No animal products are used as starting raw material ingredients, or used in processing, including lubricants, processing aids, or any other material that might migrate to the finished product.		
Chemical Comment			

N/A

Result Name	Units	Specifications	Test Value
APPEARANCE		REPORT	White crystals
SULFATE (SO4)	%	<= 0.001	<0.001
HEAVY METALS (as Pb)	ppm	<= 5	<5
BARIUM (Ba)	PASS/FAIL	= P.T. (ABOUT 0.001%)	P.T. (ABOUT 0.001%)
PHOSPHATE (PO4)	ppm	<= 5	<3
MAGNESIUM	%	<= 0.001	<0.0005
ASSAY	%	Inclusive Between 99.0 - 100.5	100.0
IDENTIFICATION	PASS/FAIL	= PASS TEST	PASS TEST
INSOLUBLE MATTER	%	<= 0.005	0.001
CHLORATE & NITRATE	%	<= 0.003	<0.001
PH 5% SOLUTION @ 25 DEG C		Inclusive Between 5.4 - 8.6	5.6
BROMIDE	%	<= 0.01	<0.01
CALCIUM	%	<= 0.002	<0.001
IODIDE	%	<= 0.002	<0.002
IRON (Fe)	ppm	<= 2	<1
SODIUM (Na)	%	<= 0.005	<0.005

Teresa Bailey-Wyche

Quality Assurance Specialist - Certificate of Analysis Fair Lawn

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*Based on suggested storage condition.

Appendix 2. Certificates of Analyses (cont.)

Thermo Fisher SCIENTIFIC

Certificate of Analysis

Product No.: 89866

Product: Calcium chloride, anhydrous, ACS, 96.0% min

Lot No.: Y04D046

Test	Limits	Results
Assay	96.0 % min	97.1 %
Titrable base	0.006 meq/g max	< 0.006 meq/g

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QC Specialist

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Appendix 3.TGA Results for the Aluminum Oxide

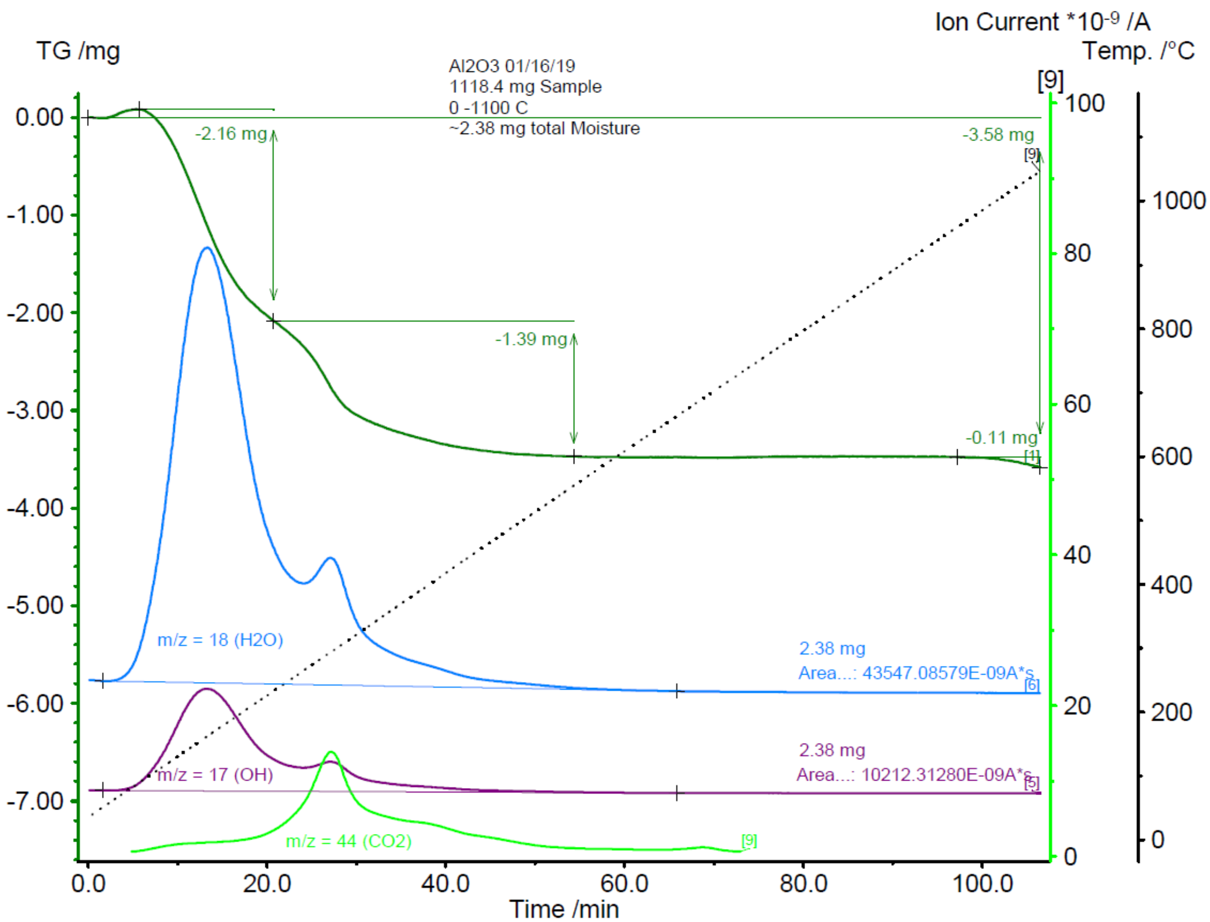

Figure 14. TGA results for SRNL unheated aluminum oxide sample A.

SRNL unheated sample A had a total mass loss of 0.32 wt% (3.58 mg). The MS detected two species: water and CO_2, and both species were removed by 600 ºC. The water content by MS was 2.38 mg or 0.21 wt%. Assuming the remainder of the mass loss was CO_2, the material had 0.11 wt% CO_2.

Appendix 3. TGA Results for the Aluminum Oxide (cont.)

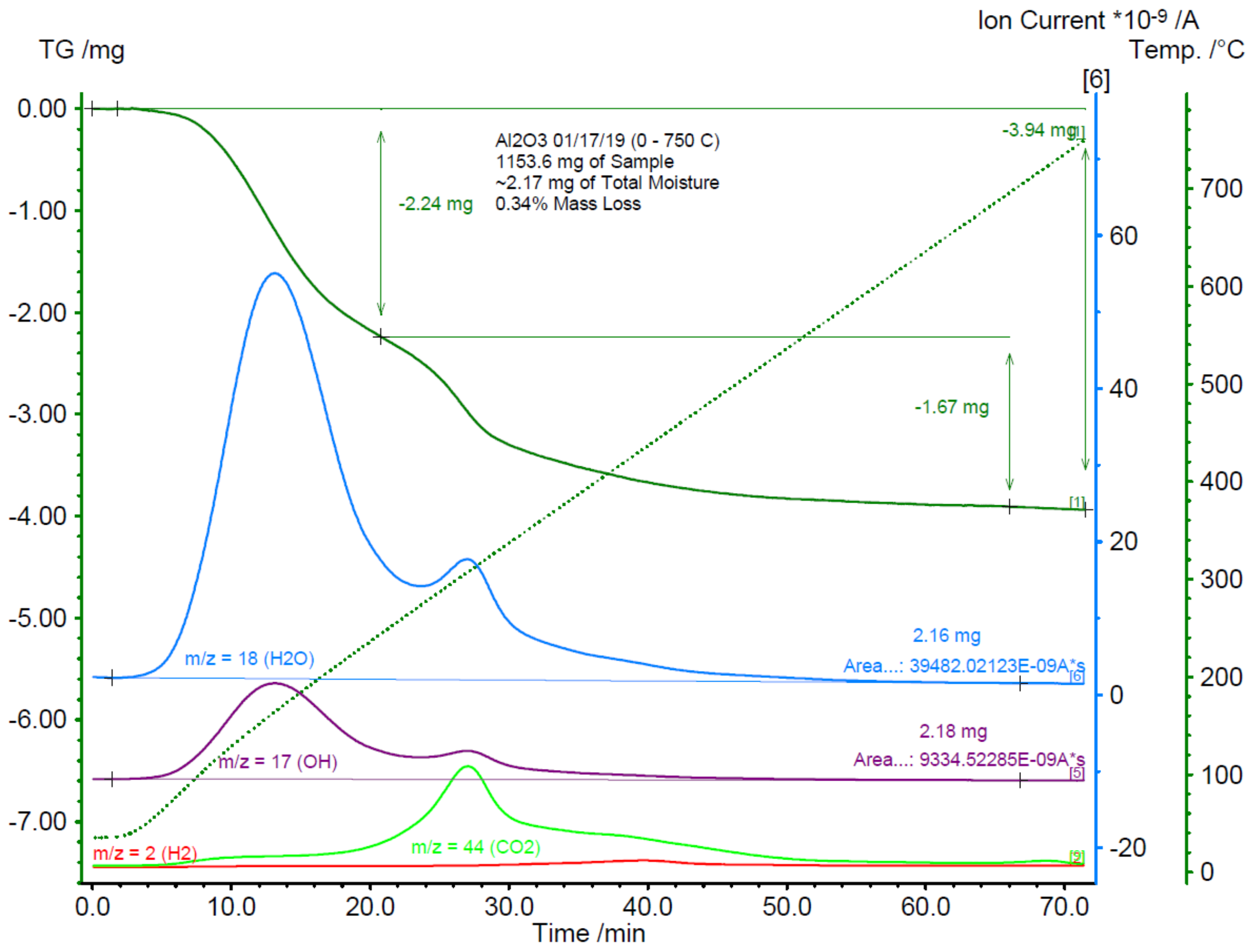

Figure 15. TGA results for SRNL unheated aluminum oxide sample B.

SRNL unheated sample B had a total mass loss of 0.34 wt% (3.94 mg). The MS detected two species: water and CO_2, and both species were removed by 600 ºC. The water content by MS was 2.17 mg or 0.19 wt%. Assuming the remainder of the mass loss was CO_2, the material had 0.15 wt% CO_2.

Appendix 3. TGA Results for the Aluminum Oxide (cont.)

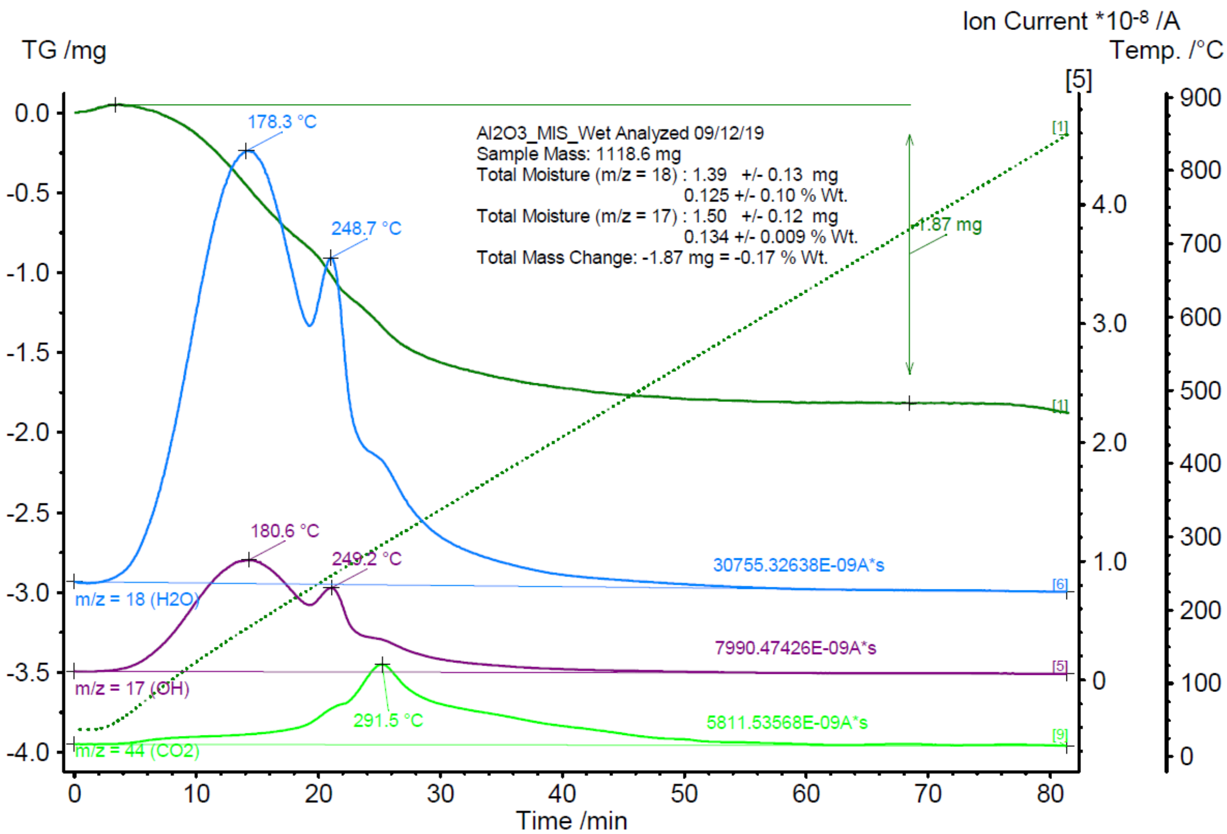

Figure 16. TGA results for the LANL unheated aluminum oxide sample.

The LANL unheated sample had a total mass loss of 0.17 wt% (1.87 mg). The MS detected two species: water and CO_2, and both species were removed by 600 ºC. The water content by MS was 1.44 mg or 0.13 wt%. Assuming the remainder of the mass loss was CO_2, the material had 0.04 wt% CO_2.

Appendix 3. TGA Results for the Aluminum Oxide (cont.)

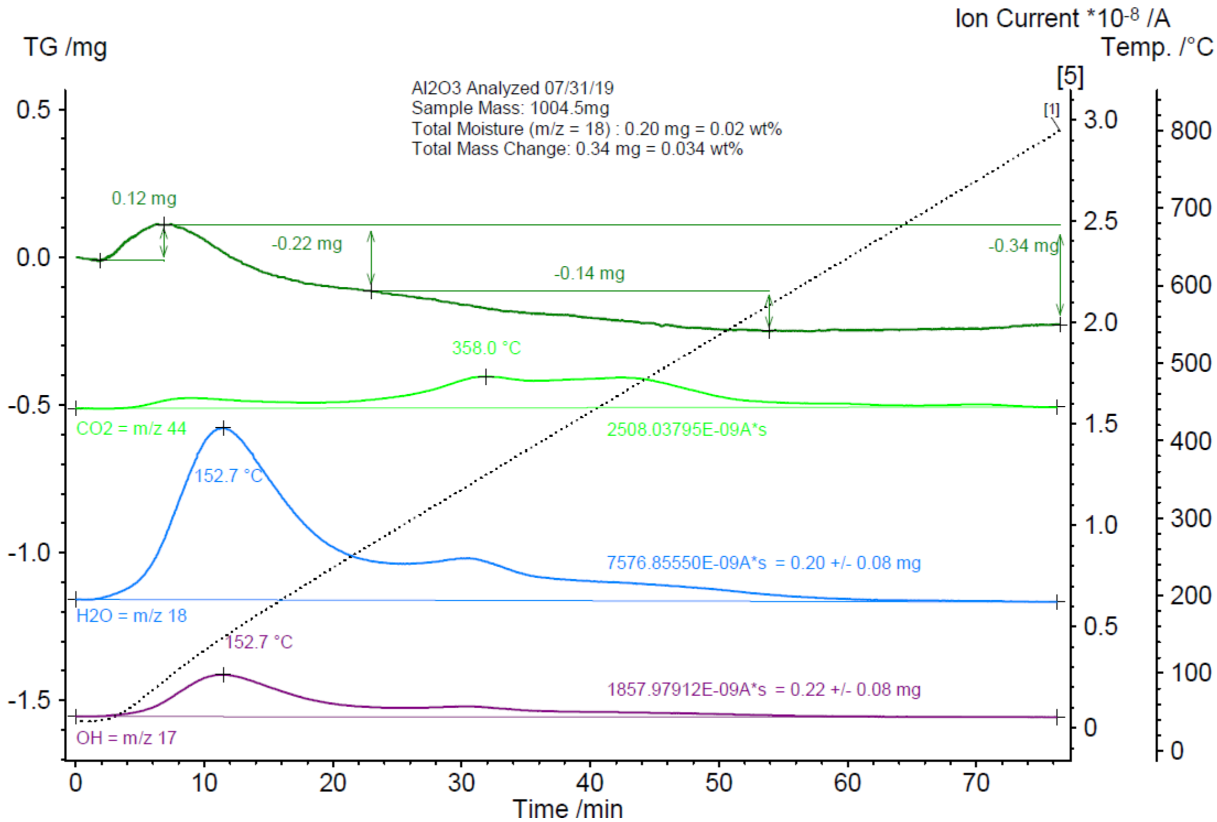

Figure 17. TGA results for the LANL heated aluminum oxide sample A.

LANL heated sample A had a total mass loss of 0.034 wt% (0.34 mg). The MS detected two species: water and CO_2, and both species were removed by 600 ºC. The water content by MS was 0.20 mg or 0.02 wt%. Assuming the remainder of the mass loss was CO_2, the material had 0.01 wt% CO_2.

Appendix 3. TGA Results for the Aluminum Oxide (cont.)

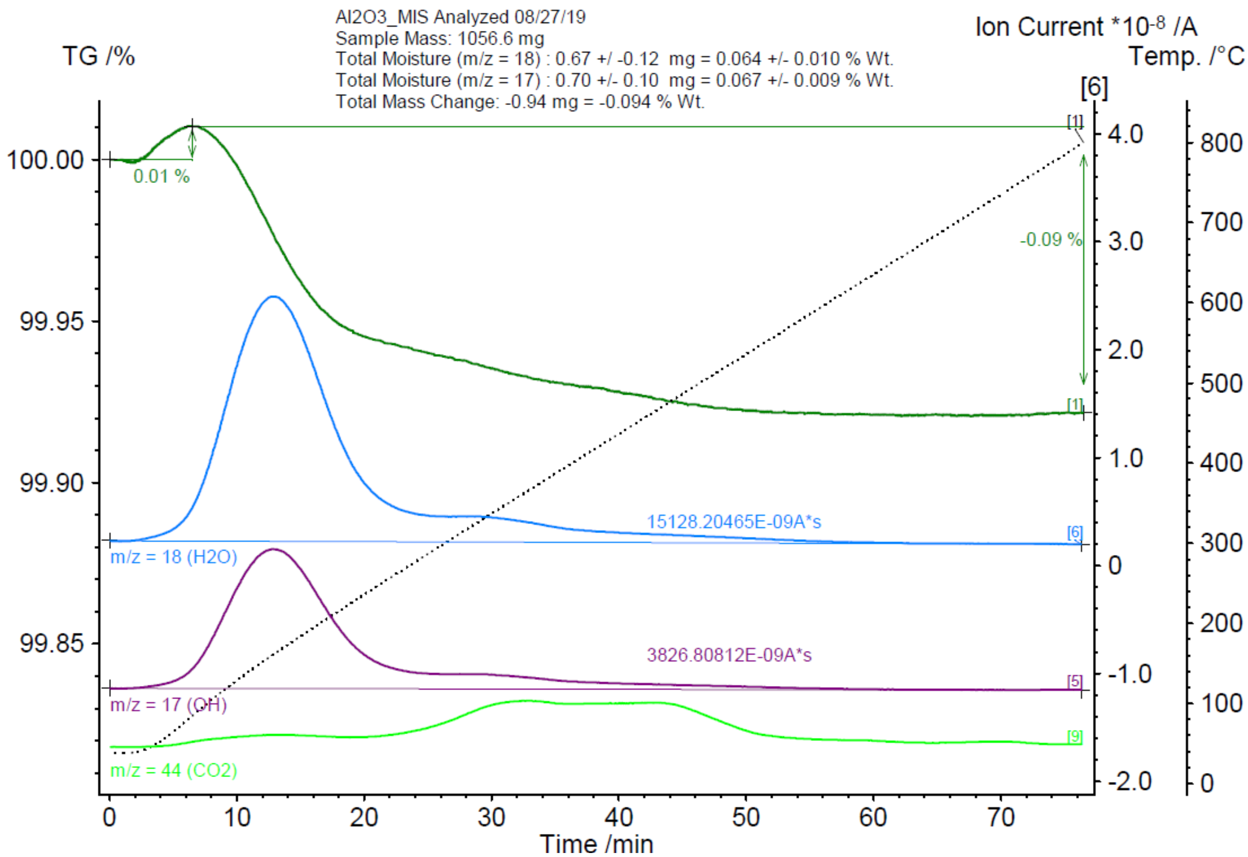

Figure 18. TGA results for the LANL heated aluminum oxide sample B.

LANL heated sample B had a total mass loss of 0.089 wt% (0.94 mg). The MS detected two species: water and CO_2, and both species were removed by 600 ºC. The water content by MS was 0.69 mg or 0.06 wt%. Assuming the remainder of the mass loss was CO_2, the material had 0.02 wt% CO_2.

Appendix 4.TGA Results for the Salt Components

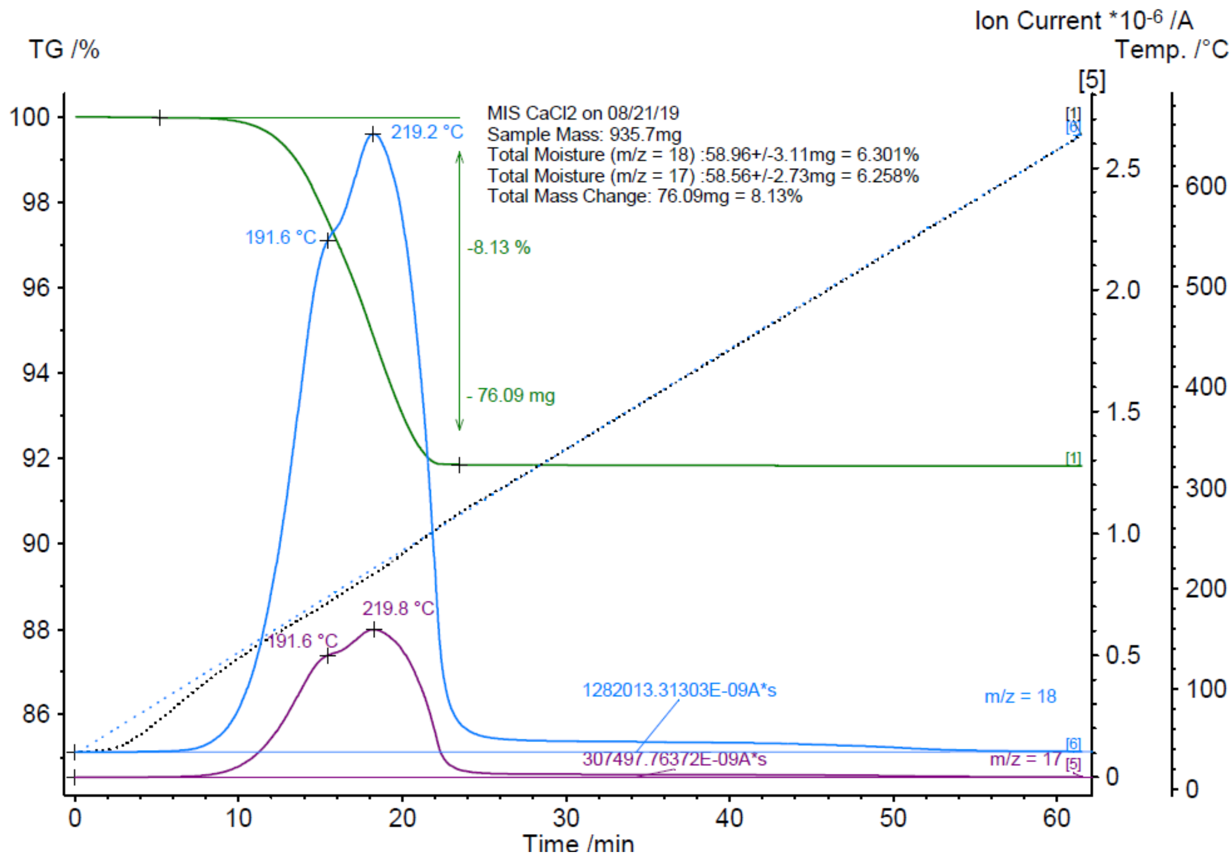

Figure 19. TGA results for the $CaCl_2$ material.

The $CaCl_2$ sample had a total mass loss of 8.13 wt% (76.09 mg). The MS detected two species: water and trace amounts of CO_2. The mass loss was attributed to removal of water, which was shown to be complete by 325 ºC. (Note. The gram amount of water as determined by the mass spectrometer exceeded the range of the calibration.)

Appendix 4. TGA Results for the Salt Components (cont.)

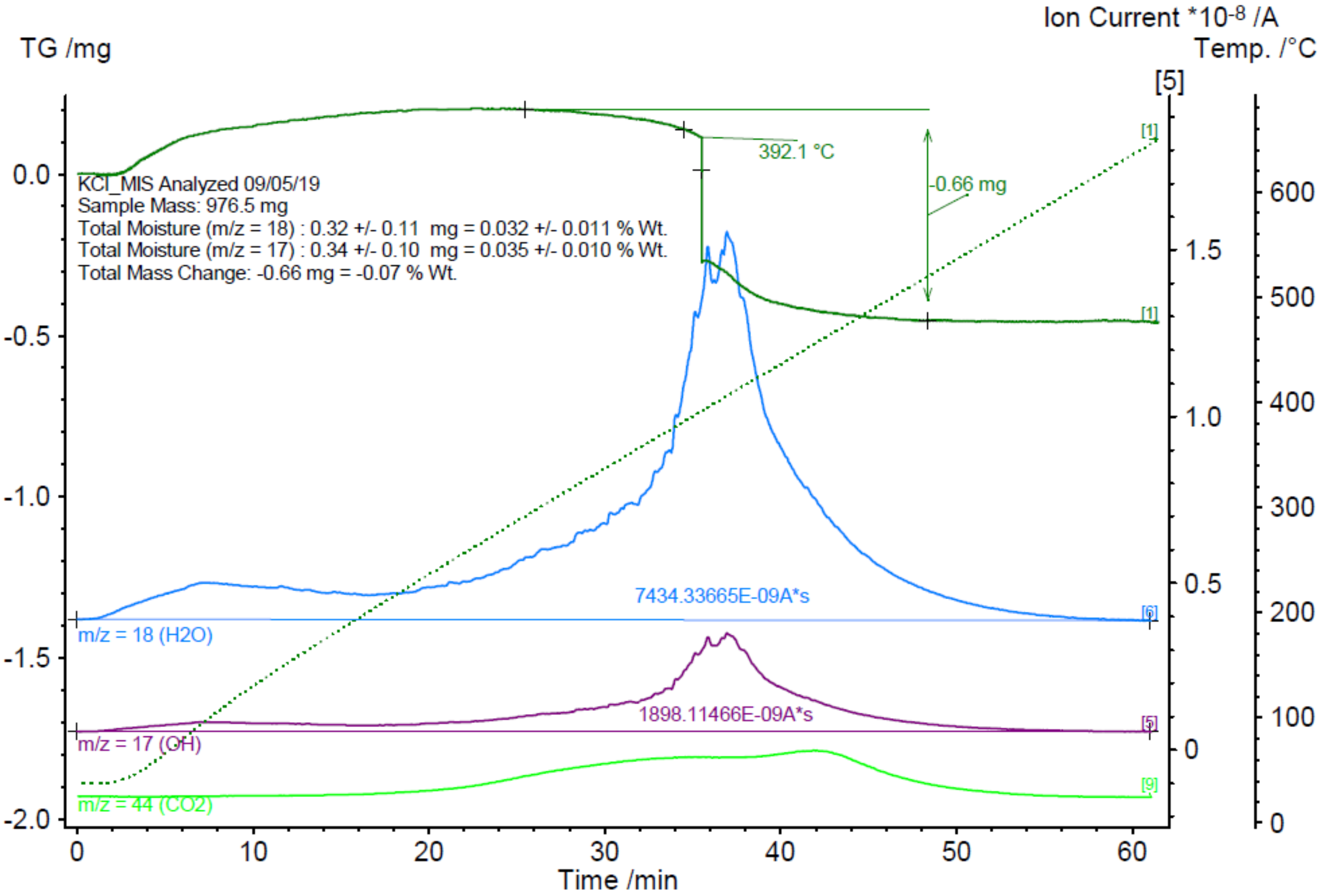

Figure 20. TGA results for the potassium chloride material.

The potassium chloride sample had a total mass loss of 0.07 wt% (0.66 mg). The MS detected two species: water and CO_2, and both species were removed by 600 ºC. The water content by MS was 0.33 mg or 0.03 wt%. The species responsible for the steep drop in the mass loss curve was not determined.

Appendix 4. TGA Results for the Salt Components (cont.)

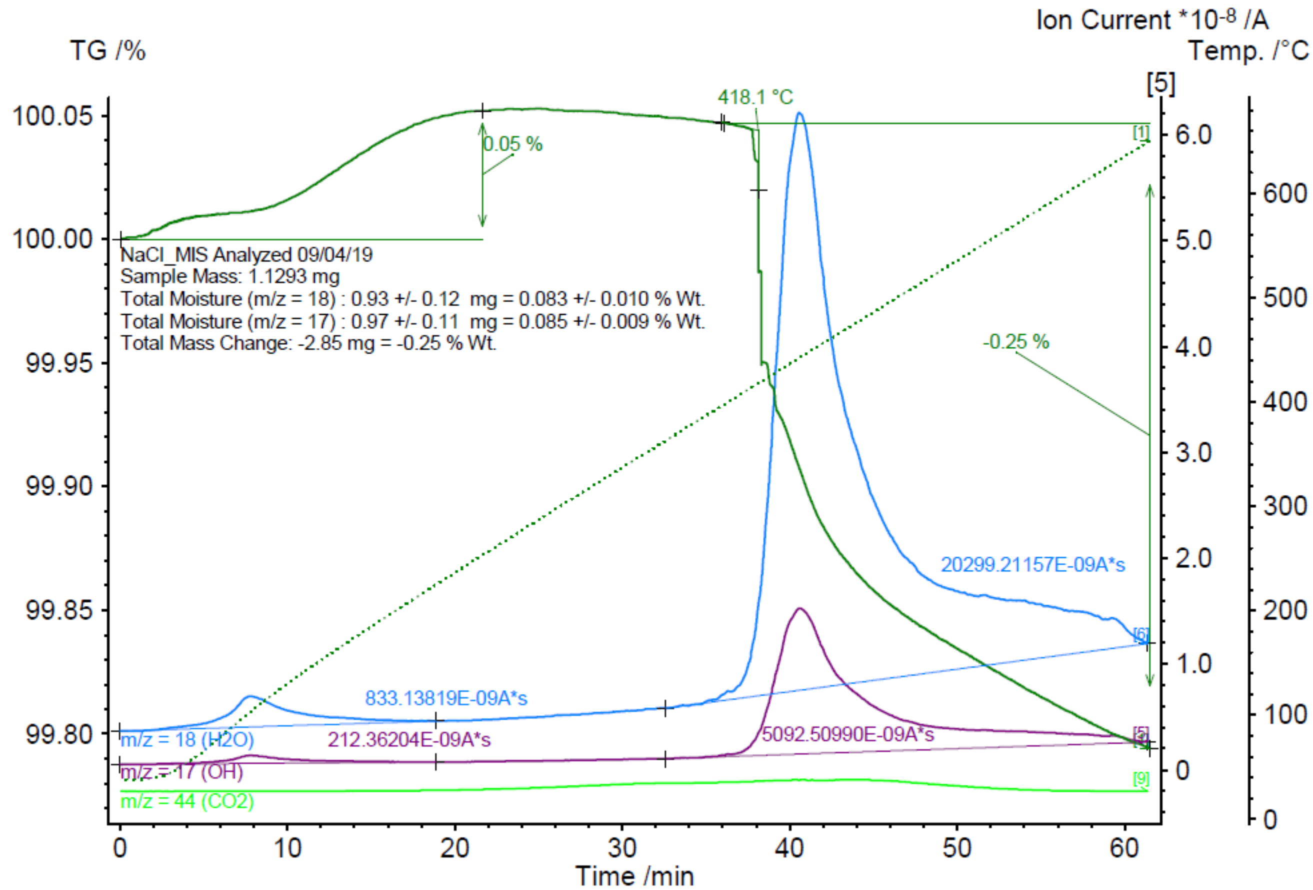

Figure 21. TGA results for the potassium chloride material.

The sodium chloride sample had a total mass loss of 0.25 wt% (2.85 mg). The MS detected two species: water and CO_2, and both species were removed by 600 ºC. The water content by MS was 0.95 mg or 0.08 wt%. The species responsible for the steep drop in the mass loss curve was not determined.

Appendix 5. Container Loading Data

Table 13. Container Loading Data

Container	Al_2O_3 Mass (g)	$CaCl_2$ Mass (g)	NaCl Mass (g)	KCl Mass (g)	Liquid Water Added (g)	*SS Disk 1 (g)	**SS Disk 2 (g)	CC Tare Weight (g)	CC Gross Weight (g)	IC Tare Weight (g)	IC Gross Weight (g)	OC Tare Weight (g)	OC Gross Weight (g)
1-E-D	1572.6	0.0	0.0	0.0	0.0	0.0	3440.5	1499.6	6512.7	1740.3	8253.0	4168.5	12421.5
2-E-12	1572.2	0.0	0.0	0.0	11.8	0.0	3441.9	1497.6	6523.5	1715.5	8239.0	4138.1	12377.1
3-E-18	1572.2	0.0	0.0	0.0	17.8	0.0	3443.9	1501.3	6535.2	1676.2	8211.4	4153.9	12365.3
4-E-6	1572.2	0.0	0.0	0.0	5.8	0.0	3443.0	1505.0	6526.0	1708.0	8234.0	4144.2	12378.2
5-E-6-S	1107.0	20.1	221.9	221.8	4.1	0.0	2603.8	1496.3	5673.1	1736.0	7409.1	4151.1	11560.2
6-E-12-S	642.1	40.3	444.1	444.1	8.4	0.0	1767.7	1504.2	4849.7	1693.8	6543.5	4161.5	10705.0
7-A-D	1958.5	0.0	0.0	0.0	0.0	1029.1	1093.5	525.4	3577.4	1179.6	5786.1	4074.0	9860.1
8-A-6	1958.2	0.0	0.0	0.0	5.7	1027.2	1093.3	522.3	3579.2	1178.5	5784.9	4092.7	9877.6
9-A-6	1958.4	0.0	0.0	0.0	5.7	1027.5	1091.8	517.0	3572.8	1178.1	5778.4	4092.5	9870.9
10-A-12	1958.2	0.0	0.0	0.0	11.8	1026.6	1091.8	525.7	3587.8	1177.4	5791.8	4078.2	9870.0
11-A-6-S	1493.2	20.1	221.9	221.8	4.1	1025.9	253.0	515.8	2729.2	1180.1	4935.2	4084.4	9019.6
12-A-12-S	734.1	40.4	444.1	444.1	8.3	1025.4	0.0	519.8	2190.5	1177.1	4393.0	4087.4	8480.4

*SS Disk 1 was placed outside of the convenience container.

**SS Disk 2 was placed inside of the convenience container.

Appendix 5. Container Loading Data (cont.)

Table 14. Weight Losses From Full Batch Heating and Post-Treatment Analyses

Container	Al_2O_3 400 °C (wt%)	Al_2O_3 600 °C (wt%)	NaCl 500 °C (wt%)	KCl 500 °C (wt%)		Al_2O_3 LOI (1000 °C) (wt%)	Al_2O_3 H_2O est. (wt%)	NaCl LOH 650 °C (wt%)	KCl LOH 650 °C (wt%)	$CaCl_2$ LOH 200 °C (wt%)
1-E-D	0.1840	n.a.	n.a.	n.a.		0.0738	0.0487	n.a.	n.a.	n.a.
2-E-12	n.a.	0.1975	n.a.	n.a.		0.0230	0.0152	n.a.	n.a.	n.a.
3-E-18	n.a.	0.1977	n.a.	n.a.		0.0210	0.0139	n.a.	n.a.	n.a.
4-E-6	n.a.	0.2041	n.a.	n.a.		0.0147	0.0097	n.a.	n.a.	n.a.
5-E-6-S	n.a.	0.2003	0.1396	0.0200		0.0189	0.0125	0.0249	0.0171	8.1410
6-E-12-S	n.a.	0.1892	0.1824	0.0407		0.0209	0.0138	0.0261	0.0252	8.1773
7-A-D	0.1796	n.a.	n.a.	n.a.		0.0355	0.0234	n.a.	n.a.	n.a.
8-A-6	n.a.	0.2012	n.a.	n.a.		0.0159	0.0105	n.a.	n.a.	n.a.
9-A-6	n.a.	0.2044	n.a.	n.a.		0.0176	0.0116	n.a.	n.a.	n.a.
10-A-12	n.a.	0.2483	n.a.	n.a.		0.0151	0.0099	n.a.	n.a.	n.a.
11-A-6-S	n.a.	0.2003	0.1396	0.0200		0.0189	0.0125	0.0249	0.0171	8.1410
12-A-12-S	n.a.	0.1977	0.1590	0.0416		0.0252	0.0166	0.0220	0.0131	8.2326