LA-UR-19-28746

Title: LANL Phase 3 Test Plan For Container Loading to Support the Fire-Induced Pressure Response and Failure Characterization of PCV/SCV/3013 Containers

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

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1.0 Introduction

1.1 Purpose

Fire testing of 3013 containers loaded with surrogate materials and water will be conducted to determine the response of 3013 containers stored in K-Area Complex (KAC) to a facility fire [1]. Based on the proposal for the test configuration and loading, the objectives of the fire test are as follows [2]:

1. "To increase understanding about 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."

Surrogate materials will be prepared at Los Alamos National Laboratory (LANL) and loaded in the 3013 containers. The containers will then be welded at LANL and shipped to Savannah River Site (SRS) for the installation of fittings and leak testing.

1.2 Scope

This test plan documents the containers and material loading parameters for the 3013 test containers and provides the necessary travelers and data sheets for material and container preparation. The 3013 outer containers include a machined version manufactured by the Westinghouse Engineered Products Division (EPD) and a flowform version manufactured by Dynamic Flowform. The machined version of the 3013 outer container will have a configuration consisting of the EPD Outer 3013, the bagless transfer inner container (BTIC), and the bagless transfer convenience container (BTCC), and was the configuration used to package Hanford materials. The flowform version of the 3013 outer container will have a configuration consisting of an the Advanced Recovering and Integrated Extraction System (ARIES) Outer 3013, and ARIES Inner 3013 container, and a crimp seal Cogema-style convenience container.

The fire tests support the KAC safety basis and must be bounding of the maximum pressure that would result in a fire scenario. Therefore, the containers are loaded with material to target a free gas volume resulting in the maximum gas pressure [3]. The materials include aluminum oxide to simulate the thermal properties of plutonium oxide; water to bound the moisture content of the stored material; and, in some cases, a chloride salt mixture to simulate high-temperature corrosion. Additionally, stainless steel disks are added to the payload to achieve a minimum free gas volume that bounds the 3013 containers in storage.

2.0 Locations

Convenience containers will be loaded inside an atmosphere controlled glovebox with helium in TA55, building 3. Container welding will be performed in the following locations:

- BTIC: TA3, SM39
- ARIES Inner 3013: TA55, building 3
- EPD Outer 3013: TA55, building 4
- ARIES Outer 3013: TA55, building 4

3.0 Procedures

The scope of work requires multiple sequential steps to prepare each uniquely loaded 3013 container per the test matrix. Each of the activities are performed according to approved LANL procedures, and data will be collected on the appropriate data sheets in those procedures. This test plan also includes data sheets and travelers to document the material preparation and loading of containers. Data sheets will signed off by LANL quality assurance and LANL operators. In the event of a breached container, a rework will be initiated.

The closing of each loaded container in the nested set will be documented in Attachment A, *Test Container Traveler.* The *Test Container Traveler* will document the final package weight for each container in the nested set (i.e. Convenience Container, Inner 3013 Container, Outer 3013 Container), the unique identifiers for each container in the nested set, and the date and time of closure for each container. The *Test Container Traveler* will also document the successful passing of the helium leak test for each Inner 3013 and Outer 3013.

Preparation of the aluminum oxide and salts for loading will be performed according to Attachment B, *Data Sheet for Material Preparation and Sampling.* Loading of convenience containers will be performed according to Attachment C, *Data Sheet for Convenience Container Loading*.

3.1 Aluminum Oxide Handling and Staging

Aluminum oxide powder is non-hazardous, but will be handled in a fume hood to control dust. The aluminum oxide will be heated in air to remove adsorbed species, primarily carbon dioxide and water. The calcination time and temperature will be specified by the MIS Working Group and approved by the MIS Working Group Chair. Batches of heated material will be staged in a dry, atmosphere-controlled glovebox prior to loading of containers to reduce the potential for adsorption of moisture and CO_2. The oxide preparation data will be collected in Attachment B, *Data Sheet for Material Preparation and Sampling*.

3.2 Chloride Salt Handling and Staging

The chloride salts (NaCl, KCl, and anhydrous $CaCl_2$) will be staged in a dry, atmosphere controlled glovebox with helium prior to loading of containers to reduce the potential for adsorption of moisture from the ambient air. The salts may be heated in dry helium to remove adsorbed water. The calcination time and temperature will be approved by the MIS Working Group Chair. The salt preparation data will be collected in Attachment B, *Data Sheet for Material Preparation and Sampling.*

3.3 Moisture Analyses

Samples of the aluminum oxide will be analyzed by thermogravitational analysis with mass spectrometry (TGA-MS) or by loss on ignition (LOI). Characterization will be performed on the material following calcination to determine the amount of water and CO_2 adsorbed on the material. The results from these analyses will be documented in Attachment B, *Data Sheet for Material Preparation and Sampling*. The measured adsorbed moisture will be deducted from the water to be added to the material during loading.

Samples of the chloride salts (NaCl, KCl, and $CaCl_2$) will be analyzed by thermogravitational analysis with mass spectrometry (TGA-MS) or by loss on heating (LOH) to determine the moisture content prior to mixing with the aluminum oxide. The TGA and LOH will be performed at temperatures below the melting point of the salts. The temperature for the LOH will be approved by the MIS Working Group Chair. The results from the moisture analyses will be documented in Attachment B, *Data Sheet for Material Preparation and Sampling*. The measured adsorbed moisture on the salts will be deducted from the water to be added to the material during loading, and the salt components will be added to the aluminum oxide based on the weight of the dry salt components.

3.4 Convenience Container Loading

Convenience containers will be loaded inside a dry atmosphere glovebox in TA55, building 3. Containers will be loaded with predetermined amounts of aluminum oxide, stainless steel filler material, water, and salt as specified in Table 1. The chloride salts will be added by simple mixing. Liquid water will be added by pipette. Data collection for the convenience container loading is completed according to Attachment C, *Data Sheet for Convenience Container Loading*.

Cogema convenience containers require crimping. Crimping is done in the dry atmosphere glovebox in TA55, building 3 according to *Canning Material Using the Manual Convenience Canner (U),* PMT4-DOP-037. After loading, the containers will be inspected for proper closure.

Loaded convenience containers will be staged and transferred to welding locations inside a SAVY 4000 container with a hermetic cap installed over the filter to minimize adsorption and desorption of moisture.

3.5 BTIC Welding

Bagless Transfer Inner Containers are welded by Pit Technologies – Assembly and Welding Group (PT-3) at TA-03 SM-39 according to *Use of Welding Processes,* IWD-0013734. Welds are made using an AMI 9-7500 orbital weld head powered by an AMI model 307 power supply. A 3/32 inch ceriated tungsten electrode set to an arc gap of 0.063 inches is used with argon shielding gas. 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. A pause in rotation corresponding with a reduced arc current allows some solidification to occur at the periphery of the weld pool, preventing the weld pool from becoming too large. Without stepped travel, the thin can wall will melt much faster than the thick plug wall and steady-state welding conditions are not achieved. Welds meet the visual inspection and leak rate criteria of HNF 7082 Rev 4 – Fluor Hanford Nuclear Material Stabilization Project Welding Manual.

The BTICs will be welded with helium backing gas to produce welds representative of welds produced at Hanford in 50% helium. When using helium backing gas, the container will be quickly purged by flowing helium and welded under helium cover gas.

3.6 ARIES Inner 3013 Container Welding:

The ARIES Inner 3013 containers will be welded at TA55, building 3 inside a helium atmosphere glovebox. Welding will be performed according to *Gas Tungsten Arc Welding,* PMT4-DOP-130. The convenience containers will be moved into the welding glovebox without a pump down, and the atmosphere of the glovebox will be adjusted to meet the requirements in PMT-DOP-130. Leak checking will be performed according to *Helium Leak Testing a 3013 Container,* PMT4-DOP-139. Data collection for the ARIES Inner 3013 welding process is completed according to *Plutonium Packaging for Storage,* PMT4-DOP-043.

3.7 EPD Outer 3013 Container Welding

The EPD Outer 3013 containers will be welded at TA55, building 4 inside a helium atmosphere glovebox. Welding will be performed according to *Outer Container Welding for Long-Term Storage,* PMT4-DOP-044. Leak checking will be performed according to PMT4-DOP-139. Data collection for the ARIES Inner 3013 welding process is completed according to *Plutonium Packaging for Storage,* PMT4-DOP-043.

3.8 ARIES Outer 3013 Container Welding

The ARIES Outer 3013 containers will be welded at TA55, building 4 inside a helium atmosphere glovebox. Welding will be performed according to *Outer Container Welding for Long-Term Storage,* PMT4-DOP-044. Leak checking will be performed according to *Helium Leak Testing a 3013 Container,* PMT4-DOP-139. Data collection for the ARIES Inner 3013 welding process is completed according to *Plutonium Packaging for Storage,* PMT4-DOP-043.

3.9 Movement of Containers

Movement of closed containers across the TA55 protected area (PA) or the TA55 material access area (MAA) boundaries will be performed according to *Inspection Process for TA-55,* TA55-AP-113 and must have a completed Inspection Verification Form (TA55-AP-113, Attachment 1) for TA55 signed by two (2) human reliability program (HRP) operators.

4.0 Test Matrix

Twelve 3013 container loading configurations have been selected for the test matrix and are given in Table 1 [1-3]. The test matrix includes six outer 3013 containers produced by the Flowform process and the associated ARIES Inner container and Cogema-type convenience container, and six Outer 3013 containers produced by machining and the associated BTIC and BTCC. The containers will be loaded with aluminum oxide, which serves as a surrogate material for plutonium oxide. Some containers will also have water, salt, and stainless steel filler material. The levels of water in the test matrix were chosen in order to bound the moisture content of the materials of the packaged in the machined and flowform outer 3013 containers. Salts are added to the containers 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. Stainless steel filler material will be added to achieve a free gas volume that is conservative for the packaged materials.

Table 1. Test matrix specifying the aluminum oxide mass, stainless filler material, mass of salt and mass of water for each test container.

	Configuration Outer/Inner/ CC	Mass Al_2O_3 (g)	Mass SS Disk 1 (Outside CC) (g)	Mass SS Disk 2 (g) (Inside CC)	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

5.0 Drawings

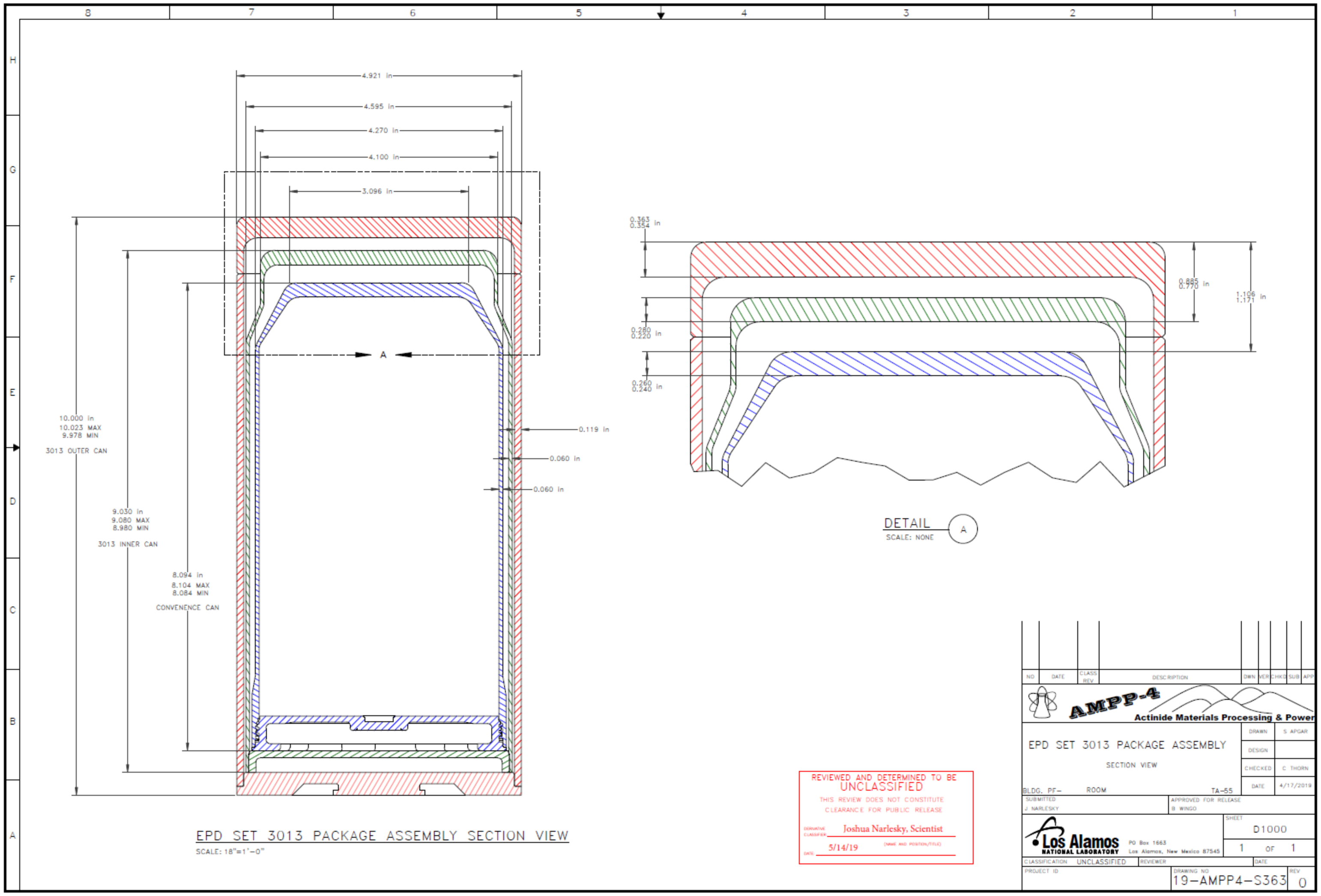

Figure 1. 3013 container assembly consisting of the EPD outer, BTIC, and BTCC.

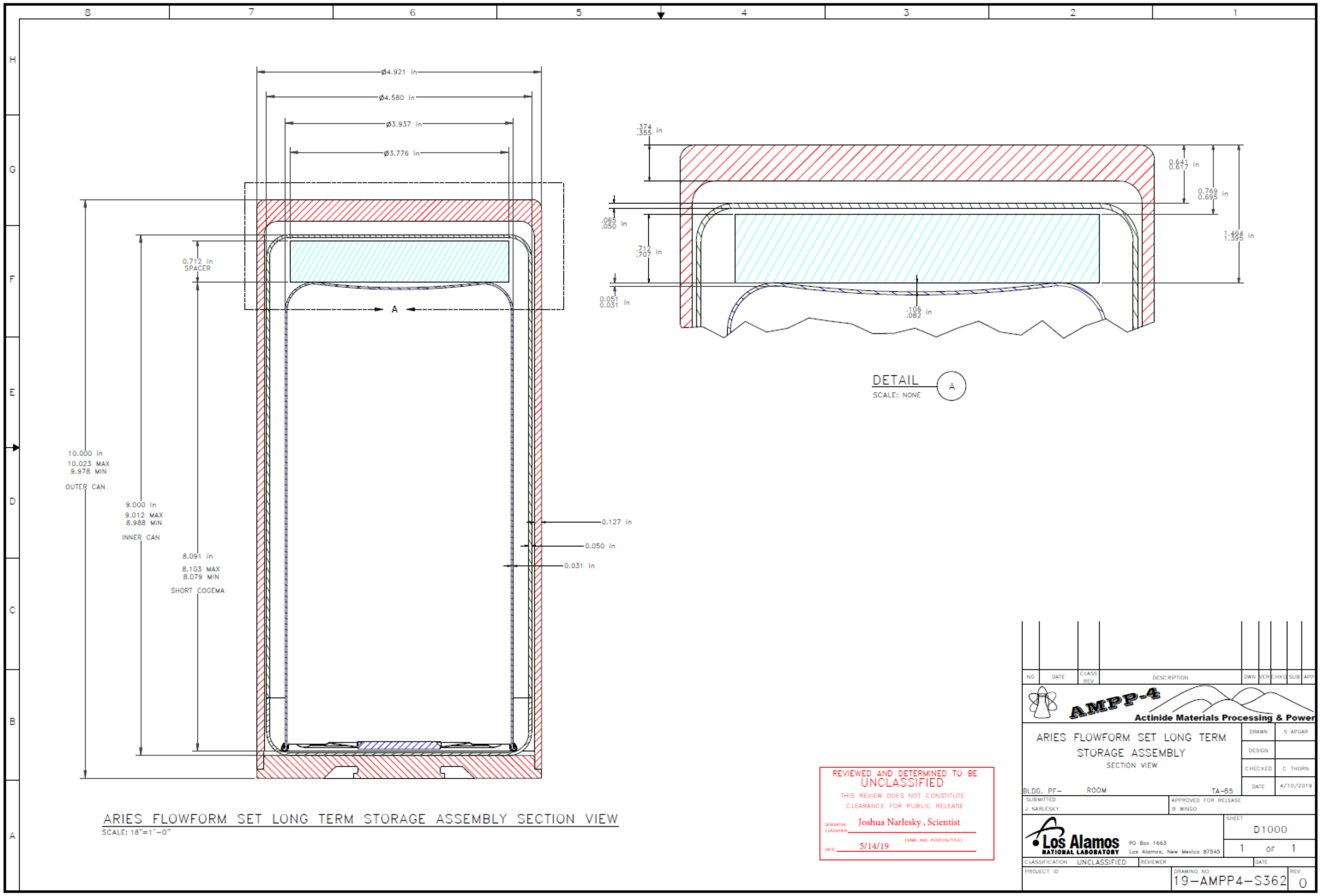

Figure 2. 3013 container assembly consisting of the ARIES outer, ARIES inner, and Cogema convenience container.

6.0 Materials and Equipment

Table 2. 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	111536	20 kg		
Check Weight Mettler Toledo	111537	20 kg		
Check Weight Mettler Toledo	111538	20 kg		

Table 3. Process Materials

Process Material Lot Number	Title/Description	Quantity
J4904515708	Aluminum oxide	22.7 kg
188345	Sodium chloride	3 kg
187537	Potassium chloride	3 kg
89866	Calcium chloride	250 g

6.1 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.

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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.
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*Based on suggested storage condition.

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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.

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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

Certified by:

QC Specialist

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7.0 Definitions and Acronyms

Term	Definition
ARIES	Advanced Recovering and Integrated Extraction System
BTCC	Bagless transfer convenience container
BTIC	Bagless transfer inner container
EPD	Engineered Products Division
HRP	Human reliability program
KAC	K-Area Complex
LANL	Los Alamos National Laboratory
LOH	Loss on heating; an analytical technique to used measure the amount of volatile constituents on a solid sample. The technique involves heating a sample in a furnace to a specified temperature and measuring the resulting change in the sample mass.
LOI	Loss on ignition; for purposes of this test plan, loss on ignition specifically refers to a loss on heating measurement performed at 1000 °C.
MIS	Materials Identification and Surveillance
SS	Stainless steel
SRS	Savanna River Site
TGA	Thermogravitational analysis

8.0 References

1. Letter from MIS Working Group to M. Maxted titled, “Fire Testing of 3013 Containers”, Dated 16 May 2019.
2. Memorandum from S. J Hensel and R. A. Sprankle to H. W. Coleman titled, “Recommended 3013 Loadings and Configuration for Fire Testing”, Dated 15 May 2019.
3. Veirs, D. K., McClard, J., and S. Hensel, “Calculation of fill and filler masses and volumes for 3013 fire test”, Los Alamos National Laboratory Report LA-UR-19-22902, Issued 15 May 2019 (rev. 2).

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*
TA55-AP-113	*Inspection Process for TA-55*
PA-AP-01040	*Records Processing Procedure for ADPSM Organizations*

9.0 Records

Records generated as a result of implementing this procedure are maintained in accordance with *Records Processing Procedure for ADPSM Organizations*, PA-AP-01040.

Record Identification	Record Type Determination	Protection/Storage Methods	Processing Instructions
Test Container Traveler	QA Record	Store records in accordance with PA-AP-01040.	When the records are ready for final disposition, the record is transferred to Records Management in accordance with PA-AP-01040.
Material Preparation and Characterization Data Sheet	QA Record		
Data Sheet for Convenience Container Loading	QA Record		

10.0 Appendices and Attachments

Appendix	Title
1	*Use of LOI/LOH to Determine Moisture Content*

Attachment	Title
A	*Test Container Traveler*
B	*Material Preparation and Characterization Data Sheet*
C	*Data Sheet for Convenience Container Loading*

Appendix 1, Use of LOI/LOH to Determine Moisture Content

Joshua Narlesky, Los Alamos National Laboratory, AMPP-4
Jared Stritzinger, Los Alamos National Laboratory, AMPP-3

Characterization of the aluminum oxide and salt components will be performed to determine the amounts of adsorbed moisture and CO_2 prior to loading test containers. This test plan specifies that thermogravitational analysis with mass spectrometry (TGA-MS) or loss on ignition (LOI) / loss on heating (LOH) are the as the methods that will be used to determine the amounts of adsorbed species on the aluminum oxide. The moisture content of the salt components will be determined by TGA or by loss on heating (LOH) to a specified temperature below the respective melting temperatures of the salt components. Due to a planned outage of the TGA system at LANL, LOI/LOH has been proposed as the primary method for determining moisture.

The use of LOI to determine the moisture content of the aluminum oxide is supported by the results from a set of LOI and TGA measurements performed on samples of aluminum oxide obtained before and after heating. The samples include the SRS unheated, LANL unheated, and LANL heated. The SRS unheated sample was obtained from the batch of aluminum oxide used for the Phase 1/2 test containers prepared by SRNL. The LANL samples were obtained from the batch of aluminum oxide procured by LANL for the Phase 3 containers. The LANL heated sample was heated to 400ºC in air for 4 hours. All three samples were stored in a dry, atmosphere-controlled glovebox with helium. The samples for TGA were loaded in conflat containers and transported to the TGA system located in a dry, air glovebox in PF4. The LOI samples were heated in the dry helium-filled glovebox where they were stored.

The TGA and LOI results for the aluminum oxide samples are summarized in Table A1. The TGA results for the SRS unheated sample and the LANL heated samples are shown Figures A1 and A2, respectively. The results of the unheated samples show that water and CO_2 are the only adsorbed species on the material. The average total mass loss for the unheated material was 0.33 wt%, of which, 64% was water. The LOI total mass loss was consistent with the TGA total mass loss.

The unheated LANL material has not received a TGA measurement. The LOI total mass was 0.19 wt%. The LANL heated material had both LOI and TGA measurements. The LOI total mass loss was consistent with the TGA total mass loss. The LOI and TGA results for the LANL heated material show that heating in air to 400ºC for 4 hours reduces the total amount of adsorbed water and CO_2 to less than 0.05 wt%. The remaining water and CO_2, respectively 0.020 wt% and 0.014 wt%, will not affect the results of the fire test.

These results show that using the total mass loss to estimate the moisture content for the heated material would result in errors of approximately 2% or less of the total water to be added to the materials. However, the TGA measurements for the heated and unheated samples show that the ratio of measured water to the total mass loss is consistent for both materials. Assuming that the ratio of the measured water to the total mass loss would be the same for all future samples, the errors could be reduced by adjusting the LOI total mass loss by the ratio of measured water to the total mass loss.

Table A1. TGA and LOI results for the aluminum oxide samples.

Sample	TGA Total Mass Loss (wt%)	H_2O (wt%)	CO_2 (wt%)	Ratio H_2O / total	LOI Total Mass Loss (wt%)
SRS Unheated	0.32	0.21[a]	0.11[b]	0.66	0.36
SRS Unheated	0.34	0.21[a]	0.13[b]	0.62	
LANL Unheated	Not Measured				0.19
LANL Heated 400ºC / 4 hr.	0.034	0.020[a]	0.014[b]	0.59	0.03

[a] measured by calibrated mass spec
[b] calculated as $Total\ Mass\ Loss - H_2O\ wt.\%$

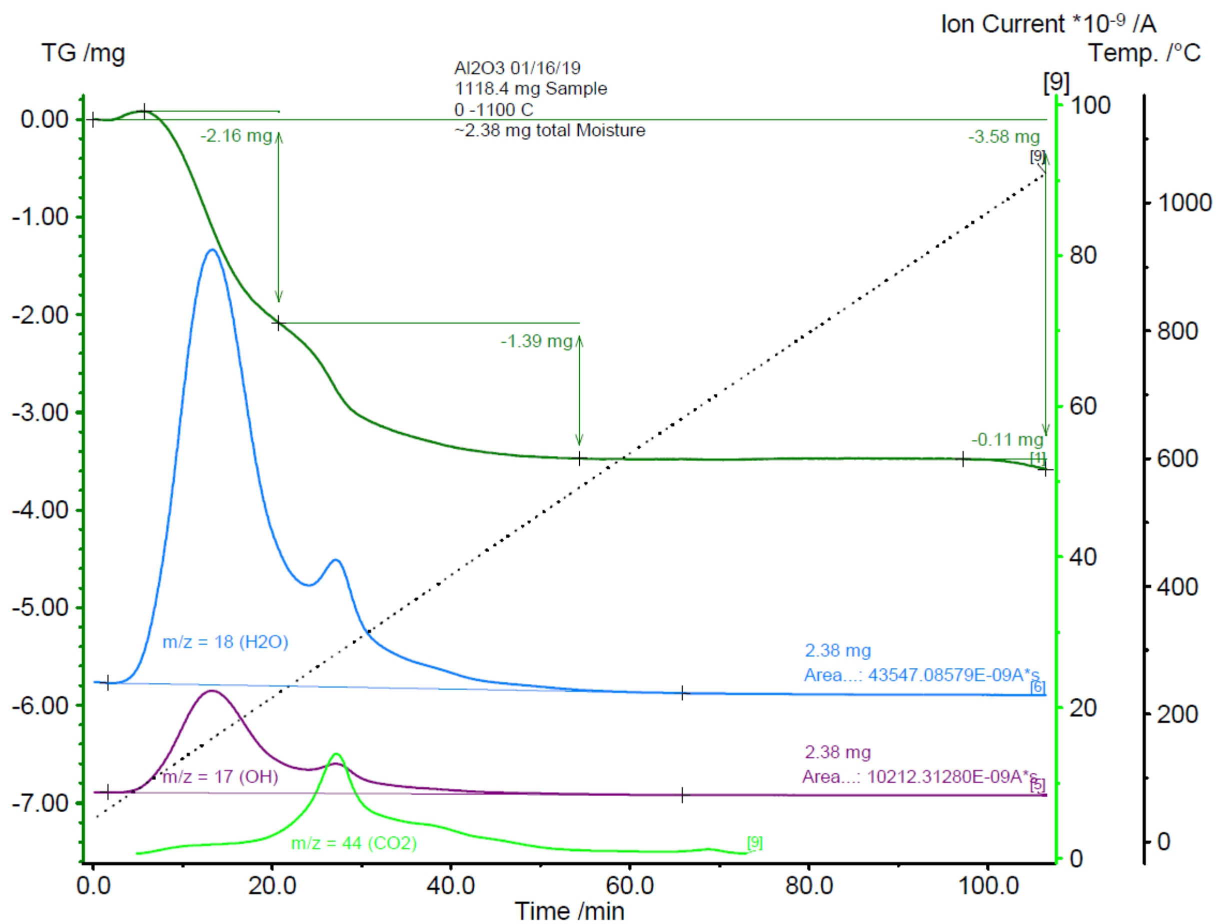

Figure A1. TGA results for the SRS unheated sample. The SRS unheated sample has a total mass loss of 0.34 wt% (3.58 mg). The MS shows two species, water and CO_2, and that both species are removed by 400 C. The MS gives the water as 2.38 mg or 0.21 wt%. Assuming the remainder of the mass loss is CO_2, the material has 0.11 wt% CO_2.

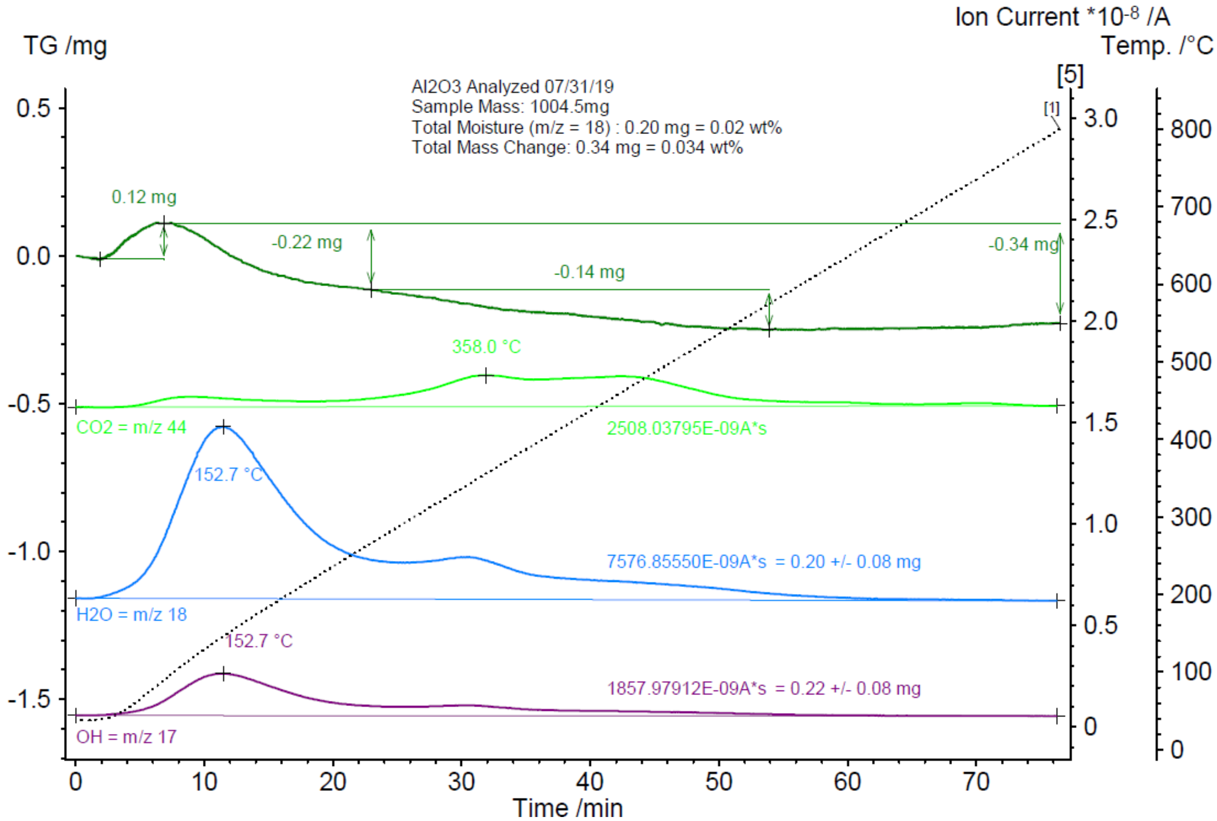

Figure A2. TGA results for the LANL heated sample. The LANL heated sample has a total mass loss of 0.034 wt% (0.34 mg). The MS shows both water and CO_2 but in smaller amounts than in the unheated material. The water CO_2 are evolved until about 600 ºC. The MS gives the water as 0.2 mg or 0.02 wt%. Assuming the remainder of the mass loss is CO_2, the material has 0.01 wt% CO_2.

The salt materials including the NaCl, KCl, and $CaCl_2$ will also be characterized before use to determine the adsorbed species. The TGA results for the $CaCl_2$ material are shown in Figure A3. The total mass loss to 650 ºC was 8.13 wt%. The MS results show that the loss was almost entirely due to removal of adsorbed water, and the loss due to adsorbed CO_2 was insignificant. The MS did not detect HCl, which indicates that the $CaCl_2$ did not hydrolyze during heating under inert conditions. Samples of $CaCl_2$ were heated to 200, 500 and 650 ºC under inert atmosphere to determine the LOH, and the results are summarized in Table A2. The results for the LOH to 200 ºC are most consistent with the TGA total mass loss.

Table A2. TGA and LOH results for the calcium chloride.

Temperature (ºC)	TGA Total Mass Loss (wt%)	LOH Total Mass Loss (wt%)
200 ºC	Not Measured	8.19
200 ºC	Not Measured	8.22
500 ºC	Not Measured	8.41
650 ºC	8.13	8.44

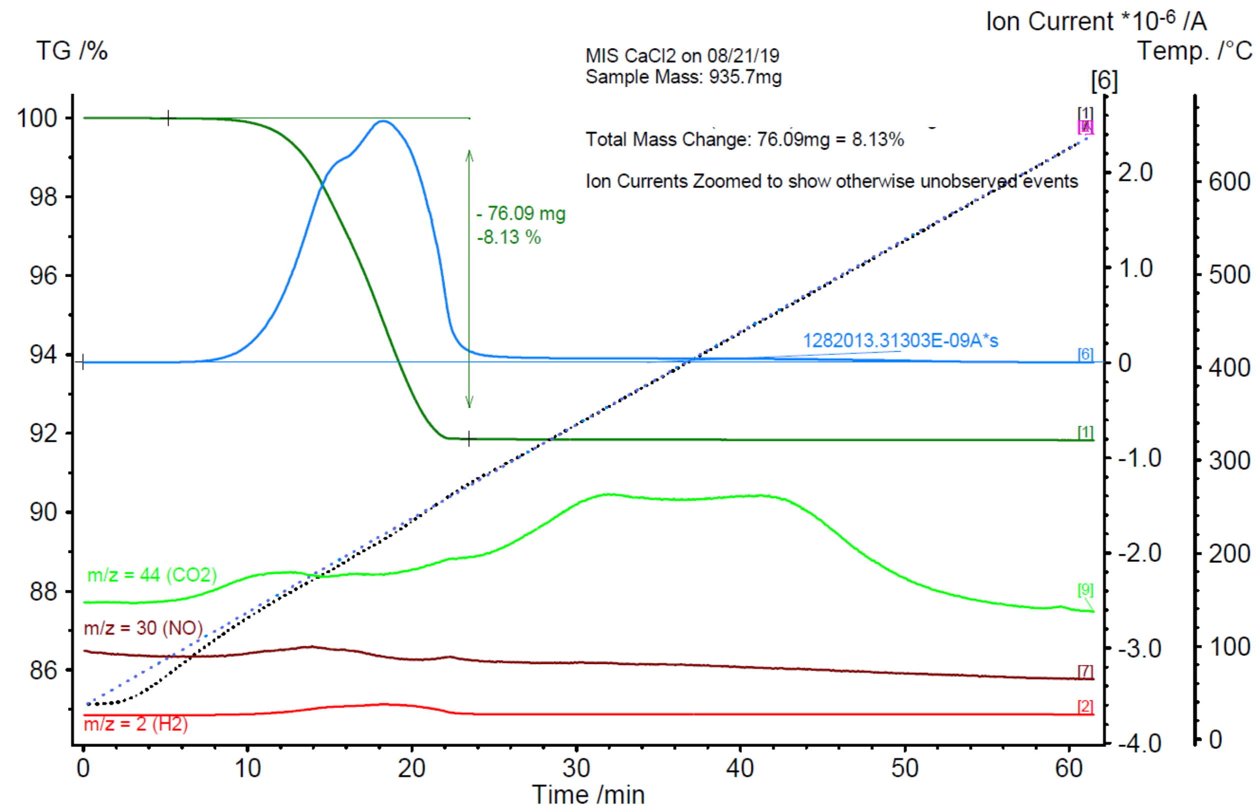

Figure A3. TGA results for the $CaCl_2$ sample. The $CaCl_2$ sample has a total mass loss of 8.13 wt% (76.09 mg). The MS shows two species, water and CO_2. The mass loss is attributed to removal of water, which was shown to be complete by 325 ºC. (The amount of water could not be accurately determined from the MS because the gram amounts exceeded the range of the calibration.)

Samples of KCl and NaCl were heated to 200, 500 and 650 ºC under inert atmosphere to determine the LOH, and the results are summarized in Table A3. Both salts show significant mass losses above 200 ºC, and additional measurements are needed explain the mass loss.

Table A3. LOH results for the potassium chloride and sodium chloride.

Temperature (ºC)	KCl LOH Total Mass Loss (wt%)	NaCl LOH Total Mass Loss (wt%)
200 ºC	0.01	0.01
500 ºC	0.20	0.36
650 ºC	0.24	0.40

Based on the TGA and LOI/LOH results obtained to date, the adsorbed moisture on the aluminum oxide can be determined by LOI performed under inert atmosphere prior to loading the convenience containers. The water can be determined by adjusting the total mass loss by the ratio of water to TGA total mass loss (average 0.62). The moisture on the calcium chloride can be determined by LOH to 200ºC performed under inert atmosphere. Additional characterization is needed to determine the adsorbed species on the KCl and NaCl.

Attachment A, Test Container Traveler

Page 1 of 1

Step	Description	Result	Initials
[1]	Record the **Test Container Number** (1-12) *(See Section 4.0).*		
[2]	Record the **Convenience Container ID**.		
[3]	Load the convenience container with material per Attachment C. Record the date/time the convenience container is closed.		
[4]	Record the mass of the loaded convenience container.		
[5]	Stage the loaded convenience container inside a SAVY-4000 with a hermetic cap. Record the SAVY-4000 Container ID and date of closure.		
[6]	Install TID on the SAVY-4000 container per TA55-AP-113 and record the TID number prior to transfer for IC welding .		
[7]	Place SS disk 1 inside the inner container so that it is oriented under the convenience container *(if applicable per Attachment C, C1[1])*.		
[8]	Record the **Inner Container ID**.		
[9]	Record the mass of the loaded inner container.		
[10]	Record the date/time the inner container is closed.		
[11]	Complete helium leak checking on the inner container and record the date/time the helium leak checking is complete.		
[12]	Install TID per TA55-AP-113 for the closed inner container and record the TID number prior to transfer for OC welding.		
[13]	Record the **Outer Container ID**.		
[14]	Record the mass of the loaded outer container.		
[15]	Record the date/time the outer container is closed.		
[16]	Complete helium leak checking on the outer container and record the date/time the helium leak checking is complete.		
[17]	Install TID per TA55-AP-113 for the closed outer container and record the TID number.		

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Attachment B, Data Sheet for Material Preparation and Sampling

Page 1 of 4

B1. Material Calcination and Sampling Parameters		
Step	**Description**	**Initials**
[1]	Record material calcination run parameters: Material | Calcination Temperature ºC | Ramp Rate ºC/min | Time at Temperature min ________ ________ ________ ________ ________ ________ ________ ________ ________ ________ ________ ________ ________ ________ ________ ________	
[2]	Record required analytical samples: Material | Analysis | Sample Size | No. Samples | Condition (Pre/Post Calc.) ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______ ______ ______ ______ g ______ ______	

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Attachment B, Data Sheet for Material Preparation and Sampling

Page 2 of 4

B2. Material Calcination and Sampling			
Step	**Description**	**Result**	**Initials**
[1]	Ensure balance is verified.		
[2]	Measure and record tare weight of furnace boat: **A**		
[3]	Identify material to be heated.		
[4]	Place material in furnace boat.		
[5]	Obtain pre-calcination analytical samples as specified in B1[2]. Assign sample IDs that incorporate the material, sample date, analysis technique, incrementing number as follows: Material-YYYYMMDD-Analysis-Number (e.g. AlOx-20190601-TGA-01); Sample ID / Sample Weight __________ __________ g __________ __________ g __________ __________ g __________ __________ g __________ __________ g __________ __________ g		
[6]	Label sample containers and stage samples in inert glovebox pending analysis.		
[7]	Measure and record the gross weight of the material in the furnace boat before heating. **B**		
[8]	Calculate and record the net weight before heating. **C=B-A**		
[9]	Place material in furnace. Calcine material per specifications in B1-1. Record the following: Material reached set point ______ : ______ Time (hh:mm) Begin furnace cool down ______ : ______ Time (hh:mm)		
[10]	Measure and record the gross weight of the material in the furnace boat after heating. **D**		

Attachment B, Data Sheet for Material Preparation and Sampling

Page 3 of 4

B2. Material Calcination and Sampling (cont.)			
Step	**Description**	**Result**	**Initials**
[11]	Calculate and record the net weight after heating. **E=D-A**		
[12]	Calculate and record the weight percent of species removed by calcination. **F= (D-B)/E × 100%**		
[13]	Obtain post-calcination analytical samples as specified in B1[2]. Assign sample IDs as described in B2[5]. Sample ID / Sample Weight __________ ______ g __________ ______ g __________ ______ g __________ ______ g __________ ______ g __________ ______ g		
[14]	Label sample containers and stage samples in inert glovebox pending analysis.		

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Attachment B, Data Sheet for Material Preparation and Sampling

Page 4 of 4

B3. Additional Characterization		☐ Not applicable per B1[2]	
Step	**Description**	**Result**	**Initials**
[1]	Obtain additional analytical samples as specified in B1[2]. Assign sample IDs as described in B2[5]. Sample ID Sample Weight ______________________ ________ g ______________________ ________ g ______________________ ________ g ______________________ ________ g ______________________ ________ g ______________________ ________ g		
[2]	Label sample containers and stage samples in inert glovebox pending analysis.		

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Attachment C, Data Sheet for Convenience Container Loading

Page 1 of 5

C1: Material Loading Parameters		
Step	**Description**	**Initials**
[1]	Record loading parameters. Test ID: ____________________ Outer Container Type: ____________________ Inner Container Type: ____________________ Convenience Container Type: ____________________ Mass SS Disk 1: ____________________g Mass SS Disk 2: ____________________g Mass Salt: ____________________g Mass NaCl (M_{NaCl}) (0.48 × Mass Salt): ____________________g Mass KCl(M_{KCl}) (0.48 × Mass Salt): ____________________g Mass $CaCl_2$ (M_{CaCl2}) (0.04 × Mass Salt): ____________________g Mass Aluminum Oxide: (M_{Al2O3}) ____________________g Total Mass Water (TMW): ____________________g	
[2]	Record the mass fraction of adsorbed water on each component. Mass Fraction Water on NaCl ($X_{W,NaCl}$): ______________ Mass Fraction Water on KCl ($X_{W,KCl}$): ______________ Mass Fraction Water on $CaCl_2$ ($X_{W,CaCl2}$): ______________ Mass Fraction Water on Al_2O_3 ($X_{W,Al2O3}$): ______________ Comments: ____ ____	

Attachment C, Data Sheet for Convenience Container Loading

Page 2 of 5

C1: Material Loading Parameters (cont.)		
[3]	Calculate the corrected mass of each component ($M_{X,C}$). $M_{NaCl,C} = M_{NaCl}$ / (1 – $X_{W,NaCl}$) $M_{NaCl,C}$ =__________ / (1 – __________) = __________g $M_{KCl,C} = M_{KCl}$ / (1– $X_{W,KCl}$) $M_{KCl,C}$ =__________ / (1 – __________) = __________g $M_{CaCl2,C} = M_{CaCl2}$ / (1– $X_{W,CaCl2}$) $M_{CaCl2,C}$ =__________ / (1 – __________) = __________g $M_{Al2O3,C} = M_{Al2O3}$ / (1– $X_{W,Al2O3}$) $M_{Al2O3,C}$ =__________ / (1 – __________) = __________g	
[4]	Calculate the corrected mass of water (CMW). (a) $X_{W,NaCl}$ × M_{NaCl} __________ × __________ = __________g (b) $X_{W,KCl}$ × M_{KCl} __________ × __________ = __________g (c) $X_{W,CaCl2}$ × M_{CaCl2} __________ × __________ = __________g (d) $X_{W,Al2O3}$ × M_{Al2O3} __________ × __________ = __________g Mass Adsorbed Water: MAW = a + b + c + d: = ________+________+________+________ = ________ g **Corrected Mass of Water: CMW** = TMW – MAW: ______________g	

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Attachment C, Data Sheet for Convenience Container Loading

Page 3 of 5

C2: Convenience Container Preparation			
Step	**Description**	**Result**	**Initials**
[1]	Ensure balance is verified.		
[2]	Obtain empty convenience container body and lid. Record ID of the convenience container body.		
[3]	Measure and record the tare weight of the empty convenience container body and lid. **A**		
[4]	Place SS Disk 2 into convenience container (if applicable) and record gross weight. **B**		
[5]	IF salt addition is required, THEN Complete C3 and add the aluminum oxide + salt mixture from C3 to the convenience container and record the gross weight. Record '0' if not adding salt. **K**		
[6]	IF salt addition is NOT required, THEN add the amount of aluminum oxide specified in C1[3] to the convenience container and record the gross weight. Record '0' if salt was added to the container. **L**		
[7]	IF moisture addition is required, THEN add slowly the corrected mass of water (CMW) to the center of the material in the convenience container by pipette and obtain the gross weight. **M**		
[8]	Close the convenience container. (Cogema type convenience containers are closed by crimping with the manual can crimper. BTCC containers lids are secured by tightening the threaded lid.		
[9]	Obtain final gross weight of loaded convenience container. **N**		

Attachment C, Data Sheet for Convenience Container Loading

Page 4 of 5

C2: Convenience Container Preparation (cont.)		
Step	**Description**	**Initials**
[10]	Calculate and record the masses of the individual components in the convenience container. Mass of Filler: (B-A): ____________________g Mass of salt added: (E) (if applicable): ____________________g Mass of water added (WA = N-L-K): ____________________g Mass of Aluminum oxide (H or L-B): ____________________g	
[11]	Calculate the total moisture (TM) based on the mass of adsorbed water (MAW) and the water added (WA) as follows: TM = MAW + WA ____________________g	

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Attachment C, Data Sheet for Convenience Container Loading

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C3: Aluminum Oxide + Salt Mixture Preparation	☐ Not applicable per C2[5]		
Step	**Description**	**Result**	**Initials**
[1]	Ensure balance is verified.		
[2]	Place empty beaker on balance. Zero balance. Weigh out the amount of NaCl specified in C1[3] into empty beaker and record the mass of NaCl.		
[3]	Place empty beaker on balance. Zero balance. Weigh out the amount of KCl specified in C1[3] into empty beaker and record the mass of KCl.		
[4]	Place empty beaker on balance. Zero balance. Weigh out the amount of $CaCl_2$ specified in C1[3] into empty beaker and record the mass of $CaCl_2$.		
[5]	Obtain empty mixing jar and place on balance. Record tare weight of mixing jar. **C**		
[6]	Add the contents of all 3 beakers to the mixing jar and record the gross weight. **D**		
[7]	Calculate and record the net weight of the salts. **E=D-C**		
[8]	Close mixing jar and mix the contents by shaking vigorously for 2 minutes.		
[9]	Obtain empty mixing jar and place on balance. Record tare weight of mixing jar. **F**		
[10]	Add the amount of aluminum oxide specified in C1[3] to the mixing jar and record the gross weight. **G**		
[11]	Calculate and record the net weight of the aluminum oxide. **H=G-F**		
[12]	Add the contents mixing jar holding the salts to the mixing jar holding the aluminum oxide and record the gross weight. **I**		
[13]	Calculate and record the net weight of the aluminum oxide + salt mixture. **J=I-F**		
[14]	Close mixing jar and mix the contents by shaking vigorously for 2 minutes.		
[15]	Return to C3[5] in Attachment C.		

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Attachment D, Data Sheet for Loss on Heating / Loss on Ignition

Page 1 of 2

D1. Material Calcination and Sampling Parameters		
Step	**Description**	**Initials**
[1]	Record parameters for loss on heating: Calcination Temperature: ____________________ Calcination Time (time at temperature): ____________________	
[2]	Measure and record weights **before** heating: Sample ID / Tare Weight (g) / Gross Weight (g) / Net Weight (g) ______________ ___________ ___________ ___________ ______________ ___________ ___________ ___________ ______________ ___________ ___________ ___________ ______________ ___________ ___________ ___________ ______________ ___________ ___________ ___________ ______________ ___________ ___________ ___________	
[3]	Place samples in furnace. Calcine material per specifications in D1[1]. Record the following: Material reached set point ______ : ______ Time (hh:mm) Begin furnace cool down ______ : ______ Time (hh:mm)	
[4]	Measure and record weights **after** heating: Sample ID / Gross Weight (g) / Net Weight (g) ______________ ___________ ___________ ______________ ___________ ___________ ______________ ___________ ___________ ______________ ___________ ___________ ______________ ___________ ___________ ______________ ___________ ___________	

Attachment D, Data Sheet for Loss on Heating / Loss on Ignition

Page 2 of 2

D1. Material Calcination and Sampling Parameters (cont.)

Step	Description	Initials
[5]	Calculate the weight change and the weight % change. Weight Change = Gross Weight After (g) – Gross Weight Before (g) Weight % Change = Weight Change (g) / Net Weight Before (g) Sample ID ______ Weight Change (g) ______ Weight % Change ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______	
[6]	Calculate the mass fraction of water based on the ratio of water to total mass loss R_W as specified by the SME. Mass Fraction of Water (X_W) = –Weight % Change × R_W × 1E-02 Sample ID ______ R_W ______ X_W ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______ ______	

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