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Simultaneous Thermal Analysis of Remediated Nitrate Salt Surrogates

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1.0 Executive Summary

The Actinide Engineering and Science Group (MET-1) have completed the Simultaneous Thermal Analysis and offgas analysis by Mass Spectrometry (STA-MS) of Remediated Nitrate Salt (RNS) surrogates (Table 1) formulated by the High Explosives Science and Technology Group (M-7). The 1.0 to 1.5 g surrogate samples were first analyzed as-received, then a new set was analyzed with 100-200 µL 10M HNO3 + 0.3 M HF added, and a third set was analyzed after 200 µL of a concentrated Pu-Am spike (in 10M HNO_3 + 0.3 M HF) was added. The acid and spike solutions were formulated by the Actinide Analytical Chemistry Group (C-AAC) using reagent-grade HNO_3 and HF, which was also used to dissolve a small quantity of mixed, high-fired PuO_2 / AmO_2 oxide.

Table 1: Sample matrix for RNS Surrogates

1% $Pb(NO_3)_2$ **15% Swheat** **84% WB-8**	**2% $Pb(NO_3)_2$** **15% Swheat** **83% WB-8**	**4% $Pb(NO_3)_2$** **15% Swheat** **81% WB-8**
1% $Pb(NO_3)_2$ **15% Swheat** **84% WB-8** **100-200 µL 10M HNO_3 + 0.3 M HF**	**2% $Pb(NO_3)_2$** **15% Swheat** **83% WB-8** **100-200 µL 10M HNO_3 + 0.3 M HF**	**4% $Pb(NO_3)_2$** **15% Swheat** **81% WB-8** **100-200 µL 10M HNO_3 + 0.3 M HF**
1% $Pb(NO_3)_2$ **15% Swheat** **84% WB-8** **200 µL Pu, Am in 10M HNO_3 + 0.3 M HF**	**2% $Pb(NO_3)_2$** **15% Swheat** **83% WB-8** **200 µL Pu, Am in 10M HNO_3 + 0.3 M HF**	**4% $Pb(NO_3)_2$** **15% Swheat** **81% WB-8** **200 µL Pu, Am in 10M HNO_3 + 0.3 M HF**

All samples were analyzed in triplicate. The thermal behavior of all surrogate samples—unspiked and spiked—is dominated by three basic phenomena: 1) an endothermic dehydration reaction which onsets between ~38 and 50 °C, 2) an exothermic reaction which onsets between 108 and 123 °C related to the rapid gas release, foaming and expansion of the sample, and 3) steady-state, and slightly exothermic, combustion of the foamed sample above ~150 °C. Stage 1 is dominated by the release of copious amounts of H_2O and mass losses between 35 and 45%. Stage 2 is marked by a sudden increase in the NO_x and CO_2 content of the offgas as H_2O begins to tail off. The sustained release of small amounts of CO_2 and lesser amounts of NO_x and H_2O is typical of Stage 3.

The thermal behavior of the RNS surrogates is remarkably consistent and repeatable, given their generally inhomogeneous nature. Varying amounts of lead nitrate in the RNS surrogates has only a slight—if any—effect on the observed thermal behavior. The surrogates containing 1% and 2% $Pb(NO_3)_2$ appeared to be slightly more reactive between ~70 and 115 °C than the formulation containing 4% $Pb(NO_3)_2$. Some of the 1% and 2% samples showed small exotherms near or at the base of the dehydration endotherm. Both the onset and cessation of foaming occur at similar temperatures and the mass losses during dehydration / foaming is also similar for all Pb concentrations.

The thermal behavior and offgas content of the as-received and acid-doped RNS surrogates are broadly similar. While several of the 1% and 2% $Pb(NO_3)_2$ samples exhibited small exotherms in the ~80 and 110°C region, this behavior was neither consistent nor predictable. In most cases, these small exotherms were accompanied by near-vertical spikes in the NO_x content of the offgas. The addition of the acid (and the Pu-Am spike) increased the slope of the foaming reaction.

When spiked with the Pu-Am solution, every surrogate sample showed significant exothermic activity and multiple, transient, near-vertical NO_x offgas maxima in the region between ~80 and 115 °C. In the Pu-Am spiked samples, these exotherms have a characteristically jagged, multi-peaked appearance, and are similar to the corresponding multiple NO_x peaks. The addition of an extra 100 μL of Pu-Am spike to one sample (300 μL of Pu-Am spike, total) did not increase the reactivity of the surrogate relative to the other Pu-Am-spiked runs.

We also analyzed the dried residue left after evaporating 200 μL of the spike in a Pt-Rh differential scanning calorimetry (DSC) pan. Phase identification was not performed, though it is likely that the residue consisted of a mixture of Pu and Am nitrate hydrates and anhydrous fluorides. Results of the STA-MS analysis show multiple losses of NO_x superimposed over a single dehydration event. Significantly, the first onset of NO_x emission occurs at ~60-65 °C. The endothermic de-nitrification reaction related to this peak is marked by a change in slope which onsets at 64.0 °C. Though the majority of the NO_x is released at much higher temperatures, the dried Pu-Am spike residue begins to release NO_x at temperatures that are ~40-50 °C lower than the first release of NO_x from $Fe(NO_3)_3{\cdot}9H_2O$ (e.g., Wayne, 2015).

In conclusion, the STA-MS testing of RNS surrogate indicates that the addition of 3.12 mg Am and 5.54 mg Pu (as 200 μL of a Pu - Am solution in 10M HNO_3 / 0.3M HF) to 1.0 to 1.5 g samples of three RNS surrogate formulations increases reactivity in the 75-115 °C region in each, relative to that of the surrogate alone. Spiking the RNS surrogate with a similar volume of the same HNO_3-HF acid solution with no SNM content changed its thermal behavior only incrementally, by comparison. We further speculate that the observed phenomena are related to the chemical reactivity of the actinide compounds, and are not related to their radioactivity.

2.0 Introduction

MET-1 completed STA-MS testing on three different RNS formulations (Table 1) on April 15, 2016. Simultaneous thermal analysis (STA) combines traditional thermogravimetric analysis (TGA – the monitoring of mass changes over a controlled temperature profile) with differential scanning calorimetry (DSC – the monitoring of heat flow into and out of the reacting sample over the same temperature profile). Gases generated by the STA are analyzed using quadrupole mass spectrometry (MS). This report details the results of these analyses, including all calibrations, documentation, and other analytical operations required under the test plan PA-PLAN-01186, "*Differential Scanning Calorimetry and Mass Spectrometry (DSC-MS) of Remediated Nitrate Salt (RNS) Surrogates,*" released for TA-55 operations on December 7, 2015.

All STA-MS operations follow the work-authorizing operating procedure for STA-MS analyses in PF-4; PMT2-MPR-DOP-015, R4, "*Thermal Analysis with Off-Gas Analysis by Quadrupole Mass Spectrometry*," approved for use on October 15, 2015. The data sheets intrinsic to PMT2-MPR-DOP-091 were used to record analytical results, and are combined with text and graphic output from the instrumentation in an Appendix to this document. The apparatus used for this study is: Netzsch STA409PC Luxx Simultaneous Thermal Analyzer, commission number 206.010.667/04; and Pfeiffer ThermoStar GSD301T3 Gas Analysis System.

3.0 Documentation

Ancillary documentation relating to RNS surrogate testing, and to the materials, software and procedures utilized are appended to this report. The content of these appendices are tabulated below (Table 2).

Table 2: List of Appendices

Appendix Number	Document Title / Number
1	PA-PLAN-01186, "*Differential Scanning Calorimetry and Mass Spectrometry (DSC-MS) of Remediated Nitrate Salt (RNS) Surrogates*" (Unclassified)
2	PMT2-MPR-DOP-015, R4, "*Thermal Analysis with Off-Gas Analysis by Quadrupole Mass Spectrometry"*
3	LA-UR-15-28116 "*Simultaneous Thermal Analysis of WIPP and LANL Waste Drum Samples: A Preliminary Report*" (Unclassified)
4	Chain of Custody documentation per QA-38, R0 "Chain of Custody," effective December 2, 2015 (Unclassified).
5	Manufacturer's Certifications for DSC Calibration Materials per DIN 51007 and ASTM E 967 (Unclassified)
6	Data Sheets from PMT2-MPR-DOP-015, R4 with text and graphic output from the Netzsch STA409PC.
7	STA-MS Lab Notebook Pages from February 17, 2016 to April 15, 2016 (Unclassified)
8	Safety / Non-Safety Software Determination, Categorization, and Software Risk Level (SRL): Form 2033

All testing was carried out according to PA-PLAN-01186, with some administrative modifications, which became necessary between the issuance of the plan and the beginning of testing. None of these changes affected the quality or integrity of the RNS surrogate analyses, and do not constitute non-conformances per LANL Policy P330-6, R10 "Nonconformance Reporting" (Effective: 03/15/16).

1) Under "Scope" (Section 1.2) only experiments on surrogate and surrogate + SNM (Phase 1 and Phase 2) have been performed. No analysis involving zeolite-based adsorbing agents has been planned, as of this writing.

2) Under "Precautions and Limitations (Section 2.0), the document used to expedite Chain of Custody for the RNS surrogate samples was not NF-QA-004, "*Sample Receipt, Processing and Storage for the Bulk Actinide Nuclear Forensics*." This entry was made in error, as it is a procedure designed for use in transporting radioactive samples. The procedure appropriately used for the transport of non-radioactive samples (and used throughout this study) was QA-38, R0 "Chain of Custody."

3) Under Section 4.1, "Addition of Radioactive Components" the sub-sampling described in steps 4.1.1 through 4.1.4 was performed at TA-9 by M-7 personnel. Multiple vials containing samples from the same surrogate lot were then sent by C-AAC to MET-1. The remaining steps in this section were carried out as-written by MET-1.

The data sheets in Appendix 6 were designed for use with actinide compounds intended for storage in 3013 cans. Due to a formatting error, page 55 (of 63) appears blank (see Appendix 2). These pages have been omitted from the data sheets. Further, the data sheets have headers that mark the document as "R3." This is a typographical error. The most current revision of the DOP on Documentum® is R4.

The nature of the RNS surrogate analyses is intrinsically different from that of actinide oxides intended for storage in 3013 cans. Two inconsistencies in the printed acceptance criteria do not apply to the RNS surrogate analyses and shall be disregarded: 1) RNS surrogate samples weigh less than 25 mg, and are not required to meet the 3< x <18 gram requirement printed on the data sheet, and 2) mass losses from the surrogate will always exceed the 3013 limit of 0.4 wt%. We also made several global, generic handwritten N/A annotations in the 'Comments' section of the data sheets, which apply to all of the runs. These are as follows:

1) M&TE: Calibration File Number for the thermocouple and DSC calibrations is N/A. Both are user-performed calibrations (UPCs) and, as such the Cal. File No. is not required. All thermocouple and DSC calibration data used for the RNS surrogate analyses are included in this document.

2) M&TE: Calibrated Thermometer for Water Chiller – calibration is no longer required and is marked N/A. The temperature readout on the chiller (constant at 18.0 °C) is sufficient is sufficient to ensure the quality of STA (TGA-DSC) analyses.

3) M&TE: Verification of temperature profile to 1100 °C is N/A – the analyses of the RNS surrogates need only go to 350 °C, per M-7.

4) Glovebox Conditions: Recording of the "Seal %RH and Temperature" is N/A: not required by the customer.

5) Total Moisture (H_2O): Recording moisture content data for the RNS surrogates is N/A: not required by the customer.

These and any other handwritten N/A markings on the data sheet are noted and explained in the "Comments" section.

4.0 Materials

4.0.1 RNS Surrogates

The RNS surrogates were fabricated by M-7 following PLAN-TA9-2443, Rev. B "*Remediated Nitrate Salt Surrogate Formulation and Testing Standard Procedure,*" released on February 16, 2016. The content of the nitrate component of the surrogate (nominally 81-84% of each sample, by weight, per Table 1) is summarized in Table 3. The remainder of the surrogate is comprised of 15% Swheat® organic adsorbing agent and varying concentrations (1, 2, and 4% by weight) of lead nitrate ($Pb(NO_3)_2$). Sampling of the surrogates into 1.0 to 1.5-gram quantities was carried out by M-7. Samples were packaged into 30 mL plastic scintillation vials, capped and placed into custody-sealed bags by C-AAC personnel (Appendix 4). The bags were over-packed in paint cans for safe transport, and received in the TA-55 Entry Control Facility (ECF) by MET-1 personnel. The paint cans were opened and the packing removed in the MET-1 Cold Lab (PF-3, Room 177), and the packaged vials were hand-carried into the TGA/DSC lab in PF-4. There, the custody-sealed bags were opened and the vials immediately introduced into the glovebox line. Chain of Custody documentation (per QC-38, R0) for all sample transfers from M-7 to MET-1 via C-AAC is included here in Appendix 4.

Table 3: Nitrate component (WB-8) of the RNS Surrogates

Material	**Milligrams**[a]	**Wt %**[b]
$Al(NO_3)_3·9\ H_20$	1883	3.20
$Ca(NO_3)_2·4\ H_2O$	7490	12.72
$Cr(NO_3)_3·9H_2O$	92	0.16
$Fe(NO_3)_3·9H_2O$	2861	4.86
$Mg(NO_3)_2·6H_2O$	21020	35.69
$NaNO_3$	4660	7.91
$(COOH)_2·2H_20$	1700	2.89
K_2CO_3	888	1.51
Water	2538	4.31

[a] *Masses are ±1 mg;* [b] *Weight % values are ±0.01 %*

4.0.2 Pu-Am Spike

The proposed mass of both Pu and Am to be added to the surrogate is 0.003 times the mass of the WB-8 salt (i.e., the total sample minus the Swheat®). These values correspond to the maximum values of the mass ratios of both Pu and Am to total nitrate salt in the parent drums of the existing RNS inventory at LANL and WCS (Veirs, 2016). The Pu-Am spike used for these studies, though similar in concentration, did not exactly match the ideal.

Acid mixtures and the Pu-Am spike were made by C-AAC personnel at TA-55 using reagent-grade acids and distilled de-ionized water. The concentrated Pu-Am spike was prepared by dissolving a mixed Pu-Am oxide in 10M HNO_3 + 0.3M HF. The dissolution process took nearly 2 weeks and resulted in 0.01947 L (19.47 mL) of a dark green solution. The total amount of dissolved mixed Am-Pu oxide was 0.8442 g, with an oxide ratio of 36% AmO_2 / 64% PuO_2. Taking 36% of 0.8442 yields 0.304 g AmO_2, and 64% of 0.8442 yields 0.540 g PuO_2. In 19.47 mL of solution, the concentrations of Am and Pu are, respectively: 0.304g Am / 0.01947L = 15.6 g/L Am, and 0.540g Pu / 0.01947L = 27.7 g/L Pu. Thus, 100 microliters (μL) of spike contain 1.56 mg Am and 2.77 mg Pu (oxide basis).

The surrogate samples weighed 1.0 to 1.5 grams, averaging 1.25 g (Brown, 2016). Of the 1.25 g samples, the total salt content was between 1.01g and 1.05g. In order to approximate the SNM levels observed in the WIPP drum (e.g., Veirs, 2016), we added 200 μL to the surrogate, to yield final mixtures containing 3.12 mg Am and 5.54 mg Pu per gram of nitrate salt mixture.

Table 4: Temperature and enthalpy calibration data for the Netzsch STA 409PC using the SRMs listed in Appendix 5

Instrument:	NETZSCH STA 409PC
File names:	WIPP temp 022916.ngb-tsv (T); WIPP 022916.ngb-esv (enthalpy)
Heating rate:	10 °C/min
Furnace:	STD SiC(PC)
Crucible:	DSC/TG pan Pt-Rh
Atmosphere:	Ar + air
Date:	2/29/2016 3:42:00 PM (T); 3:32:52 PM (enthalpy)

Substance	**Temp. Cert. °C**	*Temp. Meas. °C*	Temp. Corr. °C	**Enthalpy J/g**	*Peak Area uV*s/mg*	*Sensitivity Exp uV/mW*	Sensitivity Calc. uV/mW
Air	**36.2**	*32.8*	33.9	**--**	*--*	*--*	
Biphenyl	**69.2**	*68.7*	70.2	**-120.5**	*-114.7*	*0.952*	0.957
Benzoic acid	**122.4**	*121*	123	**-147.4**	*-132.9*	*0.902*	1.023
$RbNO_3$(trig>cub)	**164.2**	*163.6*	166	**-26.6**	*-33.22*	*1.249*	1.061
$RbNO_3$ (melt)	**285**	*282.7*	286	**-8.75**	*-9.134*	*1.044*	1.101
Ag_2SO_4	**426.4**	*421.3*	425.2	**-51.9**	*-54.25*	*1.045*	1.039
CsCl	**476**	*469.3*	473.4	**-17.2**	*-16.36*	*0.951*	0.999
K_2CrO_4	**668**	*666.2*	670.5	**-37**	*-32.36*	*0.875*	0.802
$BaCO_3$	**808**	*803.3*	807.3	**-94.9**	*-58.83*	*0.62*	0.657

Acid spikes were added to the surrogate per PA-PLAN-01186, Section 4.1. Due to a malfunction of the PF-4 trolley system, a micropipette was not available for spiking the surrogate with precisely-known volumes of acid. For SFWB8-15-1 and SFWB8-15-4, the acid spike was added using a commercially-available plastic dropper. The amount of HNO_3 / HF added to these samples was 1-2 drops, approximately equivalent to 100-250 μL. The micropipette was available for SFWB8-15-2, and was used to add 100 μL of HNO_3 / HF to the surrogate. At the time, the concentration of actinides in the final Pu-Am spike was not known, and it was assumed that 100 μL of acid would be an appropriate amount. The final concentration of Pu and Am in the Pu-Am spike required that 200 μL of spike be added to the RNS surrogates to approximate the values seen in actual waste drums. Thus, one of the acid-only runs of SFWB8-15-2 was repeated using 200 μL of acid. The micropipette was used for all Pu-Am additions, and each utilized a 200 μL volume.

After the spike was added to the sample vial, the nitrate solution was thoroughly mixed with the sample using a clean, disposable wooden stirring rod. Since the sample was already in a unique container, no sample transfer had to be performed, vastly reducing the interaction time of the sample with the very dry glovebox atmosphere. Typically, anywhere from 3 to 24 hours elapsed between the spike mixing and the initiation of the STA-MS run. Due to the intense foaming of the RNS surrogates during thermal analysis, only 10-20 mg aliquots were scooped from the sample vial directly into the Pt-Rh sample pan.

Table 5: MS gas analysis parameters.

Species	Nominal mass	Dwell Time (s)	Resolution
$(H_2)^+$	2	0.5	50
C^+	12	0.5	50
O^+	16	0.2	50
$(OH)^+$	17	0.2	50
$(H_2O)^+$	18	0.2	50
$(NO)^+$	30	0.2	50
$^{36}Ar^+$	36	0.05	50
$(CO_2)^+$	44	0.2	50
$(NO_2)^+$	46	1.0	50
$(SO)^+$	48	0.5	50

The Netzsch STA 409 PC STA-MS operated and maintained in PF-4 by MET-1 was calibrated for temperature and enthalpy measurements from 69.2 °C to 808.0 °C using a set of seven standard materials provided by the manufacturer. Individual certifications for each material are located in Appendix 5 of this document. Certified values for each standard reference material (SRM) and the calibration results are tabulated in Table 4. A balance calibration was performed on the STA-MS prior to the initiation of this study, on February 22, 2016. Samples were weighed on a benchtop lab balance located in the STA-MS glovebox (B366). This balance calibrated and checked daily by the Nuclear Component Operations Support Services Group (NCO-6) per PA-DOP-01492, "*Measurement Control of Balances and Revalidation of In-line Weights*."

Glovebox humidity and temperature were measured using a calibrated hand-held hygrometer / thermometer (Cal. File # 041888, exp. 10-25-16) maintained as Measuring and Test Equipment (M&TE) by the Production Planning and Control Group (NPI-2). A calibrated wall clock used for this study (Cal. File # 040480, exp. 8-3-16) is also maintained as M&TE by NPI-2. Calibrations of both items were performed by the on-site Standards and Calibrations Laboratory.

4.1 Software

The software used for the RNS surrogate analyses was Netzsch Proteus, version 6.1.0 / 02.09.2015 for Win XP and Win7, and Pfeiffer QuadStar / ThermoStar 32-bit, version 7.03 for W2000 and Win XP. Software risk level determinations for both are included in this report (Appendix 8). The software is COTS and is operable as-is with no modifications. The computer used for to run the software and acquire the data in PF-4 is a standalone PC with Win XP. Data from the mass spectrometer can be imported directly into Netzsch Proteus and superimposed on to the thermograms given the correct starting temperature of the analysis (see Appendix 6). A copy of the Proteus software is also maintained in the office, on a PC running the Win 7 OS to facilitate data processing and graphical display. Some data plotting was also accomplished using Origin 9.0 software, generically available through LANL's EDS system.

4.2 Samples and Run Conditions

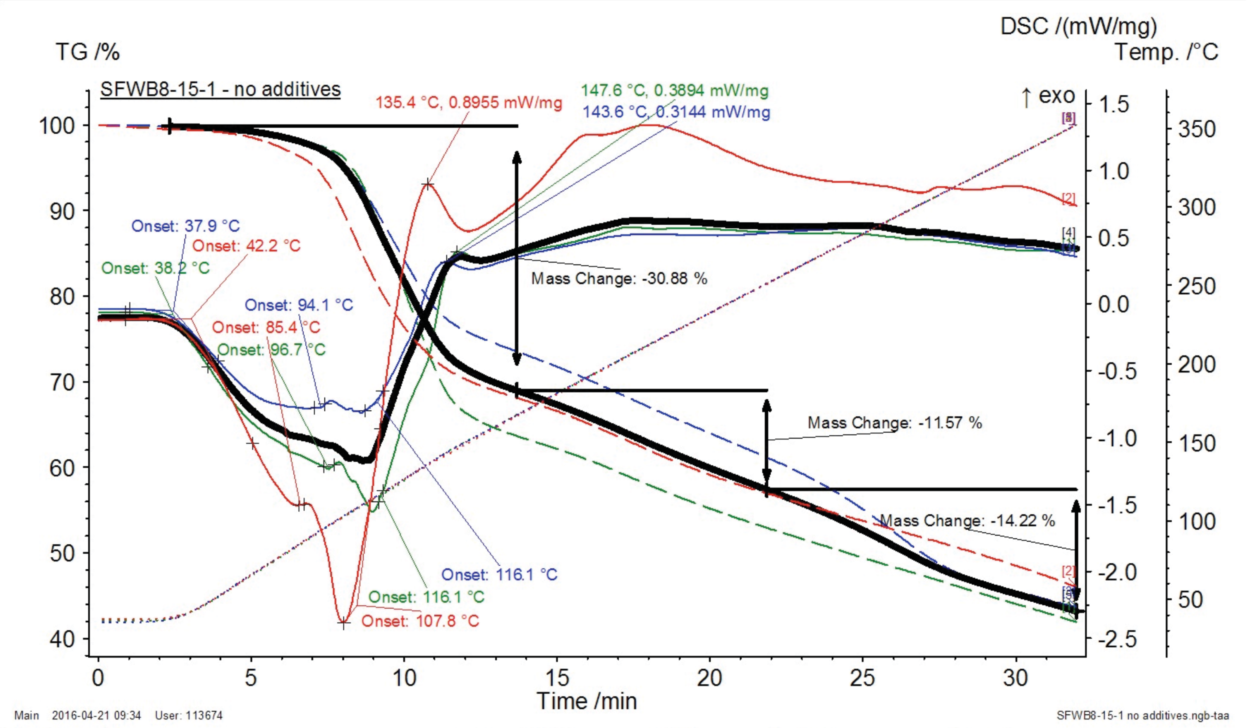
The samples consist of a yellow-brown paste containing small (~1 mm diam.) white-to-gray kernels in suspension. STA-MS analyses were conducted in triplicate, as specified in PA-PLAN-01186 R0, and were initiated either the afternoon of sample introduction, or the following morning. In all cases, analyses were complete before the 4-day expiration limit of the surrogates had been reached.

Table 6: Thermodynamic data for phase transitions in $K(NO_3)$, $Rb(NO_3)$ and $Ag_2(SO_4)$ measured in the TGA-DSC apparatus used for this study

Date / compound	Known Onset T. (°C)	Measured Onset T. (°C)	%RSD (1σ)	ΔH (J/g) known	ΔH (J/g) measured	%RSD (1σ)
03/01/2016 KNO_3	**128.7**	132.3	+2.8	**-50.0**	-62.0	-24.0
03/07/2016 KNO_3	**128.7**	131.5	+0.8	**-50.0**	-62.1	-24.2
03/15/2016 $RbNO_3$	**164.2**	165.9	+1.0	**-26.6**	-31.3	-4.7
	285	286.0	+0.4	**-8.75**	-8.64	+1.3
03/21/2016 $RbNO_3$	**164.2**	166.0	+1.1	**-26.6**	-31.5	-18.4
	285	286.0	+0.4	**-8.75**	-8.68	+0.8
03/29/2016 Ag_2SO_4	**426.4**	427.2	+0.2	**-51.9**	-51.5	+0.8
04/06/2016 $RbNO_3$	**164.2**	165.9	+1.0	**-26.6**	-30.5	-14.7
	285	285.9	+0.3	**-8.75**	-7.90	+9.7
04/12/2016 KNO_3	**128.7**	130.7	+2.3	**-50.0**	-60.8	-21.6

Disposable wooden dowels were used to convey 10-25 mg of sample into the Pt-Rh DSC pan. Samples usually consisted of 1-2 white kernels and varying amounts of the yellow-brown paste. The total time of exposure to the dry (%RH ≤0.6% at 25 – 32°C) glovebox atmosphere between the opening of the vial and the weighing of each sample was 2-4 minutes. Before each run, samples were exposed to the DSC atmosphere, a mixture of air and argon gas, for ~10-20 minutes before the initiation of analysis. This invariably caused the sample weight to decrease by evaporation prior to the initiation of the STA-MS run. For the acid-spiked and Pu-Am-spiked samples, the mass loss was significant. In these cases, the final sample mass entered into the Proteus software was that recorded immediately prior to the initiation of the run. The weight change via evaporation had no effect on the results, and both weights were recorded in the lab notebook (Appendix 7). The Ar content of the furnace atmosphere consistently varies from ~15-20% Ar and 5% O_2 (balance N_2) to 50-65% Ar and 1.5-3.5% O_2 from the beginning of the run to the end.

Figure 1: DSC data from SFWB8-15-1 with no additives. Heavy black lines represent averaged data from all three runs.



[#] Instrument	File	Date	Identity	Sample	M...	S..	Range	Atmosphere	Corr.
[1] STA 409P...	SFWB8-15-1 030716C.ngb-dsv	2016-0...	SFWB8-15-1 030...	SFWB8-15-1 030...	31...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[2] STA 409P...	SFWB8-15-1 030716B.ngb-dsv	2016-0...	SFWB8-15-1 030...	SFWB8-15-1 030...	7.98	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[3] STA 409P...	SFWB8-15-1 030716A.ngb-dsv	2016-0...	SFWB8-15-1 030...	SFWB8-15-1 030...	38...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[4] STA 409P...	average_SFWB8-15-1 030716B71_SFWB8-15-1 030716C...	2016-0...	SFWB8-15-1 030...	SFWB8-15-1 030...	26...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	---
[5] STA 409P...	average_SFWB8-15-1 030716A41_SFWB8-15-1 030716C...	2016-0...	SFWB8-15-1 030...	SFWB8-15-1 030...	26...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	---

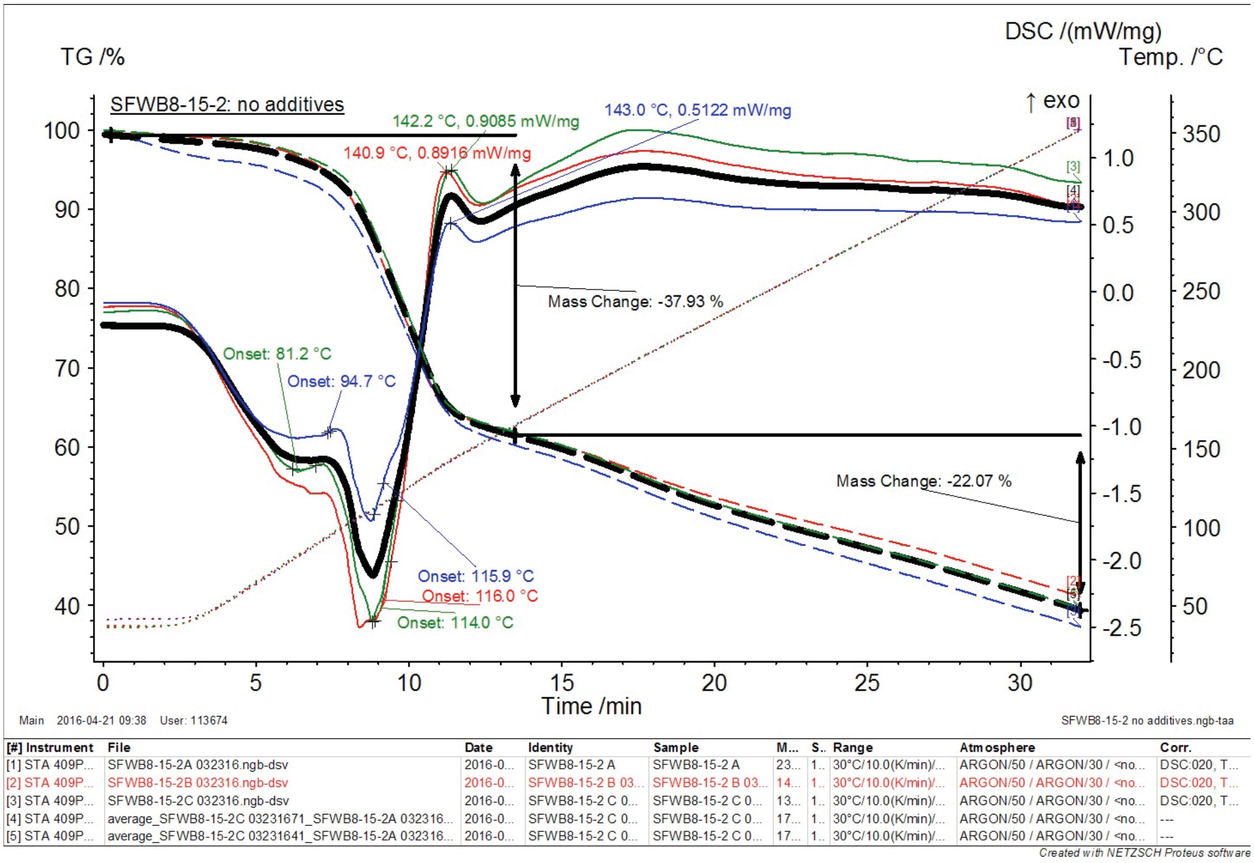
Created with NETZSCH Proteus software

Onset temperatures were obtained using the ‘onset’ capability in Proteus. Enthalpy values are approximate, and were obtained by choosing a reasonable background value on either side of the peak and integrating the peak area in Proteus. Mass changes were evaluated by using the first derivative of the mass change curve. For all DSC plots, exothermic peaks point upwards.

The STA-MS is coupled to a Pfeiffer ThermoStar quadrupole MS (GSD 301T) via a 1m long heated (200°C) silica capillary transfer line. Mass spectrometer data were acquired and processed using the Pfeiffer ThermoStar Suite (Quadstar 32-Bit, v7.03). The mass spectrometer was operated in peak hopping mode (MID), using electron ionization (EI, 70 eV). The RF polarity was normal, the ionization filament current was 1.0 mA, and the multiplier (SEM - channeltron) voltage was 1200 V during all phases of analysis. Analyzer pressure immediately prior to analysis was 9.5 $\times 10^{-7}$ to 1.2 $\times 10^{-6}$ mbar. Gas species analyzed for this study are summarized in Table 5.

The entire apparatus (STA and MS) is located inside a dry air glovebox fitted with hard-plumbed, filtered gas inlets and sealed electrical and telemetric feedthroughs. During analysis, ambient glovebox conditions were: temperature (T) = 25 to 32 °C, relative humidity (%RH) = 0.4 - 0.6%. Pre-run baseline values varied from approximately -6 to -4 μV. All samples were heated to 350 °C at a rate of 10 °C per minute.

Figure 2: DSC plot for SFWB8-15-2 with no additives. Heavy black lines represent averaged data of all 3 runs.



[#] Instrument	File	Date	Identity	Sample	M...	S..	Range	Atmosphere	Corr.
[1] STA 409P...	SFWB8-15-2A 032316.ngb-dsv	2016-0...	SFWB8-15-2 A	SFWB8-15-2 A	23...	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[2] STA 409P...	SFWB8-15-2B 032316.ngb-dsv	2016-0...	SFWB8-15-2 B 03...	SFWB8-15-2 B 03...	14...	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[3] STA 409P...	SFWB8-15-2C 032316.ngb-dsv	2016-0...	SFWB8-15-2 C 0...	SFWB8-15-2 C 0...	13...	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[4] STA 409P...	average_SFWB8-15-2C 03231671_SFWB8-15-2A 032316...	2016-0...	SFWB8-15-2 C 0...	SFWB8-15-2 C 0...	17...	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	---
[5] STA 409P...	average_SFWB8-15-2C 03231641_SFWB8-15-2A 032316...	2016-0...	SFWB8-15-2 C 0...	SFWB8-15-2 C 0...	17...	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	---

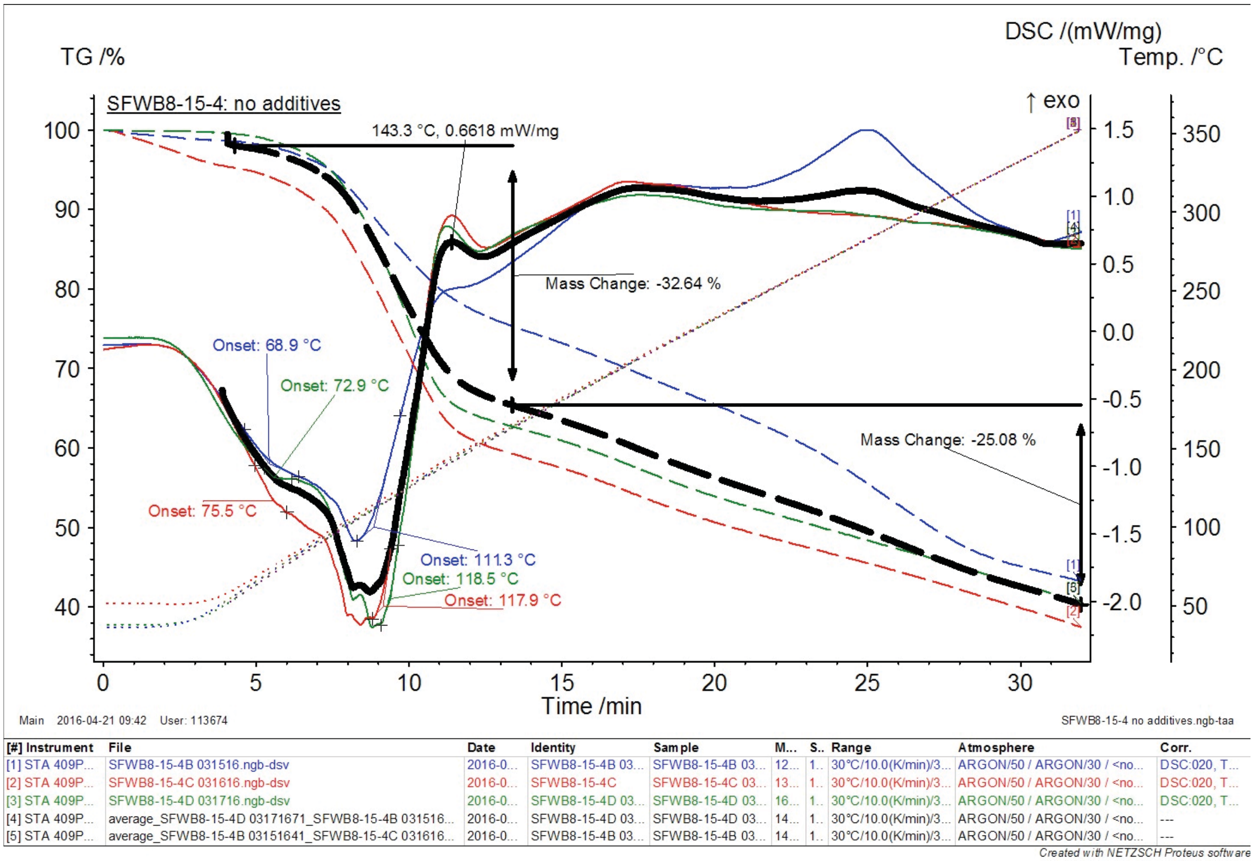
The STA was calibrated using a standard reference material (SRM) set provided by Netzsch (6.223.5-91.2), which consists of certified materials listed in Table 4 and in Appendix 5. These materials were used to construct calibration curves for the measurement of temperature and enthalpy using the DSC measurement head. Measured and calculated sensitivity values, the ratio of the reference enthalpy values to those measured in our apparatus, for each standard are also listed in Table 4. Several samples having known transition temperatures and enthalpy values (typically

KNO_3) were run during the course of RNS surrogate analysis (Table 6) to evaluate the precision and accuracy of the onset temperatures and ΔH measurements relative to the certified values. Onset temperatures vary from the certified values by <3.0%, while the enthalpy measurements (obtained by integrating the peak area using Netzsch Proteus software) vary from the certified values by ~+10% to ~-25%. Thus, we consider the enthalpy values derived from DSC curve peak integrations to be semi-quantitative.

The sample outgas is entrained in a ~80 mL/min ultra-high-purity (UHP) Ar flow. Although the TGA furnace and gas inlets were evacuated and flushed with UHP Ar prior to sample loading, some air was admitted to the sample chamber when it was opened to load the sample. At the beginning of each analysis, the Ar content of the headspace was 10 to 30%, with the balance as dry air. During analysis, Ar content steadily increased to 40-80%.

A single baseline curve run on March 8, 2016 (WIPP350CBaseline.030816.ngb-bsv) was utilized throughout this study to correct the STA results for the effects of thermal buoyancy. During an analysis on March 15, 2016, a small amount of the sample foam was deposited on the top of the DSC platform. The apparatus was subjected to overnight cleaning and bakeout. Results from calibration check samples run before and after this incident indicates that subsequent STA measurements were not changed by this event.

Figure 3: DSC data from SFWB8-15-4 with no additives. Heavy black lines represent averages of all three runs.



[#] Instrument	File	Date	Identity	Sample	M...	S..	Range	Atmosphere	Corr.
[1] STA 409P...	SFWB8-15-4B 031516.ngb-dsv	2016-0...	SFWB8-15-4B 03...	SFWB8-15-4B 03...	12...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[2] STA 409P...	SFWB8-15-4C 031616.ngb-dsv	2016-0...	SFWB8-15-4C	SFWB8-15-4C 03...	13...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[3] STA 409P...	SFWB8-15-4D 031716.ngb-dsv	2016-0...	SFWB8-15-4D 03...	SFWB8-15-4D 03...	16...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[4] STA 409P...	average_SFWB8-15-4D 03171671_SFWB8-15-4B 031516...	2016-0...	SFWB8-15-4D 03...	SFWB8-15-4D 03...	14...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	---
[5] STA 409P...	average_SFWB8-15-4B 03151641_SFWB8-15-4C 031616...	2016-0...	SFWB8-15-4B 03...	SFWB8-15-4B 03...	14...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	---

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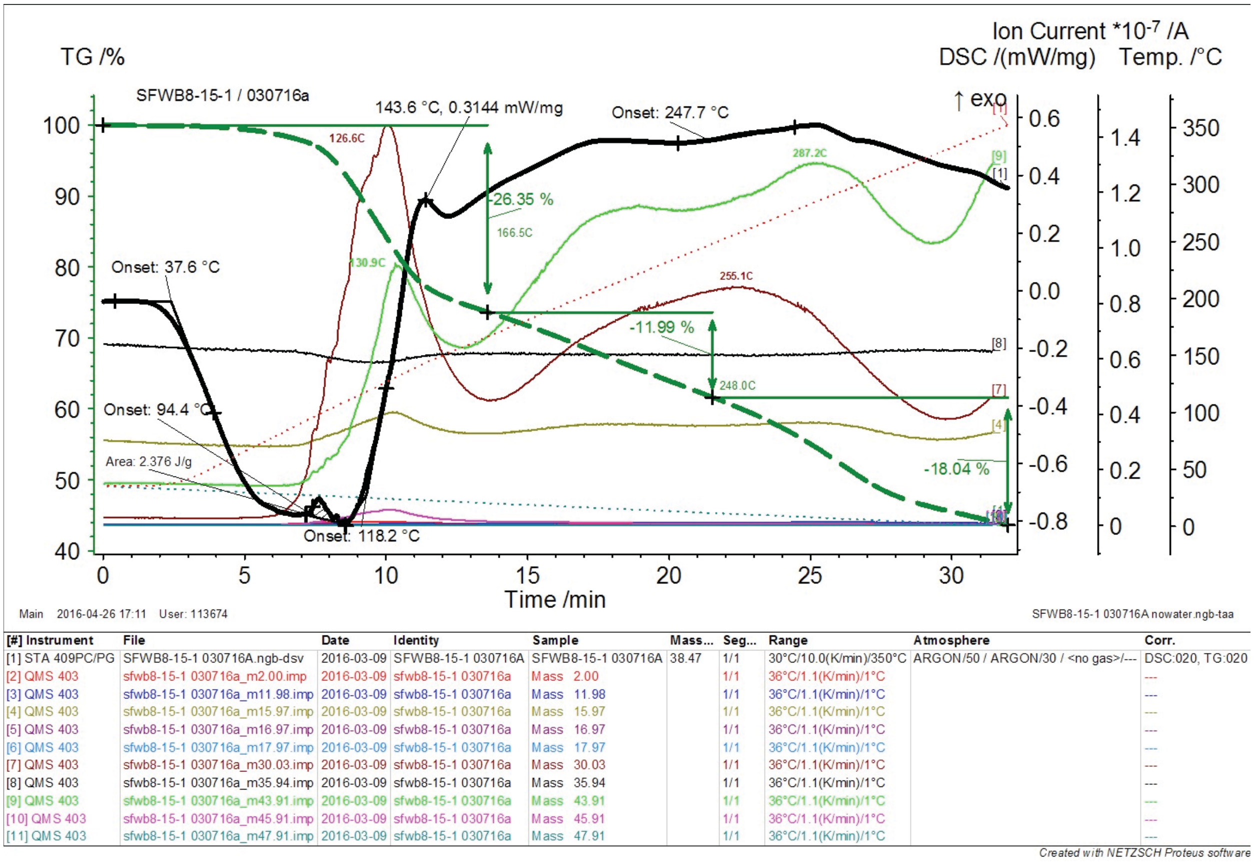
The physical appearance of the sample residue after each run indicated that considerable foaming and swelling (via pore formation) had occurred during each run. The total estimated amount of volume swelling varied from sample to sample, but was typically between ~5 and 10X the original sample volume. During each run, the foaming surrogate lifted the crucible lid off the top of the Pt-Rh pan. The extruded mass was frothy, friable, and light- to dark brown in color. Two of the 27 total runs had to be repeated because the foaming of the surrogate resulted in contact between the sample and the furnace wall, thus rendering the weight measurement invalid. Both runs were successfully repeated using smaller samples. Run sheets and data from both failed runs are included in Appendix 6.

Figure 4: Offgas data from SFWB8-15-1 (no additives) minus H_2O, showing typical trends in NO_x and CO_2 concentrations during heating. The patterns seen in this sample are typical of those seen in all un-spiked RNS surrogates.



[#] Instrument	File	Date	Identity	Sample	Mass...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 030716A.ngb-dsv	2016-03-09	SFWB8-15-1 030716A	SFWB8-15-1 030716A	38.47	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 030716a_m2.00.imp	2016-03-09	sfwb8-15-1 030716a	Mass 2.00		1/1	36°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 030716a_m11.98.imp	2016-03-09	sfwb8-15-1 030716a	Mass 11.98		1/1	36°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 030716a_m15.97.imp	2016-03-09	sfwb8-15-1 030716a	Mass 15.97		1/1	36°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 030716a_m16.97.imp	2016-03-09	sfwb8-15-1 030716a	Mass 16.97		1/1	36°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 030716a_m17.97.imp	2016-03-09	sfwb8-15-1 030716a	Mass 17.97		1/1	36°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 030716a_m30.03.imp	2016-03-09	sfwb8-15-1 030716a	Mass 30.03		1/1	36°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 030716a_m35.94.imp	2016-03-09	sfwb8-15-1 030716a	Mass 35.94		1/1	36°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 030716a_m43.91.imp	2016-03-09	sfwb8-15-1 030716a	Mass 43.91		1/1	36°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 030716a_m45.91.imp	2016-03-09	sfwb8-15-1 030716a	Mass 45.91		1/1	36°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 030716a_m47.91.imp	2016-03-09	sfwb8-15-1 030716a	Mass 47.91		1/1	36°C/1.1(K/min)/1°C		---

5.0 Results

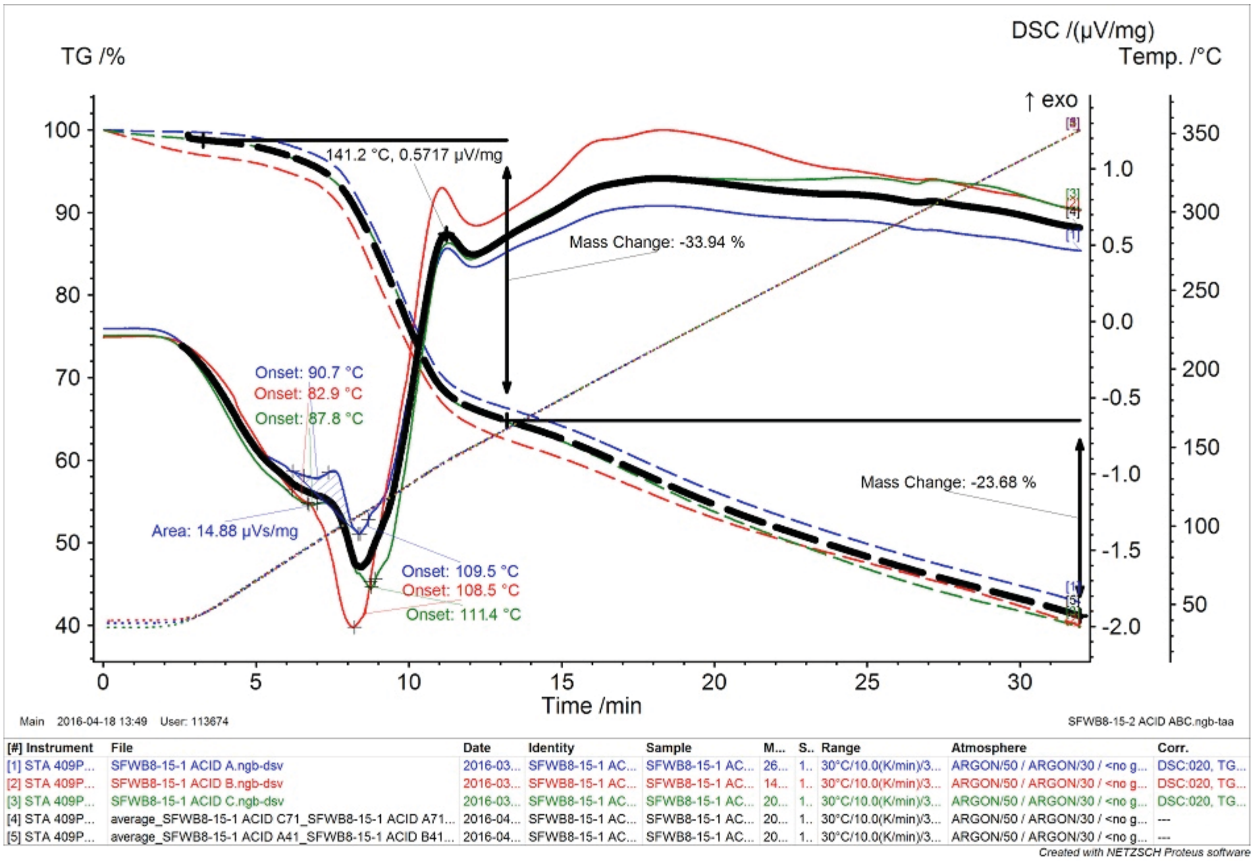
The thermal behavior of all surrogate samples in this study is dominated by three basic phenomena: 1) an endothermic dehydration reaction which onsets between ~38 and 50 °C, 2) an exothermic reaction which onsets between 108 and 123 °C related to the rapid gas release, foaming and expansion of the sample, and 3) steady-state, and slightly exothermic, combustion of the foamed sample above ~150 °C. Stage 1 is dominated by the release of copious amounts of H_2O and mass losses between 35 and 45%. Stage 2 is marked by a sudden increase in the NOx and CO_2

content of the offgas as H_2O begins to tail off. The sustained, near constant, release of small amounts of CO_2, and lesser amounts of NO_x and H_2O, is typical of Stage 3.

5.1 RNS Surrogates with no Additives

The results of the DSC-TGA analyses of the RNS surrogates run as-received are shown in Figures 1-3. Dehydration onsets between 38 and 43 °C and is followed by a change in slope, or a small exotherm, which onsets between ~69 and 97 °C. Endothermic dehydration and denitrification continues until foaming onsets between ~108 and 116 °C. The cessation of foaming is taken as the maximum point of the foaming exotherm, shown on Figures 1-3. Most of the gas release is complete below this point (135.4 - 153.8 °C), and the sample maintains quasi-equilibrium to the end of the run at 350 °C. Several of the samples exhibit a broad exotherm between 200 °C and 300 °C. Gas data indicate that this is related to, and dominated by, CO_2 emission (though NO_x and some H_2O may also be emitted). Thus, the shallow exotherms seen in this high-temperature region may be indicative of partial combustion of the extruded sample closest to the furnace.

Figure 5: DSC results for SFWB8-15-1 plus 10M HNO_3 + 0.3M HF. Heavy black lines represent averaged data.



[#] Instrument	File	Date	Identity	Sample	M...	S..	Range	Atmosphere	Corr.
[1] STA 409P...	SFWB8-15-1 ACID A.ngb-dsv	2016-03...	SFWB8-15-1 AC...	SFWB8-15-1 AC...	26...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no g...	DSC:020, TG...
[2] STA 409P...	SFWB8-15-1 ACID B.ngb-dsv	2016-03...	SFWB8-15-1 AC...	SFWB8-15-1 AC...	14...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no g...	DSC:020, TG...
[3] STA 409P...	SFWB8-15-1 ACID C.ngb-dsv	2016-03...	SFWB8-15-1 AC...	SFWB8-15-1 AC...	20...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no g...	DSC:020, TG...
[4] STA 409P...	average_SFWB8-15-1 ACID C71_SFWB8-15-1 ACID A71...	2016-04...	SFWB8-15-1 AC...	SFWB8-15-1 AC...	20...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no g...	---
[5] STA 409P...	average_SFWB8-15-1 ACID A41_SFWB8-15-1 ACID B41...	2016-04...	SFWB8-15-1 AC...	SFWB8-15-1 AC...	20...	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no g...	---

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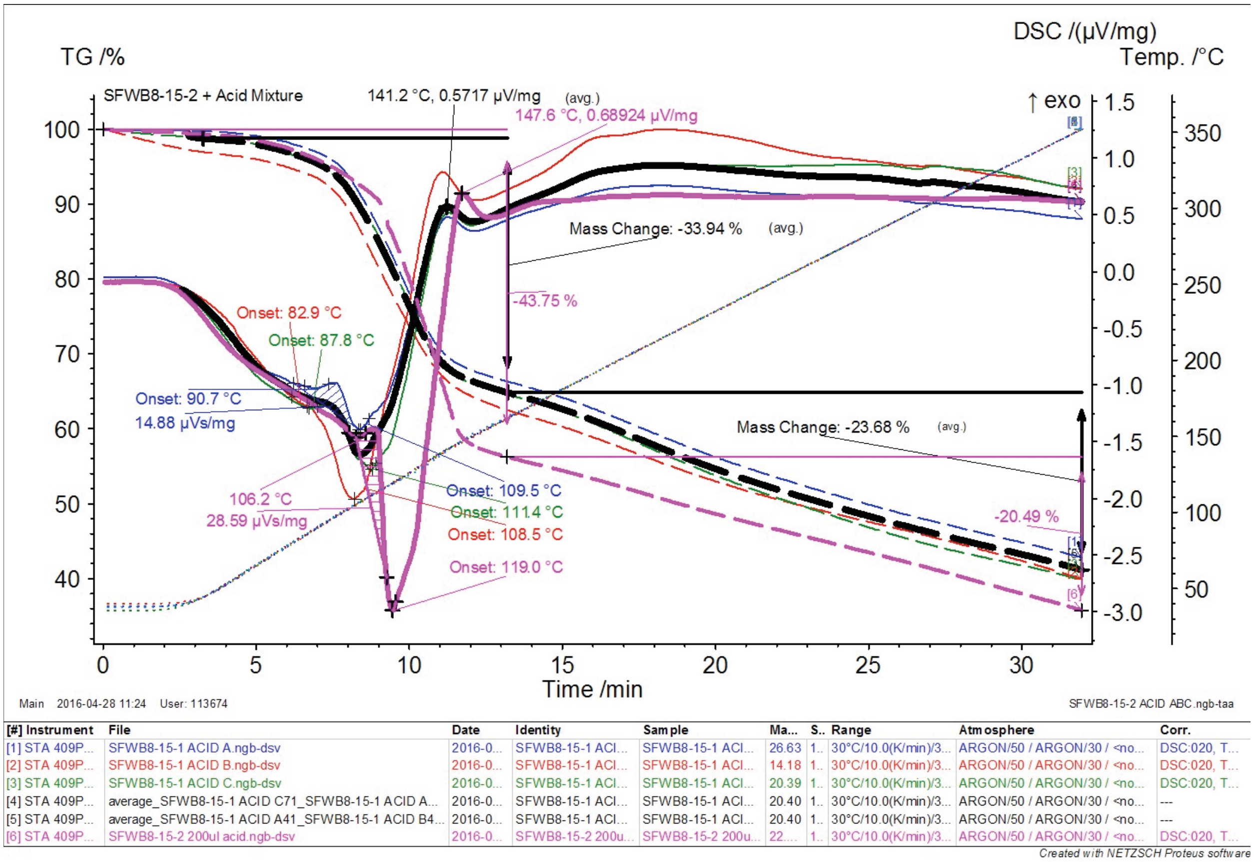
Mass change also follows a predictable pattern. Total mass loss is typically between 50 and 60 wt. % and occurs in two distinct episodes. For the raw surrogates, average mass loss during dehydration / foaming varies from 31 to 38 wt. %, and ceases between 150 and 200 °C. Steady state mass loss via decarbonation (with lesser amounts of dehydration and denitrification) occurs from ~200 °C to the end of the run at 350 °C.

We were able to infer the temperature at which the foaming occurred using the results of the first of two failed runs; SFWB8-15-4A. The mass change curve for this sample suddenly plummeted just before the termination of the steep exothermic part of the DSC curve, at ~140 °C. Amazingly the mass returned to normal at ~170 °C. We surmised that the swelling surrogate had touched the furnace wall, thereby disrupting the balance measurement. As the sample dried, it shrank and lost contact with the furnace wall, thereby restoring the balance measurement. The slightly adhesive nature of the foam may have enabled the capsule lid to remain on top of the swelling mass.

Varying amounts of lead nitrate in the unspiked RNS surrogates has little apparent effect on thermal behavior. Both the onset and cessation of foaming occur at similar temperatures and the mass losses during dehydration / foaming are also similar. Each of the raw surrogate samples displays a small exothermic peak or "shoulder" prior to the onset of foaming between 108 °C and 116 °C. In the 1% and 2% Pb nitrate surrogates (Figures 1-2), the onset of the small exotherm or "shoulder" occurs between 81°C and 97 °C. In the 4% Pb nitrate surrogate (Figure 3), there are no peaks in this region, though the enthalpy curve undergoes a noticeable slope change beginning between 69-75 °C.

Figure 6: DSC results for SFWB8-15-2 plus 10M HNO_3 + 0.3M HF. Heavy black lines represent averaged data from the three 100 μL runs. Results from the 200 μL run are shown by the magenta line.



[#] Instrument	File	Date	Identity	Sample	Ma...	S..	Range	Atmosphere	Corr.
[1] STA 409P...	SFWB8-15-1 ACID A.ngb-dsv	2016-0...	SFWB8-15-1 ACI...	SFWB8-15-1 ACI...	26.63	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[2] STA 409P...	SFWB8-15-1 ACID B.ngb-dsv	2016-0...	SFWB8-15-1 ACI...	SFWB8-15-1 ACI...	14.18	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[3] STA 409P...	SFWB8-15-1 ACID C.ngb-dsv	2016-0...	SFWB8-15-1 ACI...	SFWB8-15-1 ACI...	20.39	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[4] STA 409P...	average_SFWB8-15-1 ACID C71_SFWB8-15-1 ACID A...	2016-0...	SFWB8-15-1 ACI...	SFWB8-15-1 ACI...	20.40	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	---
[5] STA 409P...	average_SFWB8-15-1 ACID A41_SFWB8-15-1 ACID B4...	2016-0...	SFWB8-15-1 ACI...	SFWB8-15-1 ACI...	20.40	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	---
[6] STA 409P...	SFWB8-15-2 200ul acid.ngb-dsv	2016-0...	SFWB8-15-2 200u...	SFWB8-15-2 200u...	22....	1..	30°C/10.0(K/min)/3...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...

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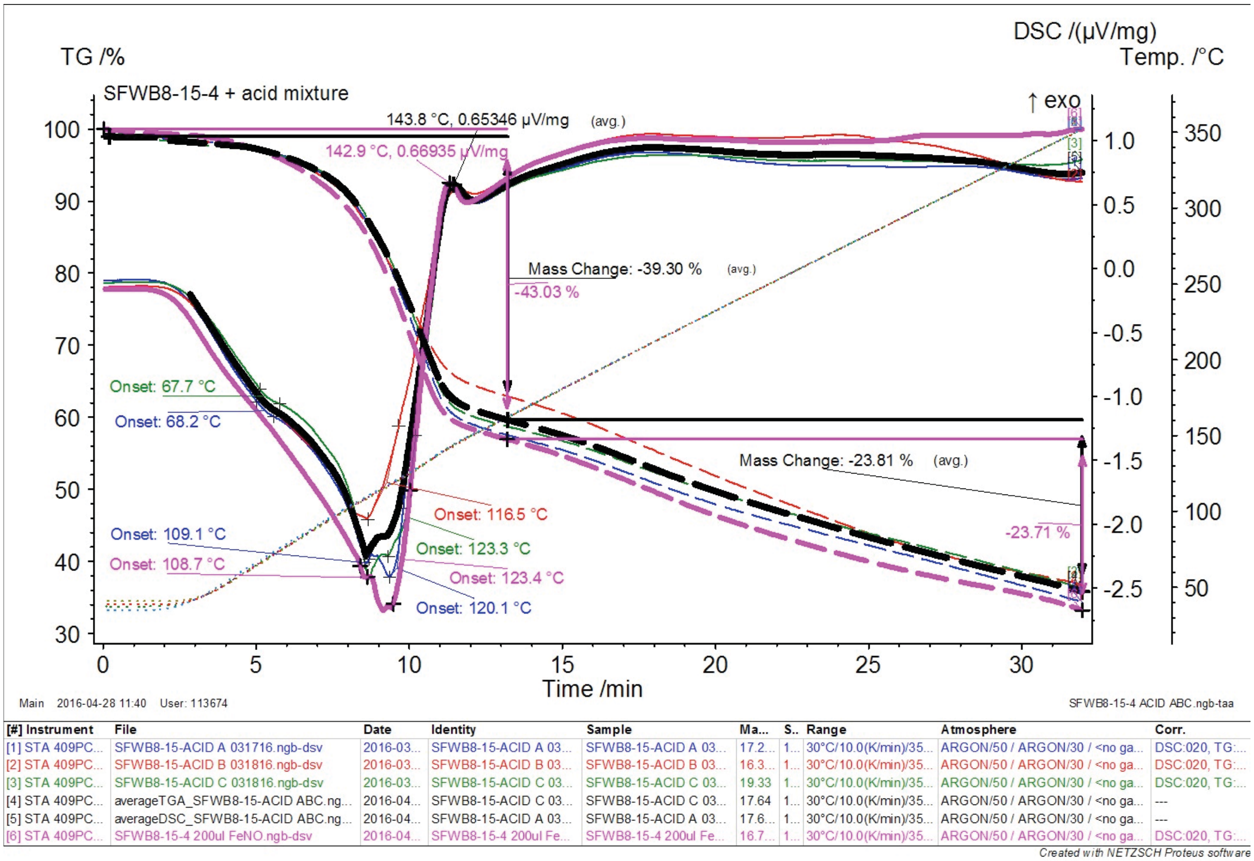
5.1.1 Offgas Data

Offgas from the as-received RNS surrogates shows little variation with changing Pb content. The emission of H_2O, starting immediately after the initiation of the analysis, dominates all other gas emissions, in terms of total ion current, by roughly two orders of magnitude. Maximum H_2O occurs between 114.4 °C and 135.4 °C, and shows no relationship to surrogate chemistry. Similarly, slight variations in the run-to-run maxima of NO_x (123.1-141.2 °C) and CO_2 (125.4-153.5 °C) also have no discernable relationship to Pb content.

Removal of the water peaks from the offgas spectra reveals the relationships between NOx and CO2 emissions and thermally-driven reactions (Figure 4). NO_x emission precedes CO_2 by ~10-20 °C in every case, and seems to coincide with either the change in slope or the onset of small exotherm between ~69 and 97 °C. Both NO_x and CO_2 ion signals rise abruptly during, or slightly before, the onset of foaming and reach peak concentrations in the offgas before foaming ceases.

Figure 7: DSC results for SFWB8-15-4 plus 10M HNO_3 + 0.3M HF. Heavy black lines represent averaged data from the three 100 μL runs. Results from the run with 200 μL Fe^{3+} nitrate solution are shown by the magenta line.



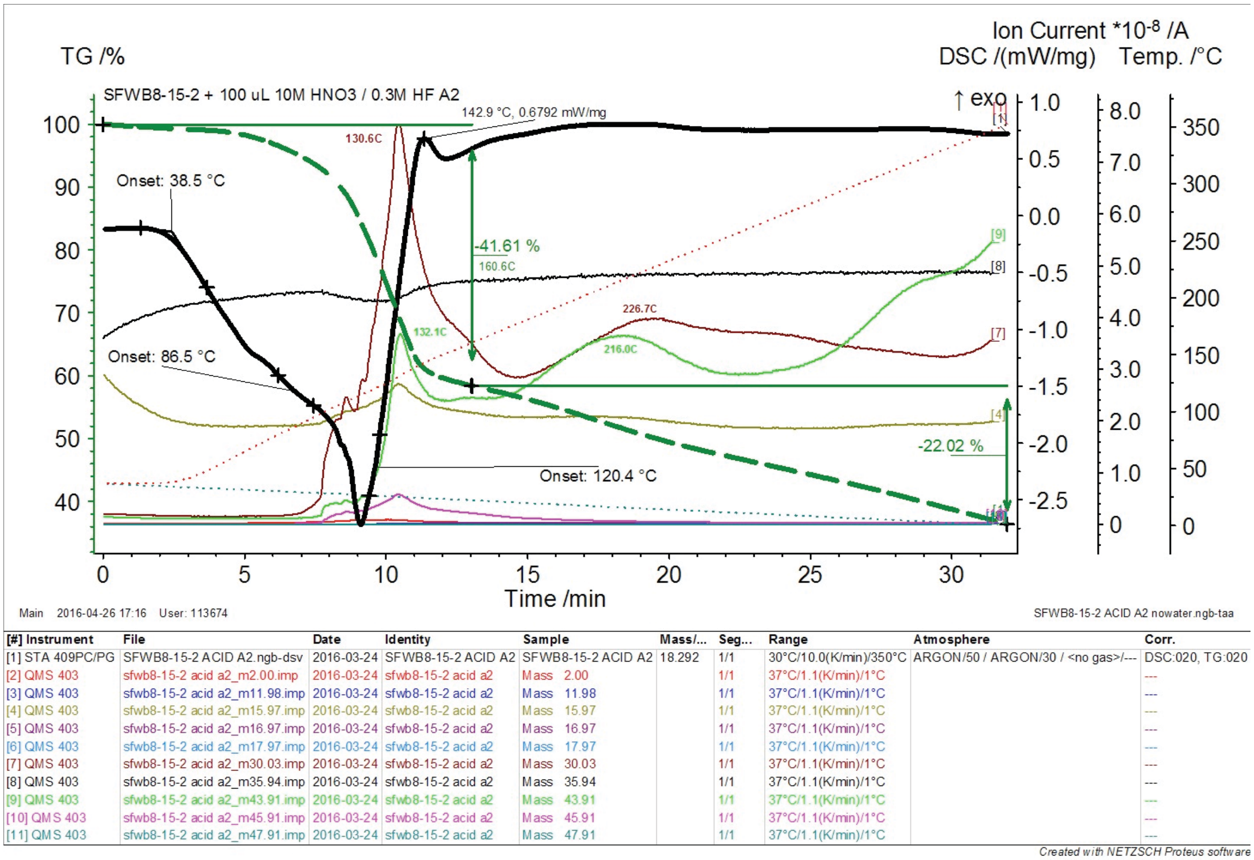
[#] Instrument	File	Date	Identity	Sample	Ma...	S..	Range	Atmosphere	Corr.
[1] STA 409PC...	SFWB8-15-ACID A 031716.ngb-dsv	2016-03...	SFWB8-15-ACID A 03...	SFWB8-15-ACID A 03...	17.2...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no ga...	DSC:020, TG:...
[2] STA 409PC...	SFWB8-15-ACID B 031816.ngb-dsv	2016-03...	SFWB8-15-ACID B 03...	SFWB8-15-ACID B 03...	16.3...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no ga...	DSC:020, TG:...
[3] STA 409PC...	SFWB8-15-ACID C 031816.ngb-dsv	2016-03...	SFWB8-15-ACID C 03...	SFWB8-15-ACID C 03...	19.33	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no ga...	DSC:020, TG:...
[4] STA 409PC...	averageTGA_SFWB8-15-ACID ABC.ng...	2016-04...	SFWB8-15-ACID C 03...	SFWB8-15-ACID C 03...	17.64	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no ga...	---
[5] STA 409PC...	averageDSC_SFWB8-15-ACID ABC.ng...	2016-04...	SFWB8-15-ACID A 03...	SFWB8-15-ACID A 03...	17.6...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no ga...	---
[6] STA 409PC...	SFWB8-15-4 200ul FeNO.ngb-dsv	2016-04...	SFWB8-15-4 200ul Fe...	SFWB8-15-4 200ul Fe...	16.7...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no ga...	DSC:020, TG:...

5.2 Surrogates plus 100-250 μL of 10M HNO_3 + 0.3M HF

Small volumes of reagent-grade acid (10M HNO_3 + 0.3M HF) were added to a second, separate, set of the SFWB8-15-1, -2, and -4 surrogates in order to examine the differences in reactivity that could result from the addition of the acid matrix used to dissolve the Pu-Am spike. Acid was added directly to the surrogate using a dropper or pipette and mixed by hand, as described in Section 4.0, of this report.

The DSC and TGA curves of the acid-spiked surrogates (Figures 5-7) are nearly identical to those of the raw surrogates with no acid added. Mass losses and onset temperatures are similar and show no consistent departures from the trends seen during the thermal analysis of the unspiked RNS surrogates. The dehydration reaction onset is between ~38-42 °C and may be followed by either a change in slope (onset T = 65.6-86.5 °C), or a small exotherm (onset T = 85.5-116.5 °C). In most cases, the small exotherm occurs before the onset of foaming. The slope of the foaming reaction is noticeably steeper in the acid-spiked samples than in the surrogates with no additives. In two runs (SFWB8-15-4 ACID C and SFWB8-15-2 ACID B) a small exotherm occurs immediately after the onset of foaming. In SFWB8-15-2 ACID B, the exotherm is sharp, distinct, and its peak is approximately coeval with a transient maximum in H_2O vapor emission at 111.7 °C. Foaming in the acid-spiked RNS surrogates onsets between ~108 and 124 °C and is complete between ~138 and 148 °C (Figures 5-7). As with the unspiked RNS surrogates, the acid-spiked samples maintain flat DSC profiles (indicative of quasi-equilibrium) above ~150 °C, though several samples exhibit a broad exotherm between 200 °C and 300 °C. Gas data from this region indicate that these shallow high-temperature exotherms are related to, and dominated by, CO_2 emission (though NO_x and some H_2O may also be emitted). It is likely that these features are indicative of partial combustion of the extruded sample closest to the furnace.

Figure 8: STA-MS data from SFWB8-15-2 with 100 μL of the acid mixture added. Water peaks removed.

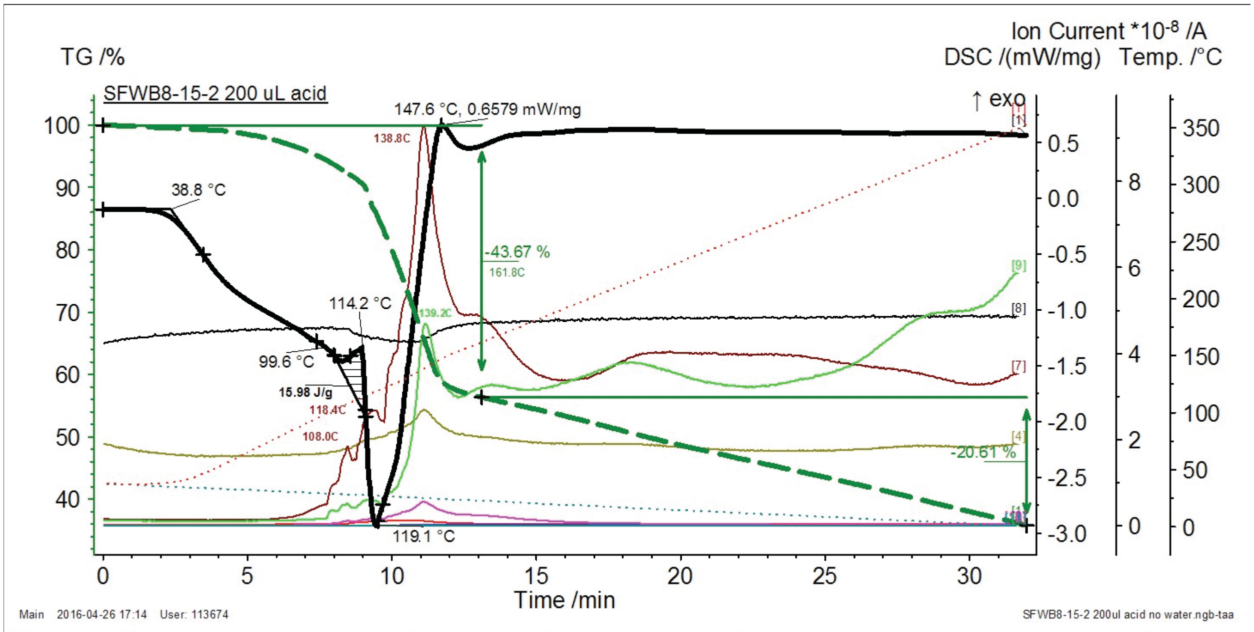


[#] Instrument	File	Date	Identity	Sample	Mass/...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 ACID A2.ngb-dsv	2016-03-24	SFWB8-15-2 ACID A2	SFWB8-15-2 ACID A2	18.292	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 acid a2_m2.00.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 2.00		1/1	37°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 acid a2_m11.98.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 11.98		1/1	37°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 acid a2_m15.97.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 15.97		1/1	37°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 acid a2_m16.97.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 16.97		1/1	37°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 acid a2_m17.97.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 17.97		1/1	37°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 acid a2_m30.03.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 30.03		1/1	37°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 acid a2_m35.94.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 35.94		1/1	37°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 acid a2_m43.91.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 43.91		1/1	37°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 acid a2_m45.91.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 45.91		1/1	37°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 acid a2_m47.91.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 47.91		1/1	37°C/1.1(K/min)/1°C		---

Mass change also follows a pattern similar to that seen for the unspiked RNS surrogate. Average total mass loss varies from ~58 to ~63 wt. % and occurs in two distinct episodes: ~34 to 39 wt. % is lost below ~160 °C, and ~24% is lost during the 'steady state' phase above ~160 °C. Average mass losses are slightly greater for the acid-spiked samples due to the additional dehydration spike itself. Slight variations in the slope of the mass change curve above 160 °C correspond to exotherms related to combustion and decarbonation.

The thermal behavior and reactivity of the acid-spiked samples is broadly similar to that of the unspiked surrogates. However, prior to the onset of foaming, we noted subtle differences in the behavior of the acid-spiked surrogates. Relative to the other RNS surrogates, SFWB8-15-4 (Figure 7) appears to be relatively unreactive prior to the onset of foaming. Results from a single sample of SFWB8-15-4 to which 200 μL of a Fe^{3+} nitrate solution was added (Figure 7) also showed little change relative to the other acid-spiked runs. Samples of SFWB8-15-2 spiked with 100 μL of the acid mixture showed relatively little reactivity between the onset of dehydration and the onset of foaming (Figure 6). When spiked with 200 μL of acid, SFWB8-15-2 showed slightly greater reactivity in the same region (Figure 6). Mass loss below ~160 °C was also slightly greater, and the onset and cessation of foaming were pushed out to slightly higher temperatures.

Figure 9: STA-MS results from SFWB8-15-2 with 200 μL of acid added. Note the multiple NO_x peaks and the sharp rise of NO_x roughly coeval with the onset of the small exotherm at 99.6°C.



[#] Instrument	File	Date	Identity	Sample	Mas...	S...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 200ul acid.ngb-dsv	2016-04-12	SFWB8-15-2 200ul acid	SFWB8-15-2 200ul acid	22.848	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 200ul acid_m2.00.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 2.00		1/1	38°C/1.2(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 200ul acid_m11.98.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 11.98		1/1	38°C/1.2(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 200ul acid_m15.97.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 15.97		1/1	38°C/1.2(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 200ul acid_m16.97.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 16.97		1/1	38°C/1.2(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 200ul acid_m17.97.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 17.97		1/1	38°C/1.2(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 200ul acid_m30.03.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 30.03		1/1	38°C/1.2(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 200ul acid_m35.94.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 35.94		1/1	38°C/1.2(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 200ul acid_m43.91.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 43.91		1/1	38°C/1.2(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 200ul acid_m45.91.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 45.91		1/1	38°C/1.2(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 200ul acid_m47.91.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 47.91		1/1	38°C/1.2(K/min)/1°C		---

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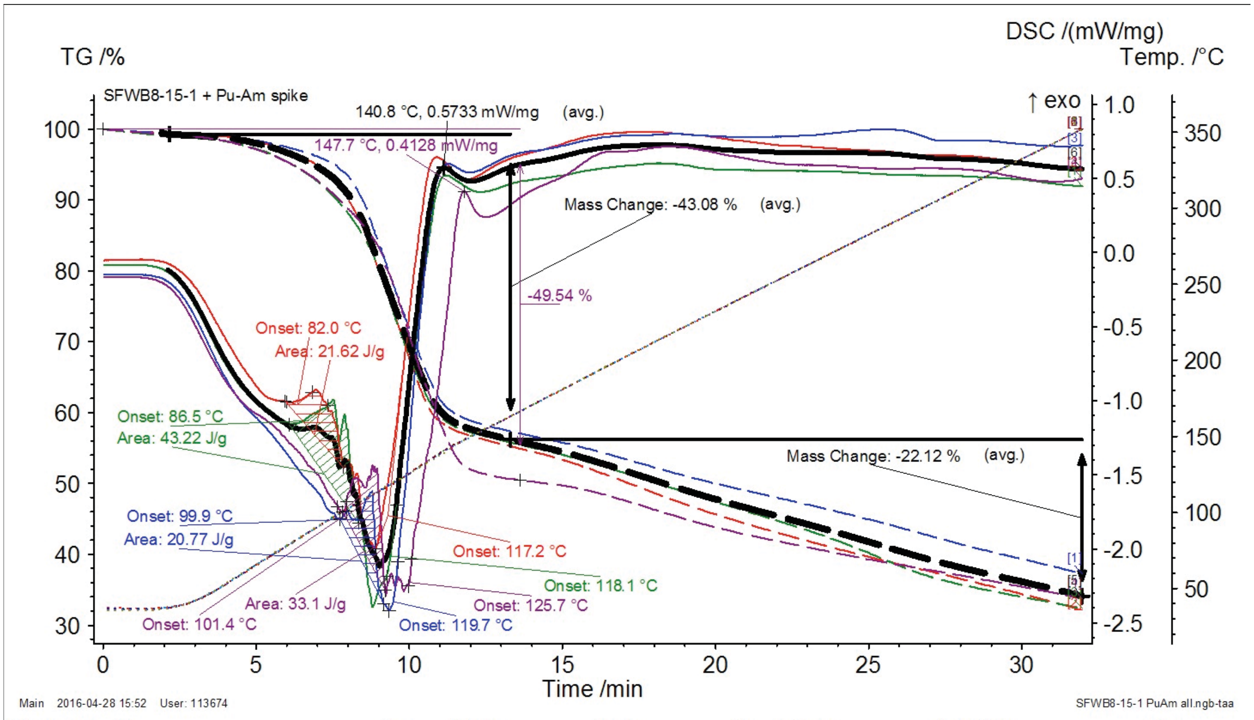
5.2.1 Offgas Data

The emission of H_2O dominates those of all other gas species, in terms of total ion current, by roughly two orders of magnitude. Maximum H_2O occurs over a similar temperature range (105.9 - 132.5 °C) as in the samples that were not spiked with acid. The same is true for the overall maxima of NO_x (117.5 - 138.8 °C) and CO_2 (126.0 – 139.2 °C) emissions. However, we observed smaller, auxiliary peaks in NO_x emission at somewhat lower temperatures (109.0 – 119.4 °C) in four of the eleven acid-spiked runs (Figures 8-9).

Removal of the water peaks from the offgas spectra reveals the relationships between NO_x and CO_2 emissions and thermally-driven reactions. The general patterns of NOx and CO_2 offgas are similar to those observed in the RNS surrogates without the acid spike added. The NO_x emission precedes CO_2 by ~10 °C in every case, and seems to coincide with either the change in slope (onset T = 65.6-86.5 °C), or the onset of small exotherm or a small exotherm (onset T = 85.5-116.5 °C) prior to the onset of foaming. In this region, qualitatively, the rise of the NOx signal appears to be somewhat sharper in the acid-spiked surrogates compared to the surrogates run without any additions. Both NO_x and CO_2 ion signals rise abruptly during, or slightly before, the onset of foaming and reach peak concentrations in the offgas before foaming ceases.

Figure 10: DSC-TGA plot from SFWB-8-15-1 plus 200 μL of the Pu-Am spike. Heavy black lines represent the average of the three 200 μL runs. The violet line is from a single run using 300 μL of the spike.



[#] Instrument	File	Date	Identity	Sample	Mas...	S..	Range	Atmosphere	Corr.
[1] STA 409PC/...	SFWB8-15-1 PuAm 1.ngb-dsv	2016-04...	SFWB8-15-1 PuAm 1	SFWB8-15-1 PuAm 1	22.3...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[2] STA 409PC/...	SFWB8-15-1 PuAm 2.ngb-dsv	2016-04...	SFWB8-15-1 PuAm 2	SFWB8-15-1 PuAm 2	19.9...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[3] STA 409PC/...	SFWB8-15-1 PuAm 3.ngb-dsv	2016-04...	SFWB8-15-1 PuAm3	SFWB8-15-1 PuAm3	17.3...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[4] STA 409PC/...	SFWB8-15-1 PuAm 400 uL.ngb-dsv	2016-04...	SFWB8-15-1 400 Pu...	SFWB8-15-1 400 Pu...	20.23	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[5] STA 409PC/...	averageTGA_SFWB8-15-1 PuAm ABC.ngb...	2016-04...	SFWB8-15-1 PuAm3	SFWB8-15-1 PuAm3	19.8...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	---
[6] STA 409PC/...	averageDSC_SFWB8-15-1 PuAm ABC.ngb...	2016-04...	SFWB8-15-1 PuAm 1	SFWB8-15-1 PuAm 1	19.8...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	---

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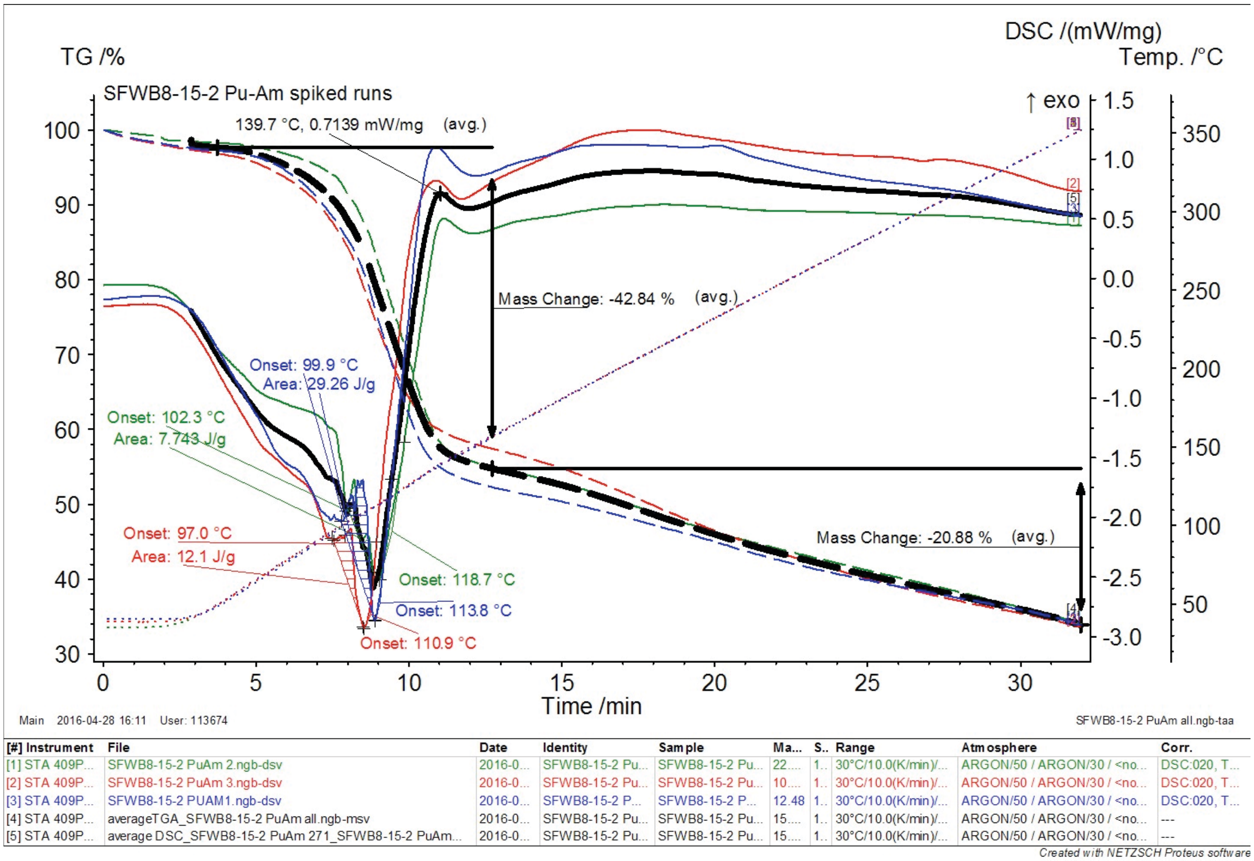
5.3 Surrogates plus 200 μL of Pu-Am spike ('SNM-spiked' samples)

The SNM-spiked surrogates were prepared in an identical manner to the acid-spiked samples. Details on the spike and sample preparation can be found in Section 4.0 of this report. Post-run, the SNM-spiked samples contained faint, but noticeable, dark horizons; presumably Pu and Am oxides formed during the TGA-DSC run. After the 200 μL trials were complete, a single TGA-DSC experiment was conducted on a surrogate spiked with 300 μL of the Pu-Am solution.

The general configuration of both the DSC and mass change curves for the SNM-spiked samples (Figures 10-12) was very similar to that observed in both the unspiked and acid-spiked surrogates that contained no SNM: endothermic dehydration followed by exothermic foaming (onset T = 111.2 – 119.9 °C) which terminates between 136.8 and 142.5 °C, followed by a near steady-state ± a minor combustion reaction which emit mostly CO_2. As with the acid-spike samples, the slope of the foaming curve is steeper than in the surrogates with no additives. Between ~80 and 120 °C, and before the onset of foaming, the DSC curves of all runs of all three SNM-spiked surrogates consistently showed slope changes and small, but significant, exothermic excursions. Most runs showed multiple small exotherms in this region – typically two – the first onsetting between 82 and 102 °C and the second between 104 and 108 °C. The magnitude of these exothermic reactions is relatively small, between ~6 and 43 J/g, and quite variable between runs. Most of these exotherms have sharp, jagged profiles with multiple small peaks.

Figure 11: DSC-TGA plot from SFWB-8-15-2 plus 200 μL of the Pu-Am spike. Heavy black lines represent the average of the three 200 μL runs.



Main 2016-04-28 16:11 User: 113674

SFWB8-15-2 PuAm all.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Ma...	S..	Range	Atmosphere	Corr.
[1] STA 409P...	SFWB8-15-2 PuAm 2.ngb-dsv	2016-0...	SFWB8-15-2 Pu...	SFWB8-15-2 Pu...	22....	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[2] STA 409P...	SFWB8-15-2 PuAm 3.ngb-dsv	2016-0...	SFWB8-15-2 Pu...	SFWB8-15-2 Pu...	10....	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[3] STA 409P...	SFWB8-15-2 PUAM1.ngb-dsv	2016-0...	SFWB8-15-2 P...	SFWB8-15-2 P...	12.48	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	DSC:020, T...
[4] STA 409P...	averageTGA_SFWB8-15-2 PuAm all.ngb-msv	2016-0...	SFWB8-15-2 Pu...	SFWB8-15-2 Pu...	15....	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	---
[5] STA 409P...	average DSC_SFWB8-15-2 PuAm 271_SFWB8-15-2 PuAm...	2016-0...	SFWB8-15-2 Pu...	SFWB8-15-2 Pu...	15....	1..	30°C/10.0(K/min)/...	ARGON/50 / ARGON/30 / <no...	---

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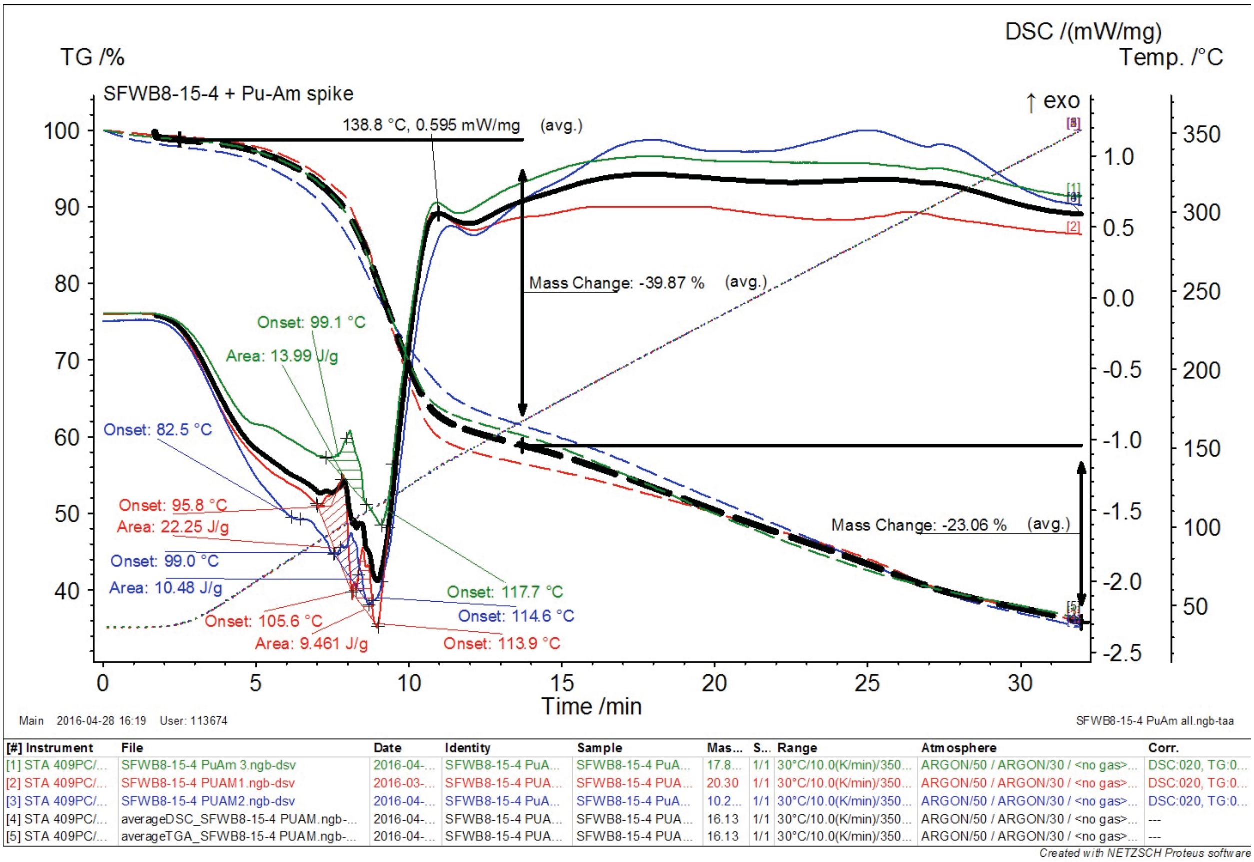
Though some of the unspiked and acid-spiked surrogates showed similar activity in the same temperature region, every single one of the SNM-spiked samples showed some exothermic reactivity superimposed on the dehydration endotherm between ~80 and 120 °C. One surrogate formulation, SFWB8-15-1 consistently showed the most reactivity in this region. Though we were only able to qualitatively estimate the heat flow generated by these reactions, the integrated peak areas of the exotherms in this formulation are were consistently greater than the others (Figure 10). Exotherms generated by reactions in the formulations with higher Pb contents were variable in both their peak height and integrated peak areas (Figures 11-12).

A single sample from SFWB8-15-1 was spiked with 300 mL of the Pu-Am solution (8.31 mg Pu and 4.68 mg Am added to ~1.0 g of surrogate). The STA results from this sample (Figure 10) were broadly similar to the 200 mL runs though both the foaming onset (125.9 °C) and cessation (147.7 °C) occurred at slightly higher temperatures. No significant increase in reactivity in the thermal region between ~80 and 120 °C was observed in the run spiked to higher SNM contents.

Figure 12: DSC-TGA plot from SFWB-8-15-4 plus 200 μL of the Pu-Am spike. Heavy black lines represent the average of the three 200 μL runs.



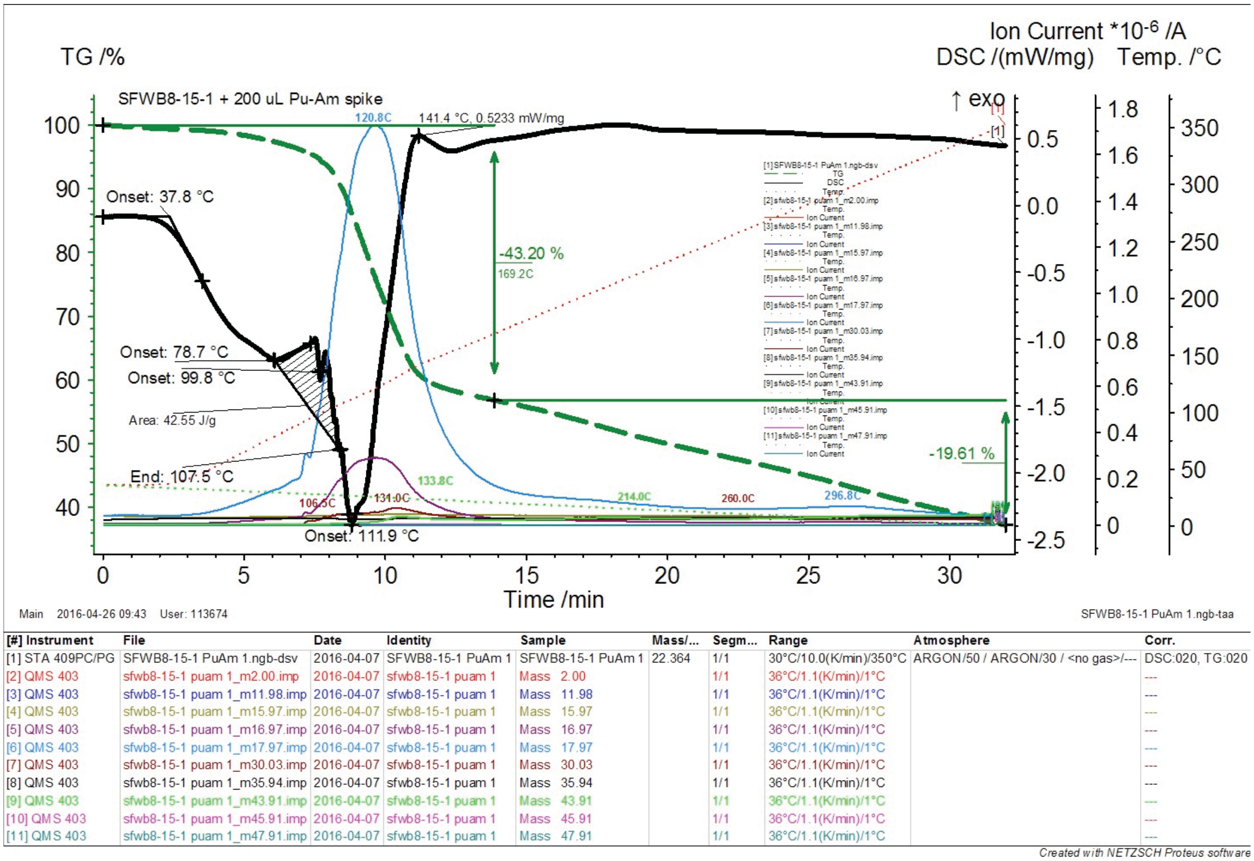
[#] Instrument	File	Date	Identity	Sample	Mas...	S...	Range	Atmosphere	Corr.
[1] STA 409PC/...	SFWB8-15-4 PuAm 3.ngb-dsv	2016-04-...	SFWB8-15-4 PuA...	SFWB8-15-4 PuA...	17.8...	1/1	30°C/10.0(K/min)/350...	ARGON/50 / ARGON/30 / <no gas>...	DSC:020, TG:0...
[2] STA 409PC/...	SFWB8-15-4 PUAM1.ngb-dsv	2016-03-...	SFWB8-15-4 PUA...	SFWB8-15-4 PUA...	20.30	1/1	30°C/10.0(K/min)/350...	ARGON/50 / ARGON/30 / <no gas>...	DSC:020, TG:0...
[3] STA 409PC/...	SFWB8-15-4 PUAM2.ngb-dsv	2016-04-...	SFWB8-15-4 PuA...	SFWB8-15-4 PuA...	10.2...	1/1	30°C/10.0(K/min)/350...	ARGON/50 / ARGON/30 / <no gas>...	DSC:020, TG:0...
[4] STA 409PC/...	averageDSC_SFWB8-15-4 PUAM.ngb-...	2016-04-...	SFWB8-15-4 PUA...	SFWB8-15-4 PUA...	16.13	1/1	30°C/10.0(K/min)/350...	ARGON/50 / ARGON/30 / <no gas>...	---
[5] STA 409PC/...	averageTGA_SFWB8-15-4 PUAM.ngb-...	2016-04-...	SFWB8-15-4 PUA...	SFWB8-15-4 PUA...	16.13	1/1	30°C/10.0(K/min)/350...	ARGON/50 / ARGON/30 / <no gas>...	---

5.3.1 Offgas Data

Similar to the unspiked and acid-spiked surrogates, the H_2O maximum for the Pu-Am spiked surrogate samples occurred between 111.4 and 129.6 °C (Figure 13). Absolute maxima for both CO_2 and NO_x emissions occur in the same temperature ranges for the SNM-spiked samples as they do for the non-SNM samples. Multiple NO_x peaks below the onset of foaming are very common in the SNM-spiked samples, however.

With the water peak removed, significant trends in NO_x and CO_2 emission become more apparent (Figure 14). The behavior of NO_x between ~70 and 115 °C in the Pu-Am spiked samples is markedly different from that seen in the unspiked and acid-spiked surrogate samples. The emission of NO_x emission starts between 65 and 75 °C, as in the other surrogates. The rise in the NO_x concentration of the offgas appears to correspond either to the onset of a slope change in the DSC curve, or to the onset of an exotherm, as in the acid-spiked surrogate samples. The Pu-Am spiked samples are distinguished by multiple, near-vertical, NO_x maxima which correspond to sharp increases in the equally irregular DSC curve (Figure 14) prior to the onset of foaming. The magnitude of the NO_x ion signal may, or may not, correlate to the magnitude of the corresponding offsets in the DSC curve.

Figure 13: DSC and offgas data from SFWB8-15-1 A, showing typical trends in mass loss, enthalpy, and gas emission.



Main 2016-04-26 09:43 User: 113674

SFWB8-15-1 PuAm 1.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 PuAm 1.ngb-dsv	2016-04-07	SFWB8-15-1 PuAm 1	SFWB8-15-1 PuAm 1	22.364	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 puam 1_m2.00.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 2.00		1/1	36°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 puam 1_m11.98.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 11.98		1/1	36°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 puam 1_m15.97.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 15.97		1/1	36°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 puam 1_m16.97.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 16.97		1/1	36°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 puam 1_m17.97.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 17.97		1/1	36°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 puam 1_m30.03.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 30.03		1/1	36°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 puam 1_m35.94.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 35.94		1/1	36°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 puam 1_m43.91.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 43.91		1/1	36°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 puam 1_m45.91.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 45.91		1/1	36°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 puam 1_m47.91.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 47.91		1/1	36°C/1.1(K/min)/1°C		---

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5.4 Thermal Behavior of the Dried Pu-Am spike

We also analyzed the dried residue left after evaporating 200 μL of the spike in a Pt-Rh differential scanning calorimetry (DSC) pan. Phase identification was not performed, though it is likely that the dried residue consisted of a mixture of Pu and Am nitrate hydrates and anhydrous fluorides. After heating to 350 °C, a small amount of black, powdery residue remained, likely Pu and Am oxides. Results of the STA-MS analysis (Figure 15) show a complex succession of multiple mass losses, endotherms and the corresponding H_2O and NO_x gas emission peaks. The DSC curve is dominated by a large dehydration endotherm which onsets at ~40 °C. A pronounced slope change onsets at 64.0 °C and corresponds to the initiation of NO_x emission, which reaches a plateau at ~80°C (122.2 °C max.). This is the first of five distinct NO_x maxima. Higher temperature endotherms onsetting at 231.6 °C and 284.3 °C correspond to distinct NO_x gas emission peaks at 242.2 °C and 292.8 °C. Though most of the mass loss occurs during dehydration (-10.60 wt. %), 3 (possibly 4) discrete de-nitrification events are marked by mass losses between -3.41 and -10.54 wt. %.

The most interesting aspect of the thermal behavior of the dried spike is its low temperature release of NO_x. Like ferric nitrate hydrate, Pu and Am nitrate hydrates would likely dehydrate, de-nitrify and react to form oxides when heated. Though the bulk of the NO_x is released at much higher temperatures, the dried Pu-Am spike begins to release NO_x at about 60 °C, which is ~40-50 °C lower than the lowest-temperature NO_x release from $Fe(NO_3)_3{\cdot}9H_2O$ (e.g., Wayne, 2015).

Little to no thermal analysis data on Pu or Am nitrate compounds exists in the literature. Bryan (1976) performed TGA analyses on three related Pu nitrate compounds: $Pu(NO_3)_4{\cdot}5H_2O$, $K_2Pu(NO_3)_6$, and $(NH_4)_2Pu(NO_3)_6$. Of these three compounds, the nitrate hydrate ($Pu(NO_3)_4{\cdot}5H_2O$) displayed mass loss beginning at the lowest temperature, approximately 100 °C, while reacting to an intermediate compound; possibly $PuO_2(NO_3)_2$. Drummond and Welch (1958) report mass loss from $Pu(NO_3)_4{\cdot}5H_2O$ starting at ~40 °C followed by "visible decomposition" at ~60 °C and dissolution in its own water of crystallization by 95-100 °C. No data on the thermal stability of Am nitrates could be found in the open literature. Some data on the thermal stability of the homologous compound to $Am(NO_3)_3{\cdot}6H_2O$, $Eu(NO_3)_3{\cdot}6H_2O$ exist (e.g., Wendlandt and Bear, 1960) which suggest that mass loss via dehydration in the Eu compound begins at ~100 °C though none of these studies, including those for Pu nitrate hydrates, have data on the coexisting offgas composition.

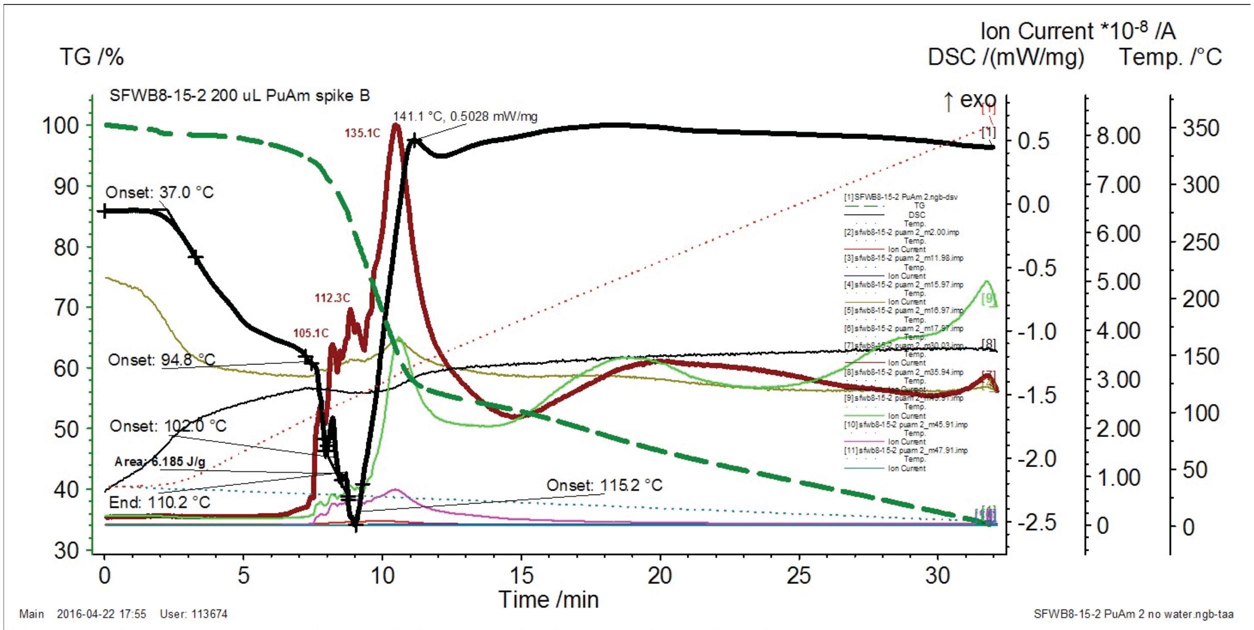
6.0 Discussion

The thermal behavior of all RNS surrogate samples—unspiked, acid-spiked and SNM-spiked—is dominated by three basic phenomena: 1) an endothermic dehydration reaction which onsets between ~38 and 50 °C, 2) an exothermic reaction which onsets between 108 and 123 °C related to the rapid gas release, foaming and expansion of the sample, and 3) steady-state, and slightly exothermic, combustion of the foamed sample above ~150-160 °C. Stage 1 is dominated by the release of copious amounts of H_2O and mass losses between 35 and 45%. Stage 2 is marked by a sudden increase in the NO_x and CO_2 content of the offgas as H_2O begins to tail off. The sustained release of small amounts of CO_2 and lesser amounts of NO_x and H_2O is typical of Stage 3.

Varying amounts of Pb nitrate has no significant effect on these basic phenomena, nor does the addition of a small amount of concentrated HNO_3+HF, though some subtle trends emerge from the few experiments conducted thus far. It appears that the RNS surrogates containing lower amounts of Pb are more reactive, particularly in the ~70-115°C temperature range. The RNS surrogate containing 1% and 2% $Pb(NO_3)_2$, both unspiked and spiked with 100 – 250 mL of 10M HNO_3+0.3M HF showed slight reactivity prior to the onset of foaming, whereas the surrogate containing 4% $Pb(NO_3)_2$ showed no such tendencies. The slope of the foaming reaction is steeper in samples spiked with acid, and with Pu-Am in acid.

The addition of 200μL of Pu-Am spike (in the same 10M HNO_3+ 0.3M HF acid matrix) causes small exothermic reactions in the surrogate. These onset between ~82 °C and ~108 °C, and precede the onset of the foaming reaction. The exothermic peaks are related to near vertical spikes in the NO_x (and also the H_2O and CO_2) content of the offgas. Each of the surrogate formulations appeared to respond similarly when the Pu-Am spike was added and the mixture heated, though the reactivity of the surrogate containing 1% $Pb(NO_3)_2$ appeared to be somewhat more vigorous.

Figure 14: DSC and gas emission data (minus H_2O and OH) from Pu-Am spiked SFWB8-15-2 B showing the relationship between NO_x emission and DSC exotherms.



[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 PuAm 2.ngb-dsv	2016-04-08	SFWB8-15-2 PuAm 2	SFWB8-15-2 PuAm 2	22.504	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 puam 2_m2.00.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 2.00		1/1	35°C/1.0(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 puam 2_m11.98.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 11.98		1/1	35°C/1.0(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 puam 2_m15.97.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 15.97		1/1	35°C/1.0(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 puam 2_m16.97.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 16.97		1/1	35°C/1.0(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 puam 2_m17.97.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 17.97		1/1	35°C/1.0(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 puam 2_m30.03.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 30.03		1/1	35°C/1.0(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 puam 2_m35.94.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 35.94		1/1	35°C/1.0(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 puam 2_m43.91.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 43.91		1/1	35°C/1.0(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 puam 2_m45.91.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 45.91		1/1	35°C/1.0(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 puam 2_m47.91.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 47.91		1/1	35°C/1.0(K/min)/1°C		---

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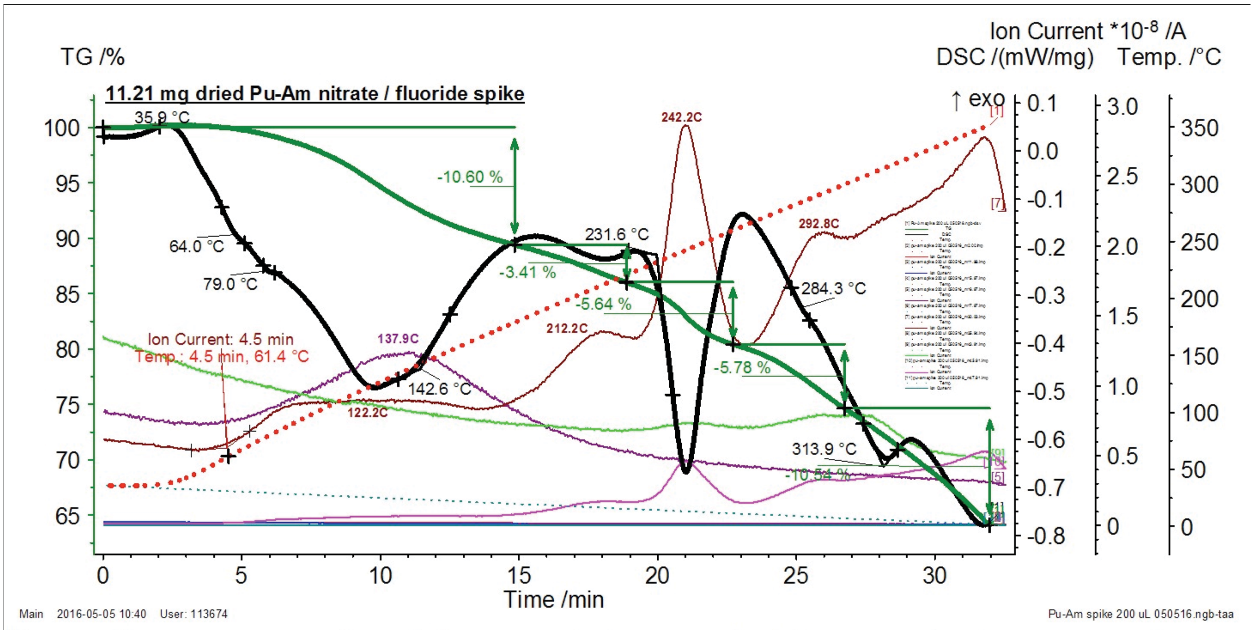
At this point, the nature and significance of these low temperature exotherms in the SNM-spiked samples is a matter of conjecture. It may be significant that the exotherms seen in the SNM-spiked samples correspond to step-like increases in the NO and NO_2 content of the offgas. This suggests that the presence of SNM ions in the surrogate hasten the breakdown of the nitrate matrix. The precise answer, unfortunately, may require further experimentation.

An STA-MS experiment on 11.21 mg of the dried residue from 200 mL of the Pu-Am spike (Figure 15) indicated that NO_x emission from actinide nitrate salts could start at temperatures well below 100 °C. Our data indicate that the evolution of NO_x initiated at ~61 °C, and remained at a plateau between ~80 °-150 °C. A previous experiment on a smaller quantity of the spike showed a transient NO_x maximum at ~92 °C. The availability of NO_x species to react with the organic constituent of the RNS surrogate at ~60 °C is significant in the context of nuclear waste remediation and storage.

Figure 15: ***DSC-MS plot showing mass change, gas emission, and enthalpy transformations (inflections and peaks) in the dried Pu-Am nitrate / fluoride spike.***



[#] Instrument	File	Date	Identity	Sample	Ma...	S..	Range	Atmosphere	Corr.
[1] STA 409PC/...	Pu-Am spike 200 uL 050516.ngb-dsv	2016-05-...	Pu-Am spike 200 uL 050...	Pu-Am spike 200 uL 050...	11....	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[2] QMS 403	pu-am spike 200 ul 050516_m2.00.i...	2016-05-...	pu-am spike 200 ul 050516	Mass 2.00		1...	36°C/1.1(K/min)/1°C		---
[3] QMS 403	pu-am spike 200 ul 050516_m11.98...	2016-05-...	pu-am spike 200 ul 050516	Mass 11.98		1...	36°C/1.1(K/min)/1°C		---
[4] QMS 403	pu-am spike 200 ul 050516_m15.97...	2016-05-...	pu-am spike 200 ul 050516	Mass 15.97		1...	36°C/1.1(K/min)/1°C		---
[5] QMS 403	pu-am spike 200 ul 050516_m16.97...	2016-05-...	pu-am spike 200 ul 050516	Mass 16.97		1...	36°C/1.1(K/min)/1°C		---
[6] QMS 403	pu-am spike 200 ul 050516_m17.97...	2016-05-...	pu-am spike 200 ul 050516	Mass 17.97		1...	36°C/1.1(K/min)/1°C		---
[7] QMS 403	pu-am spike 200 ul 050516_m30.03...	2016-05-...	pu-am spike 200 ul 050516	Mass 30.03		1...	36°C/1.1(K/min)/1°C		---
[8] QMS 403	pu-am spike 200 ul 050516_m35.94...	2016-05-...	pu-am spike 200 ul 050516	Mass 35.94		1...	36°C/1.1(K/min)/1°C		---
[9] QMS 403	pu-am spike 200 ul 050516_m43.91...	2016-05-...	pu-am spike 200 ul 050516	Mass 43.91		1...	36°C/1.1(K/min)/1°C		---
[10] QMS 403	pu-am spike 200 ul 050516_m45.91...	2016-05-...	pu-am spike 200 ul 050516	Mass 45.91		1...	36°C/1.1(K/min)/1°C		---
[11] QMS 403	pu-am spike 200 ul 050516_m47.91...	2016-05-...	pu-am spike 200 ul 050516	Mass 47.91		1...	36°C/1.1(K/min)/1°C		---

Created with NETZSCH Proteus software

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PA-DOP-01492, "Measurement Control of Balances and Revalidation of In-line Weights"

PA-PLAN-01186, Rev 0.1, "Differential Scanning Calorimetry and Mass Spectrometry (DSC-MS) of Remediated Nitrate Salt (RNS) Surrogates."

PLAN-TA9-2443, Rev. B, "Remediated Nitrate Salt Surrogate Formulation and Testing Standard Procedure"

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8.0 Acknowledgements

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MET-1

Plan

Approval Cover Sheet

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Chuck Tesch TA-55 Operations Manager	TA55-OPS	12/7/15	SIGNATURE ON FILE

Derivative Classification Review

☒ UNCLASSIFIED
  ☐ Export Controlled Information
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Guidance Used:

☐ CONFIDENTIAL ☐ SECRET
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  ☐ Formerly Restricted Data
  ☐ National Security Information

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Revision History

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Table of Contents

1.0 INTRODUCTION

1.1 Purpose

This document identifies scope and general procedural steps for performing Differential Scanning Calorimetry and Mass Spectrometry (DSC-MS) on remediated nitrate salt (RNS) surrogates to assess the impact of actinide addition on the thermal properties of these salts. This work supports the goals and objectives of the "Test Plan for Submittal to the New Mexico Environment Department" (LA-UR-15-27971), which has been developed to evaluate the efficacy of potential treatment options to remove the hazardous waste characteristic D001.

This Test Plan prescribes the requirements, responsibilities, and process for testing a range of chemical surrogates intended to mimic the energetic response of the RNS waste created during processing of legacy nitrate salts, with and without special nuclear material (SNM) content. A set of surrogates, developed and fabricated by M-7, will be tested with and without added U and/or Pu content in order to assess the impact of SNM components on thermal reactivity. This Test Plan will define the scope and technical approach for activities that implement Quality Assurance requirements relevant to formulation and testing. This Test Plan conforms to ASME NQA-1-2009A, Subpart 4.2, "Guidance on Graded Application of the Nuclear Quality Assurance (NQA) Standard for Research and Development."

1.2 Scope

This document covers the requirements for preparation of material and DSC-MS testing to gauge the response of RNS waste surrogates using Swheat Scoop© cat litter as an absorbent with and without added SNM component(s). The proposed work shall evaluate the impact of trace and minor levels of special nuclear materials (SNM) in the waste in order to determine whether surrogate thermal and reactive properties are affected by the SNM components (e.g., do they act catalytically to lower onset temperatures thereby increasing the reactivity or rate of oxidative reaction).

To begin to answer these questions, we are proposing a series of DSC-MS studies on waste surrogates (of known composition) in the following combinations:

Phase 1: RNS surrogates

Phase 2: RNS surrogates + SNM

Phase 3: RNS surrogates + zeolite ± SNM

Using surrogates fabricated as part of the overarching WIPP waste investigation, the Phase 1 analyses would provide a baseline to compare the results of the Phase 2 and Phase 3 analyses. Phase 2 analyses would be performed with the aim of determining how the presence of SNM (in the forms found in the actual LANL wastes) would affect the thermal behavior of the resulting waste product. Different forms of SNM (oxides, nitrates, residues) may be used, as well as different nitrate-to-organic ratios in the RNS surrogates. The results of these analyses would serve to provide evidence for the need for further analysis to be conducted exclusively on-site (due to SNM contamination concerns). If SNM content proves not to be an important factor in waste form reactivity, LANL would potentially pursue an external contractor to perform future experimental and analytical work.

Finally, zeolites have been proposed as a waste treatment approach by which the existing wastes could be rendered less reactive, if not completely inert. These analyses would begin to address the amount and type of zeolite best suited for inerting the existing RNS wastes. If SNM content proves to be a significant catalytic component during Phase 2, an additional round of experiments might be conducted during Phase 3 to determine the amount and type of zeolite required to inert RNS surrogates versus RNS + SNM surrogates.

This pilot study would examine 4 to 6 samples during each phase of the study, concentrating on the most reactive combinations of nitrate salts + organics in the LANL RNS waste forms. Similarly, at least 2 different forms of SNM (e.g., residues, salts, or oxides) in at least two different waste configurations (a total of 6 to 8 samples) shall be examined during Phase 2 with the aim of establishing a technical basis for including or excluding SNM content in future studies of RNS waste form stability. Finally, if needed, 4 to 6 samples of zeolite-treated RNS surrogate wastes (with or without actinides, depending on the outcome of Phase 2) shall be analyzed in order to ascertain the amounts needed to prevent spontaneous reaction and thermal runaway when mixed with the existing RNS wastes.

2.0 PRECAUTIONS AND LIMITATIONS

- Fabrication of the non-SNM surrogate materials is covered in a separate test plan. All work described in this test plan is covered by documents that have had ES&H review for all hazards and processes. The documents covering this work are:
    1. PMT2-MPR-DOP-015, Thermal Analysis with Off-Gas Analysis by Quadrupole Mass Spectrometry (U)
    2. PA-PLAN-01016, ARIES Oxide Production Project Quality Implementation Plan
    3. TA55-DOP-016, TA-55 Material Transfer Procedure
    4. NF-QA-004, Sample Receipt, Processing and Storage for the Bulk Actinide Nuclear Forensics Laboratory
    5. Process Monitoring Flow Diagram for Process/Status EOC, TA55-PMFD-01050 Experimental Oxide Characterization (EOC) MBA 712
    6. TA55-DOP-026, Operating Electronic Balances
    7. PA-CSP-01068, Material Characterization (and CSEDs referenced therein)
- Test Plan Changes: Changes to this Test Plan that redefine work scope or processes shall be documented in an approved revision. Release of the revision will require new signatures on the coversheet. Administrative changes or changes to the experimental details that do not affect the purpose or scope of the plan shall be documented in a scientific notebook per *Conduct of Research and Development* (SD601 Revision 2)

3.0 PREREQUISITES

3.1 Prerequisite Actions

- The author shall have the completed Test Plan reviewed for adequacy, accuracy, completeness and consistency.
- All reviewers will sign the front page of the test plan indicating their approval.

3.2 Training

Applicable training requirements are to be found in the DOPs and other documents listed in section 2, plus any other training (e.g., Fissile Material Handler Qualification) required to perform work in TA55, PF-4 laboratories.

Qualification and Approval of specific workers for activities in the DOPs and other documents in Section 2 are achieved through the Worker Qualification and Authorization System (WQAS) in LANL's UTrain System. When a worker is approved for a given DOP in WQAS, the RLM has acknowledged that the worker is qualified for the task.

The WQAS approvals are the only approvals needed for the activities described in this Test Plan.

4.0 PROCEDURE

This procedure describes the small scale (10-40 mg) testing and analysis of RNS surrogate and RNS surrogate + SNM formulations using Differential Scanning Calorimetry and Mass Spectrometry of the evolved gases (DSC-MS). The apparatus limits the test scale, though it is adequate for small-scale assessment of the effects of radioactive constituents on the reactivity of RNS surrogate formulations in response to heat. All activities described below are peer reviewed for technical accuracy and quality of records. Peer Review of individual tasks within a DOP follows the guidelines of P101-8, Explosives Safety. Analytical Data Sheets, Analytical Reports and other technical Memoranda are archived in PDMLink. For this activity, archived documents will include copies of lab notebooks, as applicable.

The RNS surrogate mixtures to be evaluated by this procedure will be fabricated by M-7 personnel, under test plan PLAN-TA9-2443, "*Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing Standard Procedure.*" The surrogates shall be transferred to TA-55, PF-4 using chain of custody procedures detailed in NF-QA-004 "*Sample Receipt, Processing and Storage for the Bulk Actinide Nuclear Forensics Laboratory*."

Storage and monitoring of all samples received from M-7 shall follow the guidelines presented in PLAN-TA9-2443. Samples will be labeled with their designated name, the date and time of preparation, and all appropriate hazard labels. Samples will be stored with caps secure in a normal laboratory environment. Testing will begin no earlier than 24 hours after the formulation and no later than 4 days after formulation. IF all testing cannot be started within the 30-day window, the formulation will be remade and all tests will be performed again.

The objective of DSC-MS testing and analysis is to evaluate the possible effects, catalytic or otherwise, of SNM constituents on the RNS surrogate formulations during heating. In order to approach "real world" conditions, each sample will be split and SNM shall be added to one of the subsamples. The SNM to be added will be in the form of liquid with combinations of plutonium, americium, and uranium in amounts approved by the SME. These dilute nitrate solutions containing Am, Pu, and/or U will be prepared and characterized by C-AAC. DSC-MS analyses will then be carried out on the corresponding SNM and non-SNM fractions in order to evaluate the potential effects of SNM content on surrogate reactivity. Two different surrogate formulations will be examined: one lower in nitrate salt content and another one lower in organic content. All steps involving sample preparation and manipulation shall be recorded in the official laboratory notebook and archived in PDMLink.

4.1 Addition of Radioactive Components

4.1.1 Surrogates prepared by M-7 will be received in PF-4 by C-AAC and transferred to MET-1. C-AAC shall also transfer a sufficient amount of well-characterized Pu/Am/U nitrate solution(s) to MET-1. The volume of dilute Pu-Am-U nitrate solution need not be large and, because the SNM content of the waste was relatively low, the SNM content of the RNS surrogate being tested shall be in a similar range.

4.1.2 A new container will be prepared and labeled for each sample.

4.1.3 Samples will remain sealed until the beginning of the test, and will be unsealed, as needed, in a PF-4 dry-air glovebox. The relative humidity measurement of the glovebox will be documented.

4.1.4 When opened, sample will first be divided into 2 equal sub-samples by weight.

4.1.5 A known amount of the Pu-Am nitrate solution shall be thoroughly mixed with the remaining sub-sample. The mixture shall be stored in the new sample container.

4.1.6 One sub-sample shall have an equivalent amount of nitrate solution without the SNM added to it, thoroughly mixed and returned to the original container and re-sealed.

4.1.7 The final Pu and Am concentrations in the sub-sample shall be calculated and documented in an official laboratory notebook.

4.1.8 Testing of each sub-sample will consist of DSC-MS analysis of ~20-40 mg aliquots scooped from the sample vial directly into the Pt-Rh sample pan.

4.2 DSC-MS Analysis

4.2.1 The DSC-MS procedure is documented in PMT2-MPR-DOP-015 "*Thermal Analysis with Off-Gas Analysis by Quadrupole Mass Spectrometry*" (Attachment 1). A detailed data sheet/process traveler is an integral part of this UET procedure, which also contains instructions for calibration and evaluation of data. The data sheets also contain validation information for the calibrated M&TE used in the process.

4.2.2 All testing will be carried out in triplicate and the individual results will be reported in the procedural data sheets, the laboratory notebook, and in a final report to be submitted by the PI. Analytical Laboratory reports will also be documented in PDMLink.

4.2.3 Instrument calibration constitutes a "User Performed Calibration" recognized by LANL's Standards and Calibration Lab. It utilizes 6 to 8 vendor-supplied high-purity SRMs and is fully described in PMT2-MPR-DOP-015. The instrument is a Netzsch STA-409-PC Luxx Simultaneous Thermal Analysis system. It is operated using vendor-supplied COTS software. The DSC instrument and software operation are verified using a material (e.g., KNO_3) having known transformation temperatures and enthalpy values, which are compared against the measured values. The verification measurement verifies both the temperature and enthalpy measurement capabilities of the instrument. For this work we will request that the calibration be performed within one calendar year of running the RNS surrogates, and that the instrument operation be checked daily. There are no other process aids or equipment that significantly influences the temperatures and enthalpies measured by DSC. The model and serial number of the DSC and balance used for the testing will be recorded in the laboratory report.

4.2.4 DSC measurement also requires a baseline analysis, which is used to remove thermal buoyancy effects from the data set. Only one baseline needs to be run prior to the initiation of RNS surrogate DSC analysis. The baseline file fixes all of the thermal parameters for the ensuing analytical runs and serves as a template for subsequent runs. A new baseline will be run every time run parameters are changed.

4.2.5 Mass spectrometry of the gas evolved during thermal analysis is performed using a Pfeiffer ThermoStar GSD301T3 Quadrupole Mass Spectrometer. The determination of absolute quantities of gas species is not required for this analysis; hence the MS is being used only to identify the individual evolved gas species. The only calibration that must be performed is a mass calibration, which ensures that the gas peaks are correctly positioned in the mass scale. A mass calibration shall be performed before testing starts and on a weekly basis thereafter. MS analysis is controlled, and MS data acquired, using proprietary COTS software ("Measurement" suite) supplied by the vendor.

4.2.6 Samples (10-40 mg) shall be manually scooped from the sample container into the Netzsch Pt-Rh alloy pans as needed, and a Pt-Rh lid shall be placed loosely on top of the pan. The pan is then manually transferred to the DSC measurement head, where it occupies the front analytical slot (the back slot is occupied by an empty reference pan). MS analysis and DSC software setup can be started at any time.

4.2.7 After sample loading is complete, the sample chamber is closed and Ar flow is started. An exhaust valve is opened after a slight positive pressure develops. When the gas flows and thermobalance measurements have stabilized (10-20 minutes after initiation of gas flow), analysis can begin. Previous analyses of WIPP-derived URS waste and residues have utilized a simple temperature profile consisting of a 10 °C /minute ramp to 500 °C. Unless requested, the same temperature profile for the surrogate analyses will be utilized.

4.2.8 After DSC analysis is initiated, data collection on the MS can be initiated. The MS can monitor over 10 different gas species, if needed. For these analyses, the following m/z ratios (each corresponding to a different gas species) shall be monitored:

Species	*m/z*	Dwell Time (s)	Resolution	**Species**	*m/z*	Dwell Time (s)	Resolution
$\mathbf{C^+}$	12	0.5	50	$\mathbf{(NO)^+}$	30	0.5	50
$\mathbf{O^+}$	16	0.2	50	$\mathbf{^{36}Ar^+}$	36	0.05	50
$\mathbf{(OH)^+}$	17	0.2	50	$\mathbf{(CO_2)^+}$	44	0.2	50
$\mathbf{(H_2O)^+}$	18	0.2	50	$\mathbf{(NO_2)^+/ CO_2H_2}$	46	1.0	50

Different gas species can be added or removed from the above, and analyses can be repeated by using fresh sample and the revised analysis template as needed.

4.2.9 Samples may be analyzed in duplicate, triplicate or quadruplicate, as deemed necessary by the customer/SME-PI. Establishing that no, or very little, change occurs between the SNM-bearing and non-SNM-bearing subsamples may require several repeat runs.

4.2.10 DSC-MS data are processed using proprietary Netzsch “Proteus” software which permits the user to integrate the gas MS data with the DSC data, and includes a wide variety of utilities for the determination of key reaction parameters such as peak temperature, onset temperature, end-of-reaction temperature, peak area, etc.

4.2.11 Thermograms and gas data can be exported to a COTS statistical package (e.g., Origin) to provide the end-user with relatively easy-to-understand output for inclusion in the final report.

4.3 DSC-MS Data

4.3.1 DSC-MS results from corresponding RNS surrogates that are free of SNM shall be compared to the fraction to which SNM has been added. Ultimately, the goal is to assess the differences in reactivity between the SNM-bearing fraction and the non-radioactive fraction. The criteria used to assess the differences in reactivity shall include differences in gas species released, onset temperature of the exothermic reaction(s) and the energy release(s) inferred from the peak areas on the thermogram. We anticipate that, while the numerical value of the enthalpy change (peak area) of the reaction will only be approximate, systematic differences in peak area between radioactive and non-radioactive sub-samples will correspond to real-world trends in reactivity.

4.3.2 Determination of onset temperatures, peak areas, etc., shall follow the guidelines provided in the vendor-supplied software.

5.0 QUALITY ASSURANCE

ASME NQA-1-2009A, Subpart 4.2, “Guidance on Graded Application of the Nuclear Quality Assurance (NQA) Standard for Research and Development” guided the development of this Test Plan. The test plan conforms to SD330, Los Alamos National Laboratory Quality Assurance Program. SD330 is implemented within MET-1 using PA-PLAN-01016, “*ARIES Oxide Production Project Quality Implementation Plan*.”

As part of the Quality Assurance activities for this work, the QA-SME may request tabletop and walk down reviews of documents and tasks prior to the start of formulation and analysis. The QA-SME may also request to observe any part of the work conducted under this Test Plan.

Examples of documents that the QA-SME may choose to review include calibration records for specific items, M&TE used, laboratory notebooks, chain of custody records, and data sheets derived from PMT2-MPR-DOP-015.

6.0 NONCONFORMANCES

In the event that a close out calibration or instrument check shows that the instrument is not functioning as expected (not conforming), an assessment will be made by the RLM of the impact to the relevant test or tests. The RLM, in conjunction with the appropriate SME will determine a path forward that may include reformulating and retesting RNS material.

7.0 DOCUMENT MANAGEMENT

The author shall obtain, from document management, a document control number after approval of this test plan.

8.0 TEST PLAN REVIEW AND APPROVAL

8.1.1 The author shall have the completed draft Test Plan reviewed for adequacy, accuracy, completeness, and consistency.

8.1.2 Reviewers shall be the RLM, Quality Assurance, and one or more appropriate Technical Reviewers.

8.1.3 All reviewers will sign the front page of the test plan indicating their approval.

9.0 TEST PLAN CHANGES

Changes to the issued Test Plan that redefine work scope or processes will be documented in an approved revision to this Test Plan.

Administrative changes or changes to the experimental details that do not affect the purpose or scope of the plan shall be documented in a scientific notebook per Scientific Notebooks (SDI-SP-003-Revision 0).

10.0 RECORDS AND RECORD REQUIREMENTS

Records compiled or generated by this process include:

- Calibration records for the balances and M&TE used for the sample preparation and analysis, as noted in section 4, above.
- Signed notebook pages documenting the analytical processes outlined above, and the date/time/ Z numbers of the staff involved during formulation/testing.
- Analytical Testing reports.
- Receipt documentation of the samples from M-7 to MET-1, via C-AAC.

Records will be compiled into an MET-1 memo or report that will be uploaded to PDMLink for archival purposes.

11.0 SOFTWARE QUALITY MANAGEMENT

Software used with the instruments described above is COTS and is managed through a formal Software Quality Management Plan that is part of PA-PLAN-01016 "*ARIES Oxide Production Project Quality Implementation Plan*." Software used for testing and analysis includes:

- Differential Scanning Calorimeter control software,
- Differential Scanning Calorimeter data analysis software,
- Quadrupole Mass Spectrometry software; and
- Statistical Analysis and Plotting software.

12.0 ENVIRONMENT, SAFETY, AND HEALTH

All work described above is covered by DOPs consisting of ES&H review for all hazards and processes.

13.0 RESPONSIBILITIES

13.1 Responsible Line Manager

- Verifies integration, consistency, and completeness of this Test Plan.
- Approves workers for the DOPs listed in Section 2. Approval is done through the Worker Qualification and Authorization System (WQAS).

13.2 Principal Investigator

- Verifies integration, consistency, and completeness of this Test Plan.

13.3 Technical Reviewer

- Confirms accuracy, adequacy, and completeness of this Test Plan.

13.4 Document Control

- Assigns document number and effective date for this Test Plan.

13.5 Worker

- Verifies qualification and approval for activities in WQAS before carrying out work.

14.0 ACRONYMS

Term	Description
ACS	American Chemical Society
DOE	United States Department of Energy
DSC	Differential Scanning Calorimetry
DWI	Drop Weight Impact
EP	Environmental Programs
S&CL	LANL Standards & Calibration Laboratory

Term	Description
IESL	Institutional Evaluated Supplier List
IWD	Integrated Work Document
LANL	Los Alamos National Laboratory
M&TE	Measurement and Test Equipment
QA	Quality Assurance
RNS	Remediated Nitrate Salt
TP	Test Plan
WQAS	Worker Qualification and Authorization System
M-7	Weapons Experiments High Explosives Science & Technology group

15.0 ATTACHMENTS

Number	Title
A	Test Descriptions
B	Quality Implementation Matrix

Attachment A: TEST DESCRIPTIONS

Differential Scanning Calorimetry (DSC)

Operation of the DSC in PF-4 is authorized under PMT2-MPR-DOP-015. DSC measures the thermal response of a material by monitoring the heat flow into or out of that material as it is heated at a constant ramp rate. The sample is placed in a Pt-Rh alloy pan that is covered by a perforated Pt-Rh alloy lid, but not sealed or crimped. The pan is placed on a DSC carrier platform, along with an empty reference pan. The furnace is mechanically lowered over the DSC carrier, and gas flow (UHP argon) is initiated. The furnace is ramped following a user-determined thermal profile (for our purposes, a simple ramp from room temperature at 10˚C/min to 500°C) while heat flow to the sample and reference pans is monitored. Endothermic events require more heat to flow to the sample to keep its temperature increasing at the desired ramp rate. Exothermic events cause the furnace power to be reduced for the same reason. With this method, melts, phase transitions, decomposition, and other features can be quantitatively measured.

Quadrupole Mass Spectrometry

Operation of the quadrupole mass spectrometer in conjunction with the DSC in PF-4 is authorized under PMT2-MPR-DOP-015. Gases evolved during the thermal ramp used for DSC analysis are entrained in a stream of ultra-high-purity argon (Ar) gas supplied by a dedicated, wall-mounted cylinder operated at ~7.5 psi. Gas flows to the instrument are controlled using needle valves that restrict the total gas flow to ~80 mL/min. The entrained gases are conducted through heated silica capillary into the ionization chamber of the mass spectrometer (MS). Sampling is by differential pressure, and ionization (positive or negative) is accomplished under vacuum (1.0 x 10^{-6} mbar) by electron impact. Ions are routed into the analytical sector where they are scanned using the quadrupole mass analyzer. Ions may be detected by means of a Faraday Cup, or by an electron multiplier operated at 1200 V. For these analyses, all detection is performed using the electron multiplier. Detector dwell times are specified by the user and are set up prior to the start of analysis. Species for monitoring are also specified by the user. The mass range of the MS is 1-300 amu. Once the DSC run begins, the user can initiate MS data collection. Quantitative analysis is possible, but will not be necessary for the testing and analysis described in this test plan.

Attachment B - QUALITY IMPLEMENTATION MATRIX

NQA-1 Rqmnt	DESCRIPTION	EXCERPTS FROM NQA-1 PART IV SUBPART 4.2, GUIDANCE ON GRADED APPLICATION OF NQA STANDARD FOR RESEARCH & DEVELOPMENT	TEST PLAN IMPLEMENTATION METHODOLOGY
1	Organization	601.1 General. An organization should be defined for R&D work to describe roles, responsibilities, and authorities that support achievement of work objectives. Interface responsibilities should be defined between R&D and support functional elements 601.4 Development and Support. Roles, responsibilities, and authorities should be defined for development and support activities. They should address those doing the work and those who perform independent verification that work objectives have been met. Interface responsibilities with design and engineering functions should be defined, as appropriate, to ensure that developmental results are useable.	This test plan Section 13, and by reference: SD330 LANL QA Plan SD601 Conduct of R&D P315 Conduct of Operations See also items below that outline roles and responsibilities, worker qualification, documentation, and peer review.
2	Quality Assurance Program	602.1 General. A graded approach based on importance and significance of activities is key to the successful application of the NQA standard to R&D activities. The R&D quality assurance program should be based on the proven processes that govern the performance of successful scientific research. Highly qualified and motivated people who are engaged in selective investigation activities, that are carefully reviewed by independent competent peers, will turn out documented results that are verifiable and able to withstand scrutiny by reviewers, potential users, and the entire research community. 602.4 Development and Support. Development activity entails the application of a proven theory and its extension to a practical situation. The plan that governs a developmental activity leads to a more structured management of the entire process. For example, progress is measured against a predetermined set of results that appear to be appropriate at the outset. However, there are sufficient technical. Uncertainties in a development project to warrant some	SD 330 is the institutional quality assurance program. SD601 Conduct of R&D PLAN-WXDIV-2142 is the division quality assurance plan that implements some specifics of SD330 locally. See section 3.2 for Training (and IWDs as incorporated by Reference). PA-PLAN-01016, ARIES Oxide Production Project Quality Implementation Plan

NQA-1 Rqmnt	DESCRIPTION	EXCERPTS FROM NQA-1 PART IV SUBPART 4.2, GUIDANCE ON GRADED APPLICATION OF NQA STANDARD FOR RESEARCH & DEVELOPMENT	TEST PLAN IMPLEMENTATION METHODOLOGY
		flexibility. This is frequently taken into account in the formality associated with the preparation and revision of design and process documentation, and by including in the milestones a plan for evaluating performance at various key junctures during the project. Tests are prescribed with requirements commensurate with the complexity and scale of the work, and with the associated risk to the public, workers, and environment and future success of the project.	
3	Design Control	603.4 Development and Support. For development and support activities, the level of design control should be applied to support the input needs of the design process. In some cases, considerable importance is placed on R&D results to demonstrate the acceptability of innovative design.	Not Applicable. Nothing is being designed.
4	Procurement Document Control	604.4 Development and Support. For development and support activities, the level of procurement document control should be applied to support a commercial design basis, i.e., engineering design system criteria.	SD330, P840-1, PLAN-WXDIV-2142, P1020-2, and P1020-1. In this Test Plan, the relevant procurement documents are the Certificates of Analysis from Fisher for the chemicals. These will be assembled into a memorandum that is archived in PDMLink. See sections 4, 10, and 11 of this Test Plan for more detail on specific procurement document controls.
5	Instructions, Procedures, and Drawings	605.4 Development and Support. Activities should be performed in accordance with documented instructions, procedures, or drawings, as directed by the researcher / developer.	This Test Plan and several IWDs contain the instructions and procedures needed for the work. Refer to Section 2.0 of this Test Plan and other content. Thermal Analysis of Off-Gas Analysis by Quadrapole Mass Spectroscopy, PMT2-MPR-DOP-015. P315 Conduct of Operations

NQA-1 Rqmnt	DESCRIPTION	EXCERPTS FROM NQA-1 PART IV SUBPART 4.2, GUIDANCE ON GRADED APPLICATION OF NQA STANDARD FOR RESEARCH & DEVELOPMENT	TEST PLAN IMPLEMENTATION METHODOLOGY
6	Document Control	606 NQA-1. Requirement 6; Document Control. This element is applicable to R&D activities. As a minimum, laboratory notebooks should be subject to document control procedures. Also, the process for development of intellectual property documentation should be subject to document control.	SD330, PLAN-WXDIV-2142, P1020-2, and P1020-1. In this Test Plan, Laboratory Notebook pages will be copied and attached to the Analytical Reports that are archived in PDMLink.
7	Control of Purchased Materials, Items, and Services	607 NQA-l, Requirement 7; Control of Purchased Materials, Items, and Services This element is applicable to R&D activities. The degree of application should support the desired results of the work, within the specified performance boundaries. The need to ensure conformance with specified requirements depends on the objectives of the work. If the quality of work results depends on the pedigree of materials, items, or services, the work should be planned to include this Requirement.	SD330. In this Test Plan, chemicals will be purchased from Fisher or VWR through GSS. All three vendors are on the IESL list. Chemicals will be purchased with Certificates of Analysis.
8	Identification and Control of Items	608 NQA-1, Requirement 8; Identification of Control Items. This element is applicable to R&D activities. The degree of application should support the desired results of the work, within the specified performance boundaries. If the quality of work results depends on the pedigree of materials or items (e.g., analytical chemistry), this Requirement applies.	SD330.In this Test Plan, individual items needing specific controls have been identified either specifically or by implication (e.g. the statement that a measurement requires a certain tolerance). For those items, either S&CL control is required or the use of an internal standard to verify operation is used. See sections 4, 10, and 11 of this Test Plan for more detail on specific item control.

NQA-1 Rqmnt	DESCRIPTION	EXCERPTS FROM NQA-1 PART IV SUBPART 4.2, GUIDANCE ON GRADED APPLICATION OF NQA STANDARD FOR RESEARCH & DEVELOPMENT	TEST PLAN IMPLEMENTATION METHODOLOGY
9	Control of Processes	609 NQA-l, Requirement 9; Control of Processes, 609.1 General. The control of processes varies considerably as one advances from basic research through development. 609.4 Development and Support. Process control during this phase is formalized. Formalization occurs at the project or program level. Work processes and supporting activities are defined, and work and operating procedures are developed and implemented with respect to safety considerations, quality, cost, schedule, and programmatic mission. Methods of implementation and training requirements are formally defined.	SD330 and Documents referenced in the Test Plan that control work process development at the division level.
10	Inspection	610.1 General. Basic and applied research activities are not amenable to inspection. Consideration may be given to performing inspection-like activities on basic and applied research to establish process or product control limits. 610.4 Development and Support. The researcher/ developer should anticipate the need and plan for inspection criteria for advanced development work to interface with design process needs.	Inspection of received items is carried out by the receiver checking to ensure that the lot number of the received item matches the lot number on the Certificate of Analysis. Inspection of instruments includes verifying that the internal standards are showing expected results. These activities are part of everyday formulation/analysis activities and are not formally called out in the Test Plan.
11	Test Control	611.1 General. Test control does not apply uniformly to basic and applied research. Where applicable, test methods and characteristics shall be documented and the approaches and procedures recorded. Test control does not apply to basic and applied research activities in which hypotheses are being evaluated. It does apply to support activities associated with the conduct of research. 611.4 Development and Support. Characteristics to be tested and test methods should be specified. The test results should be documented and their conformance to acceptance criteria	The specific test methods and outputs are documented above along with descriptions of the evidence used to ensure that they are conforming to expected performance. This Test Plan constitutes the planning of the tests. Test results will be documented in Analytical Reports that are archived in PDMLink. See sections 9 and 10 of this Test Plan for details on test control.

NQA-1 Rqmnt	DESCRIPTION	EXCERPTS FROM NQA-1 PART IV SUBPART 4.2, GUIDANCE ON GRADED APPLICATION OF NQA STANDARD FOR RESEARCH & DEVELOPMENT	TEST PLAN IMPLEMENTATION METHODOLOGY
		evaluated. Tests required should be planned, executed, documented, and evaluated.	
12	Control of Measuring and Test Equipment	612.1 General. The researcher should specify the requirements of accuracy, precision, and repeatability of measuring and test equipment (M&TE). These requirements have different implications for basic, applied, and development work. 612.4 Development and Support. During the process development stage and for all R&D support activities, M&TE should be controlled. The degree of control should be dependent on the application of the measurement.	Specific items needing S&CL calibration are called out in the test plan either specifically or through implication by statement of a required tolerance. Calibration of the DSC-MS is administered by the S&CL as a User Performed Calibration (UPC), and is documented by the user. Calibration files and results shall be made available to technical reviewers, Management, and Quality personnel. Items not called out in those fashions are controlled through the use of internal standards that verify their operation. Use of M&TE in PF-4 is controlled under RPS-AP-025 "*Control of Measuring and Test Equipment (M&TE)*."
13	Handling, Storage, and Shipping	613 Handling. Storage And Shipping. This element is applicable to R&D activities. Good laboratory practices may be defined as instructions used for conducting the activity.	"Handling" in performance of this R&D work is addressed by SD601, Conduct of R&D, the content of this test plan, including Integrated Work Documents (IWDs) incorporated by reference.
14	Inspection, Test, and Operating Status	614.1 General. This criterion has limited applicability for R&D activities. 614.4 Development and Support. The status of items and processes for which inspections and tests are specified, should be identified by tags, markings, inspection and test records, or other suitable means. The authority for application and removal of inspection and test identification should be specified.	P330-2. This only applies to items calibrated by S&CL. These items have visible calibration stickers attached. Any item that is "calibrated" per this Test Plan is understood to be on the S&CL program.

NQA-1 Rqmnt	DESCRIPTION	EXCERPTS FROM NQA-1 PART IV SUBPART 4.2, GUIDANCE ON GRADED APPLICATION OF NQA STANDARD FOR RESEARCH & DEVELOPMENT	TEST PLAN IMPLEMENTATION METHODOLOGY
15	Control of Nonconforming Items	615 This Requirement should apply only to R&D support activities. The results of R&D activities are not expected to meet predetermined requirements; therefore, obtaining unexpected results does not constitute a nonconforming condition. The point at which a nonconformance can be identified is the point at which development work has transitioned into design or production of engineered items.	Per Part IV, Subpart 4.2, para 103.4, this applies to calibrated items. If calibrated items or items checked with internal standards show nonconformances, per this Test Plan, an assessment will be made by the RLM and then, in conjunction with the SME, a path forward will be determined. This may include reformulation and/or retesting. See section 6.0 of this Test Plan for details on nonconforming items.
16	Corrective Action	616.1 General. Conditions adverse to quality can be identified for R&D activities, depending on the certainty of operating assumptions and expected results. The documentation, reporting, and tracking of conditions adverse to quality are done at the discretion of the researcher. 616.4 Development and Support. Responsibility should be defined for the identification, cause, and corrective action for significant conditions adverse to quality; these should be documented and reported to appropriate levels of management. Follow-up actions should be taken to verify implementation and effectiveness of corrective action.	Corrective action will apply items as noted above and to the Test Plan and associated documentation. Item nonconformance corrective action is described above and in the Test Plan. Document nonconformance includes everything from simple typographic errors to incorrect process and procedures. Per this Test Plan, nonconformances that do not affect the purpose or scope may be documented in a scientific notebook. Other nonconformances will be documented in an approved revision to the document. This guidance is consistent with the M division Technical Plan and Integrated Work Document policies, AP-WXDIV-2385 and AP-JDIV-1019. See section 6 of this Test Plan for details on Corrective Actions. MAP-0002 "*Corrective Action*"

NQA-1 Rqmnt	DESCRIPTION	EXCERPTS FROM NQA-1 PART IV SUBPART 4.2, GUIDANCE ON GRADED APPLICATION OF NQA STANDARD FOR RESEARCH & DEVELOPMENT	TEST PLAN IMPLEMENTATION METHODOLOGY
17	Quality Assurance Records	617 Quality Assurance Records. This element is applicable to R&D activities. In many cases, the notebook or journal of the researcher is the QA record. Controls are needed for these documents, e.g., maintain copies of critical pages or access-controlled filing when not in use to preserve process repeatability and the QA record. Electronic media may be used to record data and should be subject to appropriate administrative controls for handling and storage of data.	SD 330. Documents will be captured in memoranda or reports that are archived in PDMLink. See sections 7 and 10 of this Test Plan for more detail on records. P1020-1, Laboratory Records Management
19	Audits	618.1 General. Planned requirements are not always defined for R&D work; therefore, audits should be conducted in a graded manner. R&D audit activities include normally accepted assessment practices, peer reviews, or both. 618.4 Development and Support. Responsibility should be defined for audits and the results of these audits should be documented and reported to appropriate levels of management. Follow-up actions should be taken to verify implementation and effectiveness of corrective action.	Section 5.0 of this Test Plan guides the usage of surveillances. Surveillances may include tabletop and walk down reviews of documents and tasks prior to start of work and during actual execution. Surveillances will be carried out at the discretion of the QA-SME and coordinated with the Principle Investigator.
*	Software QA	Note: the NQA-1 Subpart 4.2 guidance on R&D does not specifically address the use of Software, however, the DOE QA Order 414.1D and EM QA Program, EM-QA-01 Rev. 1, establish requirements for safety and non-safety software using a graded approach. Established LANL Software QA programs and procedures defining controls for the acquisition, development, and/or use of software should be applied. This includes commercial off-the-shelf (COTS) software used for the control of instrumentation and the recording of data obtained by instrumentation.	SD 330. Software quality will be documented in division implemented SQM forms. All software is COTS and is standard software used in many different places. Software used (including current revisions) is specified under PMT2-MPR-DOP-015, and is covered under PA-PLAN-01016, “*ARIES Oxide Production Project Quality Implementation Plan*.” See sections 4 and 11 of this Test Plan for details on Software QA.

*Application of Software QA requirements to this scope of work is a requirement of DOE O 414.1D and EM-QA-001 Rev. 1.

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Title: Simultaneous Thermal Analysis of WIPP and LANL Waste Drum Samples: A Preliminary Report

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SIMULTANEOUS THERMAL ANALYSIS OF WIPP AND LANL WASTE DRUM SAMPLES: A PRELIMINARY REPORT

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Background

On Friday, February 14, 2014, an incident in P7R7 of the WIPP underground repository released radioactive material into the environment. The direct cause of the event was a breached transuranic (TRU) waste container, subsequently identified as Drum 68660. Photographic and other evidence indicates that the breach of 68660 was caused by an exothermic event. Subsequent investigations (Britt, 2015; Clark and Funk, 2015; Wilson et al., 2015; Clark, 2015) indicate that the combination of nitrate salts, pH neutralizing chemicals, and organic-based adsorbent represented a potentially energetic mixture.

The materials inside the breached steel drum consisted of remediated, 30- to 40-year old, Pu processing wastes from LANL. The contents were processed and repackaged in 2014. Processing activities at LANL included: 1) neutralization of acidic liquid contents, 2) sorption of the neutralized liquid, and 3) mixing of acidic nitrate salts with an absorber to meet waste acceptance criteria. The contents of 68660 and its sibling, 68685, were derived from the same parent drum, S855793. Drum S855793 originally contained ten plastic bags of acidic nitrate salts, and four bags of mixed nitrate and oxalate salts generated in 1985 by Pu recovery operations. These salts were predominantly oxalic acid, hydrated nitrate salts of Mg, Ca, and Fe, anhydrous $Na(NO_3)$, and minor amounts of anhydrous and hydrous nitrate salts of Pb, Al, K, Cr, and Ni. Other major components include sorbed water, nitric acid, dissolved nitrates, an absorbent (Swheat Scoop®) and a neutralizer (KolorSafe®). The contents of 68660 are described in greater detail in Appendix E of Wilson et al. (2015).

S855793 contained approximately two gallons of free liquids: presumably a mixture of water, nitric acid, and dissolved nitrates. The liquid was drained from the parent drum and from the salt-filled bags and neutralized using KolorSafe®; a color-indicating neutralizing agent composed of triethanolamine (TEA) and an indicator dye. The resulting near-neutral liquid was then solidified using Swheat Scoop®, and placed on top of solid “job control” waste (e.g., plastic bags and bottles, and a glove-box glove) in 68660. Swheat Scoop® absorbents are 100% wheat-derived solids, composed of starch (65-70 wt. %), proteins (14 wt. %), plus lignin, lipids, cellulosic polymers, enzymes, and an indigenous microbial population.

It is likely that the starch component in the Swheat served as the predominant fuel component in 68660, though other organics such as low-density and high-density polyethylene and polyvinyl chloride were also present. Nitrate salts and nitric acid both serve as oxidizing and nitrating agents when in contact with carbonaceous materials. If nitric acid is neutralized with TEA the corresponding nitrate salt, triethanolammonium nitrate (TEAN), is produced. TEAN is more reactive than TEA as it comprises both the fuel (organic) and oxidizer (nitrate) in the same molecule. TEAN melts at 80 °C and decomposes exothermically beginning at about 250 °C (Bracuti, 1992).

The remaining damp acidic nitrate salts from S855793 were also combined with Swheat Scoop®. This nitrate salt / Swheat mixture was distributed between 68660 and 68685 in somewhat different configurations. In 68660, the salt mixture was placed on top of the layer of neutralized and sorbed liquid. In 68685, there were no sorbed liquids, and the salt mixture was placed directly into the bottom of the drum and overlain with a lead blanket from the parent drum (Wilson et al., 2015). The physical configuration of the wastes within 68660 may have contributed to the reactivity of the contents (Wilson et al., 2015). Wastes were situated within the drum in a manner that created several interfacial regions having contrasting chemistries and thermal conductivities: (1) solid "job control" and neutralized sorbed liquid, (2) neutralized sorbed liquid and nitrate-salt mixture, (3) neutralized sorbed liquid and polyethylene liner, (4) nitrate-salt mixture and polyethylene liner, and (5) nitrate-salt mixture and drum head space. Wastes having different compositions were situated as layers within the drum, and the resulting interface between adjacent layers of neutralized-and-sorbed liquid + organics and nitrate salt + organics may have created localized reactive zones that led to the thermal runaway event.

Table 1: Identification and physical appearance for each sample analyzed using DSC-MS

Sample ID	Drum Number	Description (before run – samples ground and dried at 110 °C unless otherwise noted)	Description (after run)
4174-1-6	S802701	Translucent crystals, not ground – from top layer	White-clear glassy residue
4174-2-7	S813389	White-gray powder and crusty aggregates w/darker granules –from top layer, not ground	Clear, frothy, glass-like residue
69120	S855793	Sample 1: White / tan powder & aggregates w/ black & white specks – monolith 1 scraped from emptied parent drum	Reddish brown ('nutmeg') crust
69120	S855793	Sample 2: White-tan powder w/brown flecks – monolith 2 scraped from emptied parent drum	Reddish brown ('nutmeg') powder
69120	S855793	Sample 3: Brown-gray powder w/brown & white flecks – monolith 3 scraped from emptied parent drum	Reddish brown ('nutmeg') crust
69139	S864694	Gray-tan powder w/white flecks – scraped from emptied parent drum	Reddish brown ('nutmeg') powder
68492	S822952	Brown-gray powder w/white flecks – scraped from emptied parent drum	Brick-red crust
68645	S818449	Yellowish-tan powder, slightly hygroscopic, w/white flecks – scraped from emptied parent drum	Reddish brown ('nutmeg') crust
69173	S851436	Tan-gray powder – scraped from emptied parent drum	Dark brown / black powder

An interdisciplinary study conducted by multiple DOE laboratories (Wilson et al., 2015) concluded: "*The physical evidence from P7R7, combined with thorough analysis of available sample data, chemical reactivity testing, and analytical modeling, indicates that Drum 68660 breached as a result of internal reactions that were exothermic in nature. The Technical Assessment Team (TAT) hypothesizes that a sequence of exothermic chemical reactions led to a thermal runaway, which resulted in solids, radioactivity, and hot gases being released from the drum.*"

The report also concludes that the contents of 68660 did not detonate. Rather, the breach was the result of "*…internal chemical reactions that generated heat and produced gases that built up pressure sufficient to overcome the drum vent and seal*." Thermal analyses of samples from 68660 were an important part of the data set in Wilson et al. (2015), but these did not include DSC measurements. While we had no access to samples from drum 68660, samples from its parent (S855793) were available to us. We also analyzed analogous materials sampled from four different drums that had similar contents and age.

A Brief Description of the Samples

Samples analyzed for this study fall into four broad categories (Table 1).

1. Pure nitrate and nitrate hydrate salts (Na, K, Pb, Mg, Fe^{3+}), and calcium oxalate hydrate.
2. Samples of nitrate salts taken from the top layer of waste inside two full parent drums. (S802701 and S813389) that had never been processed. These are referred to as "top layer salts" in this report.
3. Salts scooped out of the bottom of four parent drums that had been emptied at WCRRF. These are referred to as "residual salts" in this report.
4. Three "monolith" samples labelled 69120 (1-3) are residual salts from drum S855793, the parent of 68660. These samples consist of discrete chunks of solid material found in the material scraped from the interior of the drum.

We performed simultaneous thermal analysis and mass spectrometry of the offgas (STA-MS) on pure reagent-grade Na, Pb, Mg, Fe (III), and K nitrates and Ca oxalate obtained from known sources from within LANL's Chemistry and Manufacturing Engineering Technologies divisions in order to compare the thermal behavior of these compounds with that of the mixtures found in the various waste forms.

Top layer salts are comprised of largely water-soluble hydrated nitrate salts of Na, K, Ca, Mg, and Al that are bagged-out and packed in poly-lined drums. In many instances, the bags have deteriorated due to radiolysis. The residual salt samples (Table 1) from the emptied drums are essentially the 'leftovers' of the first phase of the waste drum remediation process; wherein parent drums are emptied into a glovebox, the wastes treated as required and then repackaged into separate daughter drums that are eventually transported to WIPP. The residual salts consist of discrete chunks of heterogeneous material (monolith), or aggregates of "moist, free-flowing" to "moist, clumping," macroscopically heterogeneous solids (Chamberlin and Martinez, 2014; Chamberlin et al., 2015).

While the residual salts ostensibly constitute untreated, unremediated nitrate and oxalate salts and related residues (bits of lead shielding, rust particles from the drum interior, etc.), it is possible that they have been contaminated with Swheat Scoop®. There are two possible avenues for cross-contamination with Swheat Scoop® (Schumann, 2015):

1) Wet salts from the parent drum were emptied into a glovebox at WCRRF and combined with the neutralizer and adsorbent manually, using hand tools. The same tools are also used to remove salt bags and other items from the parent drum. If hand tools were contaminated with Swheat during sorbent mixing and were then used to scoop residues out of the parent drums, cross contamination of the residues left in the drum (and later sampled for analysis) is possible.

2) During the time these drums were processed at WCRRF, it was a routine 'best management practice' to add a small amount of Swheat to an empty parent at the end of the process, before it was bagged-off, in order to ensure that any remnants of liquid in the emptied drum were completely absorbed and no longer problematic.

Table 2: MS operating parameters. Only OH, H_2O, NO, ^{36}Ar, and CO_2 were measured during every analysis

Species	*m/z*	Dwell Time (s)	Resolution	**Species**	*m/z*	Dwell Time (s)	Resolution
$(H_2)^+$	2	0.5	50	**$(NO)^+$**	30	0.2	50
He^+	4	0.5	50	**$(O_2)^+$**	32	0.05	50
C^+	12	0.5	50	**$^{36}Ar^+$**	36	0.05	50
O^+	16	0.2	50	**$(C_3H_5)^+$**	41	1.0	50
$(OH)^+$	17	0.2	50	**$(CO_2)^+$**	44	0.2	50
$(H_2O)^+$	18	0.2	50	**$(NO_2)^+$**	46	1.0	50
$(C_2H_3)^+$	27	1.0	50	**$(SO)^+$**	48	0.5	50
$(C_2H_5)^+$	29	1.0	50				

Table 3: DSC temperature profiles

	Preparation	**Analysis**
Profile 1	10 °C/minute ramp to 110 °C, 10 min. isotherm @ 110 °C	5 °C/minute ramp to 350 °C
Profile 2	10 °C/minute ramp to 110 °C, 10 min. isotherm @ 110 °C	2.5 °C/minute ramp to 350 °C
Profile 3	10 °C/minute ramp to 110 °C, 10 min. isotherm @ 100 °C	5 °C/minute ramp to 500 °C
Profile 4	none	10 °C/minute ramp to 1100 °C

Table 4: Calibration set 6.223.5.-91.2, RT-900 °C (for Pt-crucibles)

Material	**T_{rxn} (°C)**	**Enthalpy (J/g)**	**Purity**	**Molecular Weight (amu)**	**K =($\Delta H_{ref}/\Delta H_m$)**
$C_6H_5C_6H_5$	69.2	120.4	99%	154.21	1.040
C_6H_5COOH	122.3	147.3	>99.9%	122.12	1.096
$RbNO_3$	164.2	26.6	99.99%	147.48	1.126
$RbNO_3$	285.0	8.75	99.99%	147.48	1.142
Ag_2SO_4	426.4	51.7	99.999%	311.8	1.064
CsCl	476.0	17.2	99.999+%	168.36	1.021
K_2CrO_4	668	38.9	99.5%	194.20	0.806
$BaCO_3$	808	94.9	99.98%	197.35	0.666

Analytical Details

DSC-MS runs were performed using a Netzsch STA 409PC Luxx, operated in simultaneous TG-DSC mode. The TGA-DSC measurement head (part number 6.227.1-70+S) is designed so that thermogravimetric

(TG), differential scanning calorimetry (DSC), and evolved gas data can be collected from the same sample, simultaneously, during a single experiment. The resolution of the thermobalance is 2 μg, and the thermocouple (an integral part of the measurement head itself) is accurate to within ~0.2 °C. All data were collected and corrected for buoyancy and background effects using proprietary Netzsch "Measurement" software (v4.3.1; 17-5-2004) and processed using proprietary Netzsch "Proteus" software (v4.3.1; 17-5-2004). Onset temperatures were obtained using the 'onset' capability in Proteus. Enthalpy values were obtained by choosing a reasonable background value on either side of the peak and integrating the peak area in Proteus. Mass changes were evaluated by using the first derivative of the mass change curve (labeled "DTG" in all plots). For all energy plots, exothermic peaks point upwards.

Table 5: Thermodynamic data for phase transitions in K(NO_3) from Breuer and Eysel (1982) and measured in the TGA-DSC apparatus used for this study

Transition	Onset T. (°C) literature	Onset T. (°C) measured	%RSD	DH (J/g) literature	DH (J/g) measured	%RSD
2nd order	**129**	129.5	+0.4	**-49.47**	-55.95	+13.1
melting	**334**	335.6	+0.5	**-98.35**	-94.93	-3.5
2nd order	**129**	129.0	0	**-49.47**	-55.26	+11.7
melting	**334**	335.7	+0.5	**-98.35**	-92.43	-6.0
2nd order	**129**	128.9	-0.08	**-49.47**	-56.19	+13.6
melting	**334**	335.7	+0.5	**-98.35**	-91.86	-6.6

The STA-MS is coupled to a Pfeiffer ThermoStar quadrupole MS (GSD 301T) via a 1m long heated (200 °C) silica capillary transfer line. Mass spectrometer data were acquired and processed using the Pfeiffer ThermoStar Suite (Quadstar 32-Bit, v7.03). The mass spectrometer was operated in peak hopping mode (MID), using electron ionization (EI, 70 eV). Aside from OH and H_2O, the following species were monitored for each sample: O, NO, ^{36}Ar, CO_2, and NO_2. In addition, the following species were also monitored during selected analyses: H_2, O_2, and SO. The RF polarity was normal, the ionization filament current was 1.0 mA, and the multiplier (SEM - channeltron) voltage was 1200 V during all phases of analysis. The remaining operating parameters for the GSD 301T are summarized in Table 2.

The entire apparatus (DSC and MS) is located inside a dry air glovebox fitted with hard-plumbed, filtered gas inlets and sealed electrical and telemetric feedthroughs. During analysis, ambient glovebox conditions were: temperature (T) = 27 to 32 °C, relative humidity (%RH) = 0.0 - 0.3%. Pre-run baseline values varied from approximately -6 to -5 μV.

To facilitate the thermal analysis of small (10-50 mg) quantities, the residual salt and monolith samples were dried in air at 100 °C and ground to a fine powder by C-AAC personnel. Top layer salt samples S802701 and S813389 were dried without grinding. Immediately prior to analysis, all samples were re-ground using an agate mortar and pestle. For STA-MS analysis, each nitrate and WIPP / LANL waste sample was subjected to a 10 minute-long isothermal drying interval at ~110 °C to remove reabsorbed atmospheric moisture from the samples. All relevant data was collected following the isothermal step, which served only to purge the sample of water adsorbed after the samples were first prepared by C-AAC. For all nitrate samples, and for the first round of (uncalibrated) exploratory WIPP sample runs, temperature Profile 1 was used (Table 3). Following calibration of the measurement head, Profile 2 was used in an effort to resolve the broad enthalpy peaks into discrete events which could then be related to specific chemical reactions. When that effort revealed no new data, selected WIPP samples were re-run using Profile 3. Only the calcium oxalate samples were run using Profile 4 in TGA-MS samples (no DSC data collected).

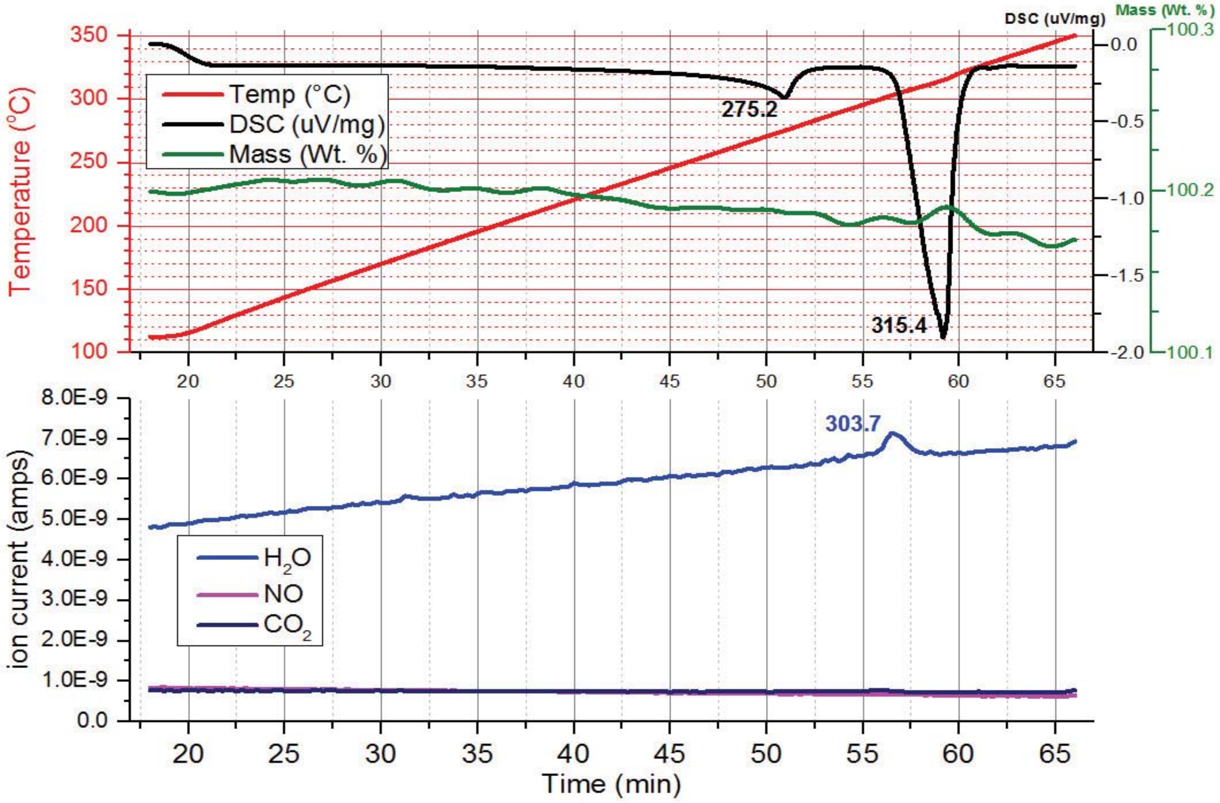
The STA was calibrated using a standard reference material (SRM) set provided by Netzsch (6.223.5-91.2), which consists of the materials listed in Table 4. These materials were used to construct calibration curves for the measurement of temperature and enthalpy using the TGA-DSC measurement head. K-values, the ratio of the reference enthalpy values to those measured in our apparatus ($\Delta H_{ref}/\Delta H_m$), for each standard are also listed in Table 4. A KNO_3 sample (25.53 mg) was run in triplicate (Table 5) to evaluate the precision and accuracy of the onset temperatures and ΔH measurements relative to literature values (Breuer and Eysel, 1982). Onset temperatures vary from known literature values by <1.0%, while the enthalpy measurements (obtained by integrating the peak area using Netzsch Proteus software) vary from the values in Breuer and Eysel (1982) by ~+7% to ~-14%.

Table 6: Summary of XRD phase identification results (major phases in S855793 in bold text), total Pu, volatiles (mass loss after the as-received solid was heated in air for 15 hrs. at 200 ^{0}C), combustibles (additional mass loss after the 200 ^{0}C material was heated in air for an additional 1 hr. at 600 ^{0}C), and calculated non-nitrate combustible content from Martinez et al., 2015; Chamberlin and Martinez, 2014, and Chamberlin, et al 2015.

Sample	**Origin**	**Phases detected by XRD**	**Total Pu (μg/g)**	**Wt. % Volatiles**	**Wt. % Combustible (total)**	**Wt. % Combustible (non-nitrate)**
S802701	S802701	$NaNO_3$	14	11.8 *(LOI to 110 °C)*	n.a.	n.a.
S813389	S813389	$NaNO_3$, $Mg(NO_3)_2$	60	24.5 *(LOI to 110 °C)*	n.a.	n.a.
69139	S864694	amorphous, $Pb(NO_3)_2$, $NaNO_3$	496	18.7	46.1	27.8
68492	S822952	amorphous, $Pb(NO_3)_2$, $NaNO_3$	83	20.6	57.3	38.6
68645	S818449	amorphous, $Pb(NO_3)_2$, $NaNO_3$	96	37.3	27.9	11.6
69173	S851436	amorphous, $PbCO_3$, $Pb(NO_3)_2$, $NaNO_3$	41	14.4	35.3	22.8
69120-1	S855793	**$NaNO_3$, $Pb(NO_3)_2$**, amorphous, $Pb_2(NO_3)_2(C_2O_4)$, PbO_2	394	6.92 *(LOI to 110 °C)*	n.a.	n.a.
69120-2	S855793	**$NaNO_2$, $PbCO_3$**, amorphous, $NaNO_3$, Na_3ONO_2, $MgCO_3$, $Pb(NO_3)_2$, Pb_2OCO_3	33	2.15 *(LOI to 110 °C)*	n.a.	n.a.
69120-3	S855793	**$NaNO_3$, $Pb(NO_3)_2$**, amorphous, PbO_2, $Pb_2(NO_3)_2(C_2O_4)$	262	25.65 *(LOI to 110 °C)*	~32.8 (110-600 °C)	n.a.

The sample outgas is entrained in a ~80 mL/min ultra-high-purity (UHP) Ar flow. Although the TGA furnace and gas inlets were evacuated and flushed with UHP Ar prior to sample loading, some air was admitted to the sample chamber when it was opened to load the sample. At the beginning of each analysis, the Ar content of the headspace was between 10 to 40%, with the balance as dry air. During analysis, Ar content steadily increased to 40-70%. Samples were transferred using a spatula into a clean platinum-rhodium (Pt-Rh) STA pan, placed on the DSC carrier and covered with a pierced Pt-Rh lid. The reference pan was an empty Pt-Rh pan-and-lid assembly.

Figure 1: DSC-MS data for sodium nitrate to 350 °C



Results

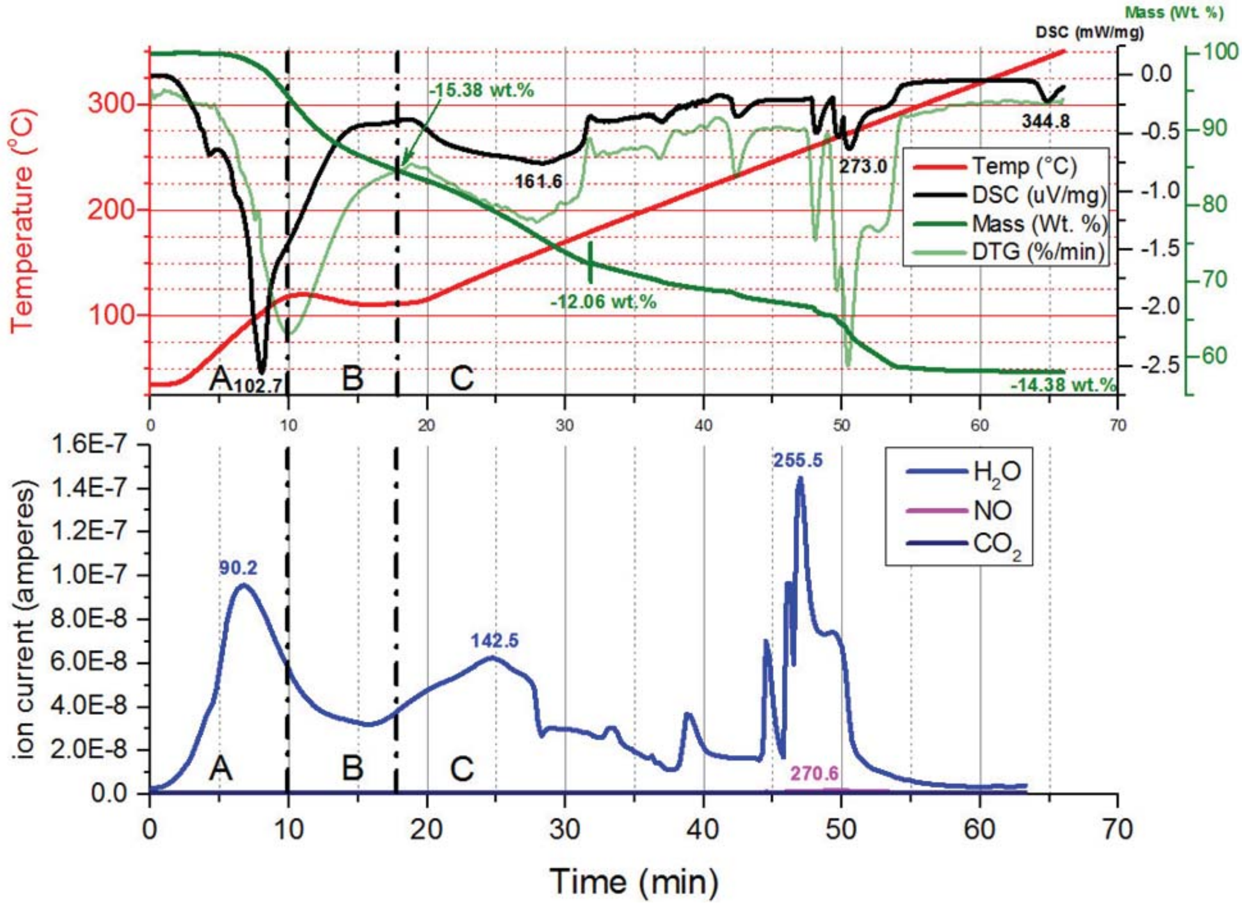
Pure Nitrates and Oxalate

The XRD data reported by Martinez, et al. (2015), Drake (2014), and Chamberlin, et al (2015) indicate that both $Pb(NO_3)_2$ and $NaNO_3$ are major components of the drum residues, along with varying amounts of amorphous material and an array of other constituents, including Pb mixed salts and sodium nitrite(Table 6). In order to evaluate the response of parent drum samples, we analyzed selected nitrates and nitrate hydrates. DSC results from these samples were in broad agreement with those from published work.

Calcium oxalate hydrate samples (0.60 to 11.58 mg) were previously run in 2013 using Profile 4 in TGA-MS mode (i.e., no DSC data). We identified three distinct mass loss events. The first, occurring between 130 and 167 °C is a simple dehydration, resulting in mass losses of -12.0 to 16.2 wt. %. Two separate decarbonation reactions occur at 463.0 to 471.8 °C (CO_2 + CO: mass loss: -18.8 to -25.3 wt. %) and 652.2 to 735.5 °C (CO_2 only: -29.7 to -34.7 wt. %).

All nitrate and nitrate hydrate samples were run in STA-MS mode. One of the primary constituents of the WIPP and LANL drum samples is $Na(NO_3)$ which, as a pure phase, shows no mass change, but did show two significant endothermic reactions (Bauer et al., 2009) below 350 °C: a second order phase transition at 275.2 °C, and melting at 315.4 °C (Figure 1). A small amount of H_2O is evolved during melting. The other major nitrate constituent, $Pb(NO_3)_2$, shows no reactivity or mass change whatsoever below 350 °C, with just minor evolution of NO at ~300 °C.

Figure 2: DSC-MS data from $Mg(NO_3)_2 \cdot 6H_2O$ using Profile 1. Regions A, B, and C correspond to different furnace programming segments (see Table 3).



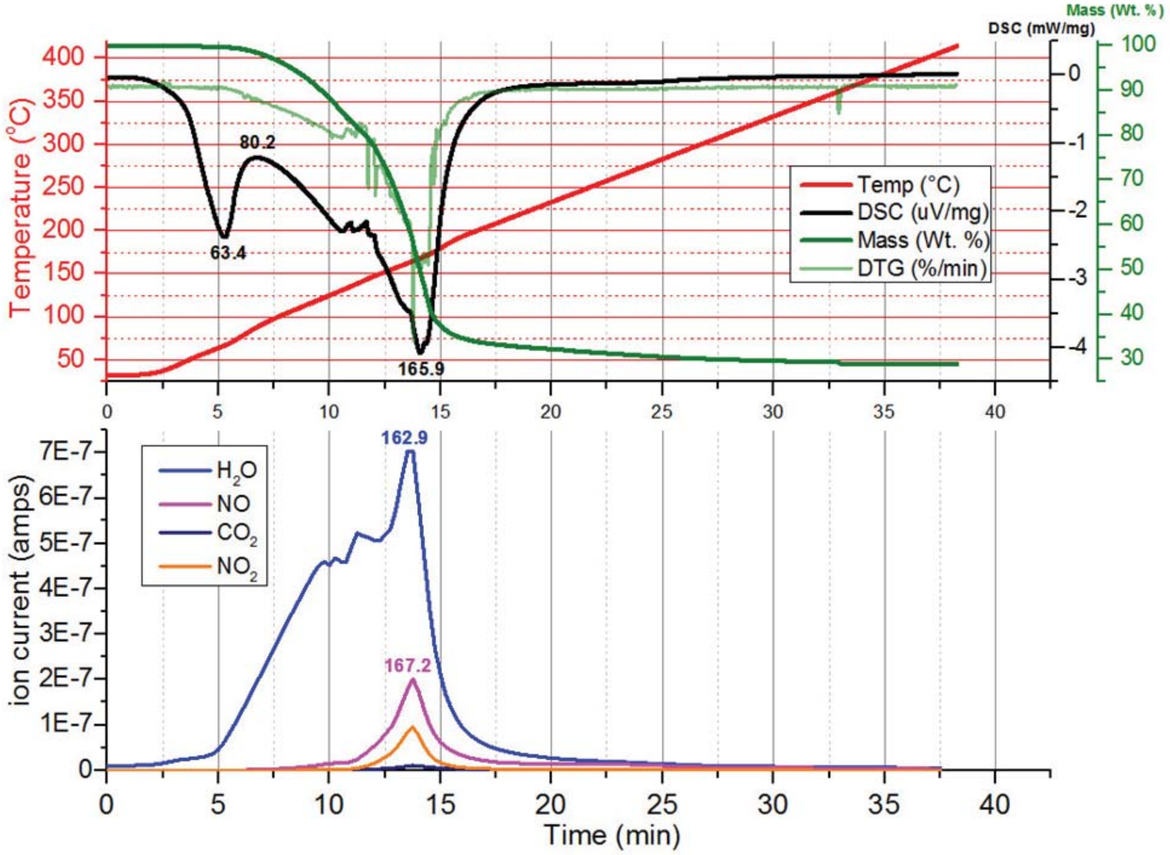
Similarly, $K(NO_3)$, a possible minor constituent of the nitrate salt fraction, shows no mass change and two significant endothermic peaks at 136.8 °C and 341.5 °C, each associated with barely detectable releases of H_2O and CO_2 below 350 °C. Magnesium nitrate hexahydrate, $Mg(NO_3)_2 \cdot 6H_2O$, dehydrates in three distinct stages (Figure 2) with onset temperatures of 42.5, 112.8 and 259.9 °C. Water derived from these endothermic dehydration reactions dominates the gas releases up to 350 °C. The total mass change was -26.4 wt. %.

The only trivalent nitrate salt examined as part of our preliminary study was ferric nitrate hydrate, $Fe(NO_3)_3 \cdot 9H_2O$. Iron (III) nitrate hydrate released significant H_2O and NO_x during the initial part of the isothermal drying step, and therefore was re-run in STA-MS mode using Profile 4 to 400 °C, without the isothermal drying step. Also thought to be a constituent of the nitrate fraction in both the LANL and WIPP drums, iron (III) nitrate was also the only compound to react to a different end-product and the only one that released significant amounts of H_2O and NO_x below 350 °C (Figure 3).

Results from $Fe(NO_3)_3 \cdot 9H_2O$ are similar to those obtained by previous workers (ElMasry et al, 1998; Wieczorek-Ciurowa and Kozak, 1999). When heated to 500 °C at 10 °C/min, dehydration onsets at ~46 °C, while the release of NO begins at ~110 °C. The H_2O peak reaches a maximum value between ~140 and 165 °C. The emission of NO, NO_2, and CO_2 reach a maximum simultaneously, at ~162-168 °C.

Dehydration and denitrification appear to be largely endothermic in nature, though within the overall endothermic trend one or several smaller exothermic peaks appear at ~79-88 °C, ~135-143 °C, and ~163 °C. The endotherm peaks at ~167 °C, almost simultaneously with the maximum NO / NO_2 / CO_2 gas release. The total mass lost up to 400 °C was ~71 to 76 wt. %. In each case, an insoluble reddish-brown to black residue remained, presumably comprised of Fe oxides or oxyhydroxides (e.g., Gadalla and Yu, 1990; Wieczorek-Ciurowa and Kozak, 1999).

Figure 3: DSC-MS data (to 410 °C) from Fe(III) nitrate hydrate.

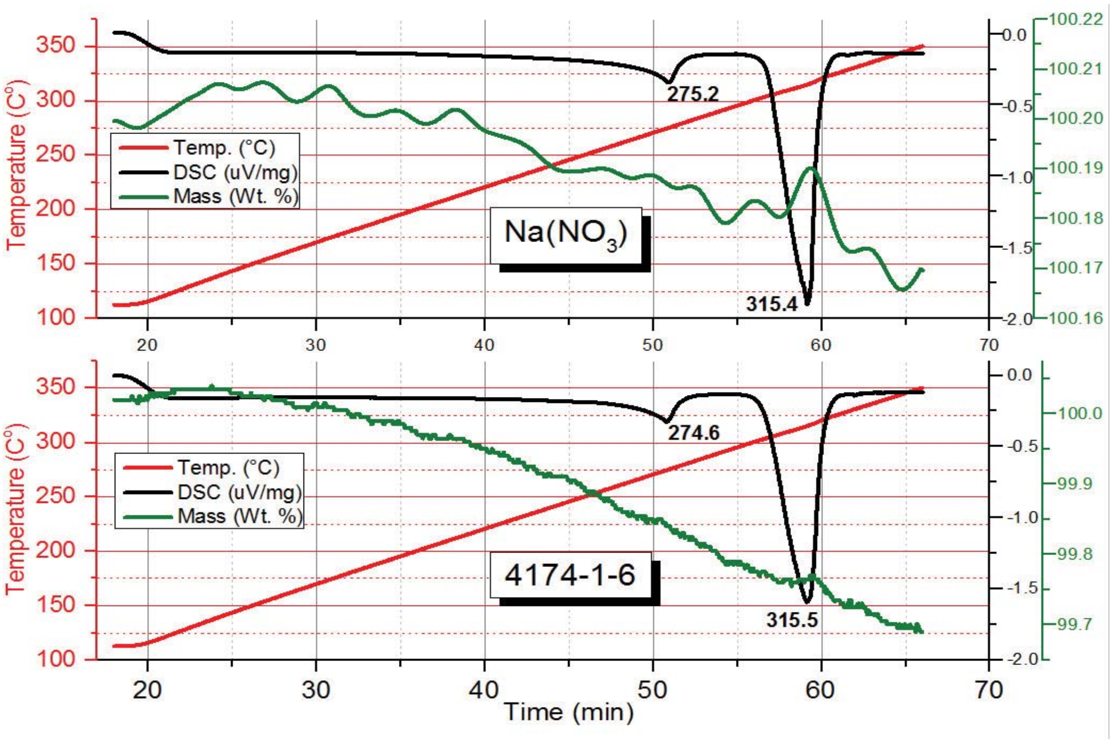


LANL Parent Drum Samples

Top Layer Salts

When analyzed by DSC-MS using Profile 1 (Figure 4), the salt sample from the top layer of S802701 behaved identically to pure Na(NO_3). This sample was not analyzed further. A similar sample, S813389, which contained greater amounts of $Mg(NO_3)_2 \cdot 6H_2O$ (Drake 2014), was analyzed using Profile 4 (Figure 5). DSC-MS data from S813389 do not resemble those from S802701, despite its Na content (~8.5 wt. %). The thermal behavior of this sample somewhat resembles that of Mg nitrate hexahydrate, except that dehydration is complete in a single event. S813389 loses mass in 3 discrete steps: endothermic dehydration (-7.15 wt. %), which onsets at 92.0 °C and peaks at 111.1 °C (though H_2O emission reaches a maximum at 195.5 °C); incipient de-nitrification (-3.72 wt. %) from 316–394 °C, and denitrification (-25.19 wt. %) which starts at ~395 °C and continues to the end of the run. The second de-nitrification reaction was not complete when the run was terminated at 500 °C.

Figure 4: Comparison of DSC data from pure reagent grade Na(NO_3) [top], and Sample S802701 [bottom].



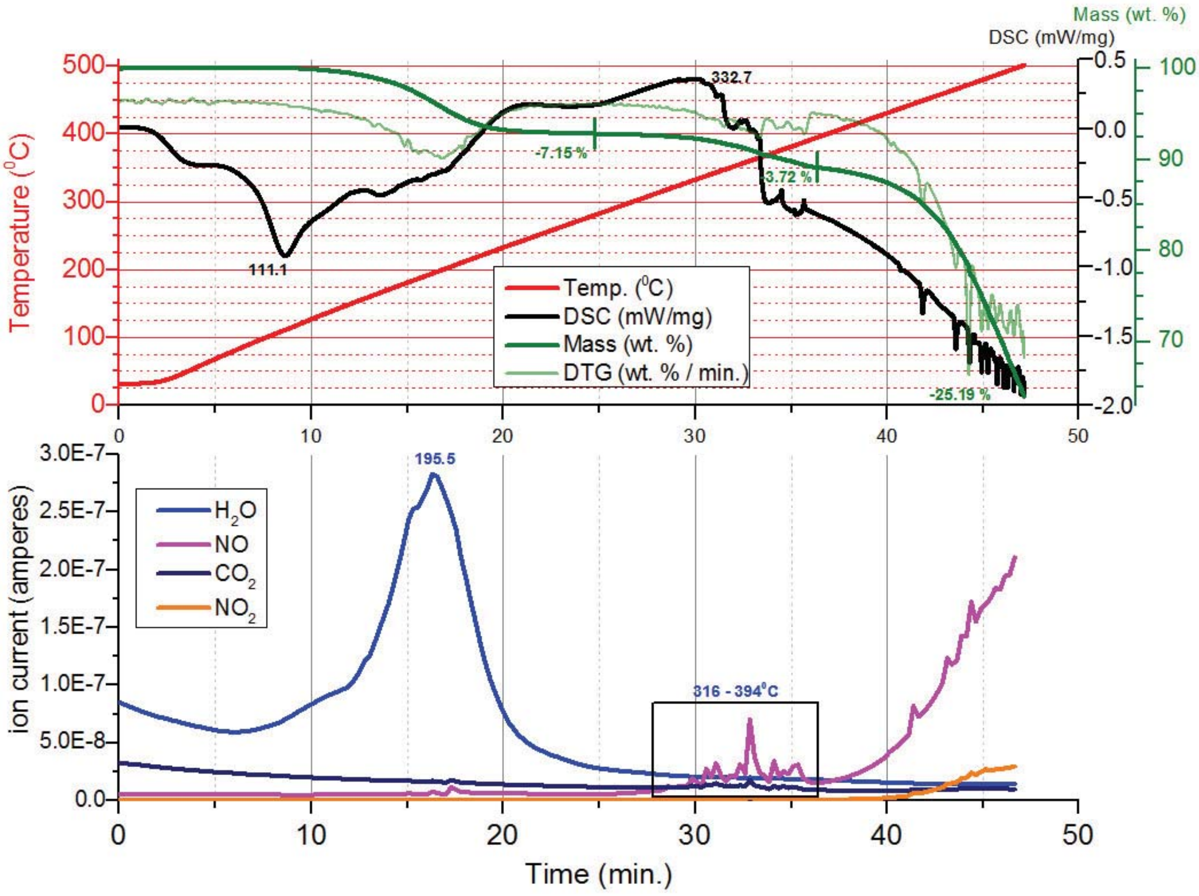
Residual Salts

68492 – S822592: Analyzed using Profile 3 to 500 °C, this sample (Figure 6) lost 43.45 wt. % in two overlapping stages: -27.42 wt. % from 100 °C to 304.2 °C and an additional -16.03 wt. % from 304.2 °C to 500 °C. Both mass loss events were accompanied by gas releases and overlapping exothermic reactions. The first reaction onsets between 217.0 and 227.0 °C and quickly rises to an energy peak at 254.6 °C. This reaction corresponds to a dehydration event with concomitant lesser and approximately equal releases of NO and CO_2. The second reaction maximized at 342.6 °C and was accompanied by further release of CO_2 and NO, with little or no further dehydration. As the run ended, beginning at ~430 °C, a third broadly exothermic reaction initiates and is associated with further CO_2 release. An interesting characteristic of this sample is the small endothermic peak at 131.2 °C, likely a second-order phase transition in one of the solid constituents.

68645 – S818449: Between 100 °C and 500 °C, this sample (Figure 7) lost a total of 46.51 wt. % in three overlapping stages: -9.31 wt. % from 100 °C to 248.2 °C, -13.43 wt. % from 248.2 °C to 321.2 °C, and an additional 23.77 wt. % from 321.2 °C to 500 °C. Each of these mass losses bound corresponding, overlapping exothermic reactions and gas release events. Onset of the first reaction occurs between 160.4 and 174.4 °C and reaches a maximum at ~216.3 °C. The product of this reaction is primarily H_2O vapor. The second reaction onsets at ~240.2 °C, peaks at 305.0 °C, and releases both NO and CO_2. The H_2O from the previous reaction is still being released at this time. The third reaction onsets at ~348.0 °C and rises quite rapidly to a maximum at 365.5 °C. This reaction generates substantial amounts of NO and CO_2. A final dehydration produces a water peak at 377.8 °C, with little impact on the mass change curve.

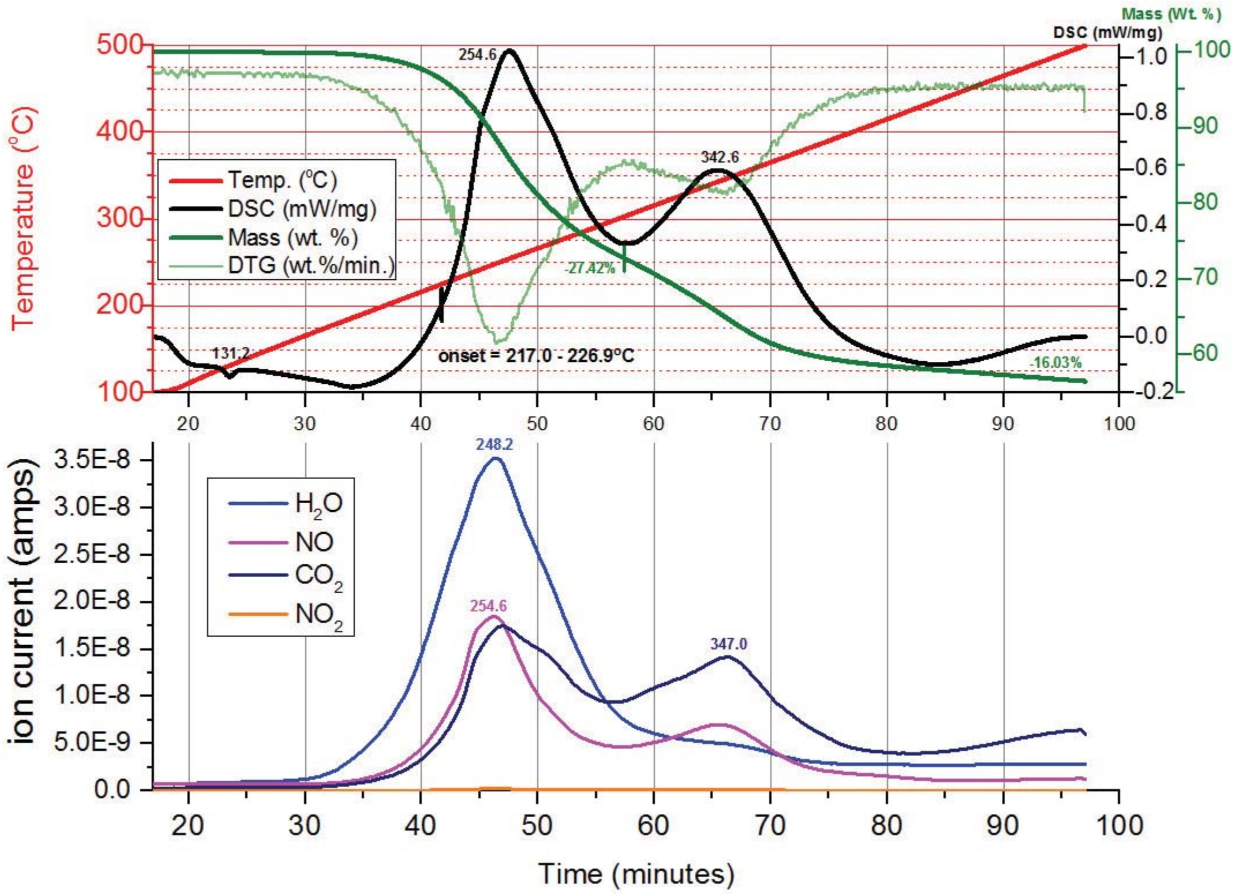
69173 – S851436: Analyzed using Profile 3 to 500 °C, this sample (Figure 8) lost 44.46 wt. % in two stages: -19.78 wt. % from 100 °C to 271.7 °C and an additional -24.68 wt. % from 271.7 °C to 500 °C. The first mass loss was accompanied by a very large H_2O peak (255.1 °C), with subordinate and simultaneous releases of NO and CO_2. A significant exotherm (onset temperature = 204.8 °C; peak @ 257.7 °C) occurs simultaneously with the de-gassing reaction. Subsequent mass loss is more or less continuous. Small CO_2 emissions occur at 304.4 °C and 396.2 °C, and coincide with minor endothermic peaks at 302.4 °C and 399.2 °C. Both also coincide with small mass loss events, as indicated by the DTG curve in Figure 8.

Figure 5: DSC-MS data from the top layer salt sample S813389.



<u>69139 – S864694</u>: Between 100 and 500 °C, this sample (Figure 9) lost a total of 34.56 wt. % in four identifiable stages: -13.33 wt. % from 100 to 280.1 °C, -10.38 wt. % from 280.1 to 336.6 °C, -7.99 wt. % between 336.6 to 473.9 °C and an additional 2.86 wt. % from 473.9 °C to the termination of the run at 500 °C. Each of these mass losses bound corresponding, overlapping exothermic reactions and gas release events. Onset of the first and most exothermic reaction occurs between 223.6 and 234.1 °C and reaches a maximum at 253.2 °C. The product of this reaction is primarily H_2O vapor, with lesser amounts of NO and CO_2. The second reaction onsets at ~293.6 °C, peaks at 309.8 °C and releases mostly CO_2 with lesser amounts of NO and little or no H_2O. The third reaction shows a broad exotherm at ~348.1 °C with coeval releases of CO_2 and NO. The mass loss between 473.9 °C to the termination of the run at 500 °C doesn't correspond to a reaction, but is accompanied by NO emission that peaks at 473.9 °C.

Figure 6: DSC-MS data (to 500 °C) for residual salts from LANL parent drum S822952, sample 68492.

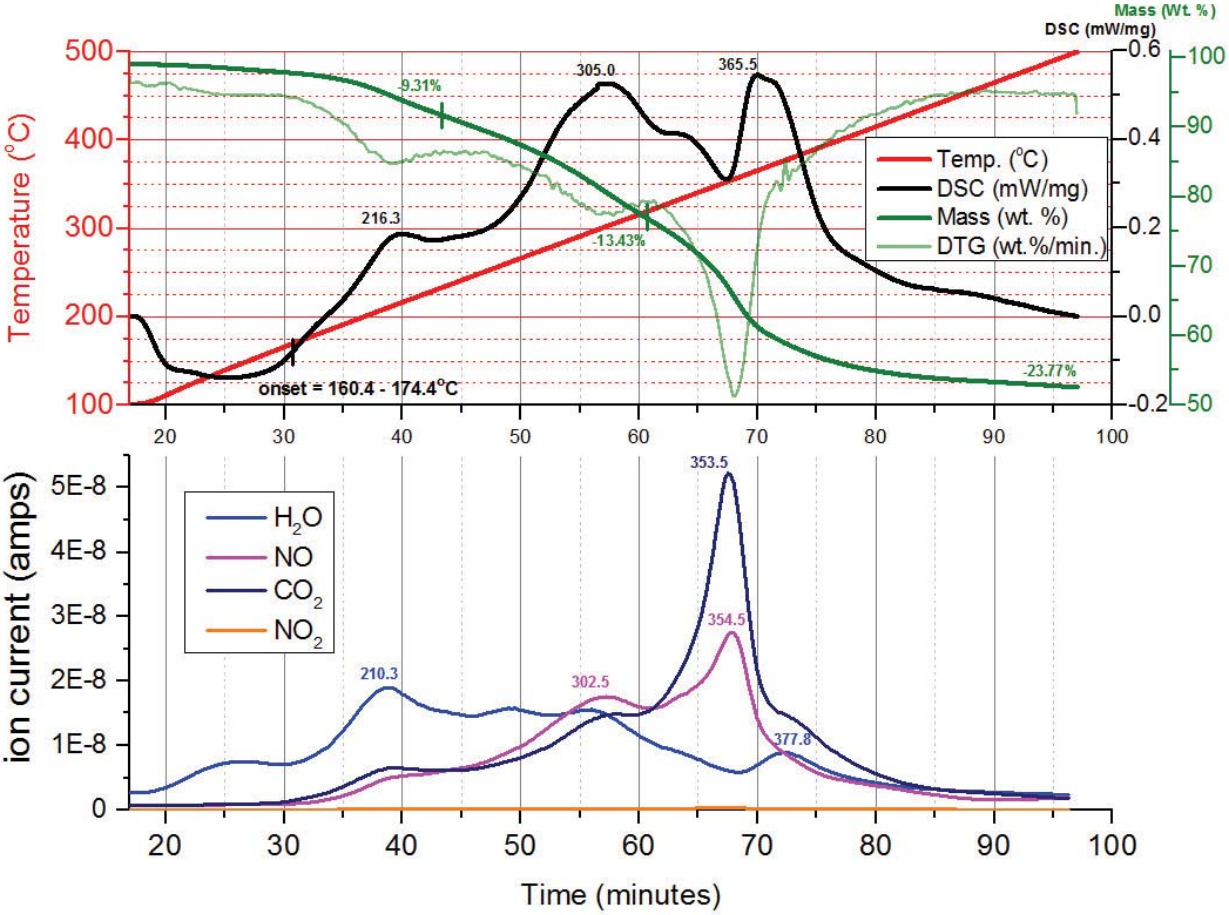


Monolith Samples from LANL Parent Drum S855793

Each of these samples is different, macroscopically (Figure 10), and each has a different mineralogy as shown by X-Ray diffraction results (Martinez, et al., 2015; and Table 6). We performed a preliminary analysis on each sample using Profile 1 (Table 3), and then each sample was re-analyzed using Profile 4. Results from both analyses are consistent, though only the results to 500 °C (Profile 4) are discussed here. Mass loss data does not include the loss of hygroscopic water sorbed during the interval between sample preparation and analysis. After analysis to 500 °C, the residue was a crusty, red-brown powder that resembles ground nutmeg in appearance. The red-brown color of the residue suggests the presence of either red lead oxide (PbO) or Fe^{3+} oxides and oxyhydroxides.

Monolith Sample 1 – 69120-1: XRD identified four phases in this sample. Of the four, $NaNO_3$ and $Pb(NO_3)_2$ are the most abundant, while smaller quantities of $Pb_2(NO_3)_2(C_2O_4)$ and PbO_2 can be inferred from the relative intensities of the XRD peaks. In addition, XRD analysis indicates that there is a sizable amorphous component in this sample (Martinez, et al., 2015). The DSC results are quite complicated (Figure 11), and the determination of onset temperatures is, in most cases, complicated by overlapping reactions. The data show at least three exothermic peaks at 194.2 °C, 238.3 °C, and 303.6 °C, and at least three endothermic peaks at 150.5 °C, 263.6 °C, 365.9 °C. The last of these endothermic reactions has an onset temperature of ~355.0 °C. A final endotherm onsets at ~451 °C and has not reacted to completion by the end of the run at 500 °C. The mass change curve is similarly complex, with up to 5 distinct mass loss events between 100 °C and 500 °C. The total mass loss from the 38.98 mg sample was -26.11%, significantly less than observed from the other samples.

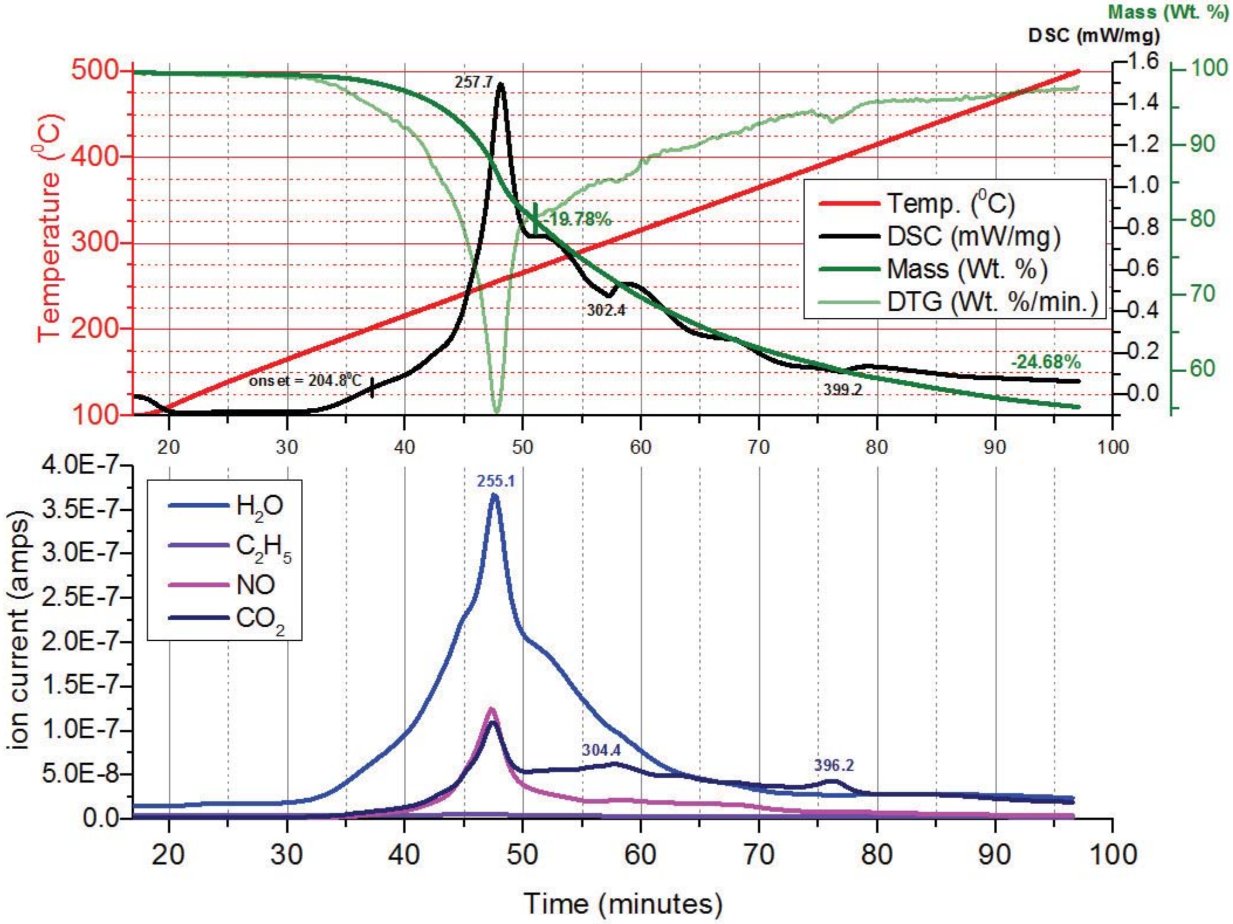
Figure 7: DSC-MS data (to 500 °C) for salt residue LANL parent drum S818449, sample 68645.



The first mass loss, -2.43%, occurs between 100 °C and 262.6 °C and corresponds to dehydration (H_2O peak at 232.3 °C). An endotherm and two exothermic peaks occur in the region, followed by an endotherm that also peaks at 262.6 °C. Between 262.6 °C and 316.3 °C, the sample loses an additional 14.04 wt. %, exothermically (303.6 °C) accompanied by the evolution of a large amount of CO_2, NO, and NO_2. Very little to no water is lost during this step, nor is any evolved during any of the subsequent gas-release events. The next mass loss, 3.64 wt. %, occurs between 316.3 °C and 347.0 °C and corresponds to a shoulder in the DSC curve and a discrete pulse of CO_2 (330.2 °C) and NO (324.7 °C). An additional decarbonation occurs between 347.0 °C and 398.2 °C (CO_2 peak at 363.4 °C) is endothermic (peak at 365.9 °C), and results in a mass loss of 2.17 wt. %. A final mass loss (-3.83 wt. %) corresponds to the endothermic trend which onsets at ~451.0 °C and continues through the end of the run. This reaction is coeval with the release of large amounts of NO and NO_2.

Monolith Sample 2 – 69120-2: Cerussite ($PbCO_3$), sodium nitrite ($NaNO_2$), and amorphous material are the primary constituents of this sample, with minor amounts of $NaNO_3$, $Pb(NO_3)_2$, shannonite ($Pb_2O(CO_3)$), sodium nitrate oxide ($Na_3O(NO_2)$), and magnesite ($Mg(CO_3)$). The DSC-MS results (Figure 12) from this sample are, again, quite complicated and consist of a succession of moderate endo- and exothermic reactions, multiple mass loss steps, and multiple gas evolution events. Over the 100 °C to 500 °C temperature interval, 69120-2 released less gas and lost less mass (total = -19.76 wt. % of 41.28 mg) than 69120-1. Endothermic reactions on the DSC curve (i.e., pointing downwards) correspond to four of the five significant gas releases from the sample; the first onsetting at ~157.3 °C. The mass loss curve shows at least five discrete steps at 201.1 °C, 262.1 °C, 331.1 °C, 380.3 °C, and 452.9 °C.

Figure 8: DSC-MS data (to 500 °C) for residual salt from LANL parent drum S851436, sample 69173



The first mass loss, -1.42 wt. %, occurs between 100 °C and 201.1 °C and corresponds to endothermic (184.3 °C) dehydration (H_2O peak at 184.8 °C). A second mass loss event (-2.90 wt. %) occurs between 201.1 °C and 262.1 °C, and may be related to the first of three decarbonation events, at 233.1 °C. Decarbonation terminates in the vicinity of an exothermic reaction which onsets at 234.6 °C and peaks at 261.6 °C. The largest magnitude mass loss, -6.09 wt. %, occurs between 262.1 °C and 331.1 °C, and corresponds to the endothermic (307.2 °C) release of CO_2 and a minor amount of NO at 305.7 °C. The next mass loss, between 331.1 °C and 380.3 °C corresponds to a third CO_2 release (endothermic reaction onsetting at 340.5 °C and peaking at 347.5 °C), this time not accompanied by significant NO. The last two mass loss events, between 380.3 °C and 452.9 °C, and from 452.9 °C to the end of the run, correspond to NO releases at 432.5 °C, and 495.6 °C. Both of these de-nitrification reactions seem to be related to endotherms that peak at 435.9 °C and 495.6 °C. The latter reaction onset occurs at 467.8 °C.

Figure 9: DSC-MS data (to 500 °C) for residual salt from LANL parent drum S864694, sample 69139

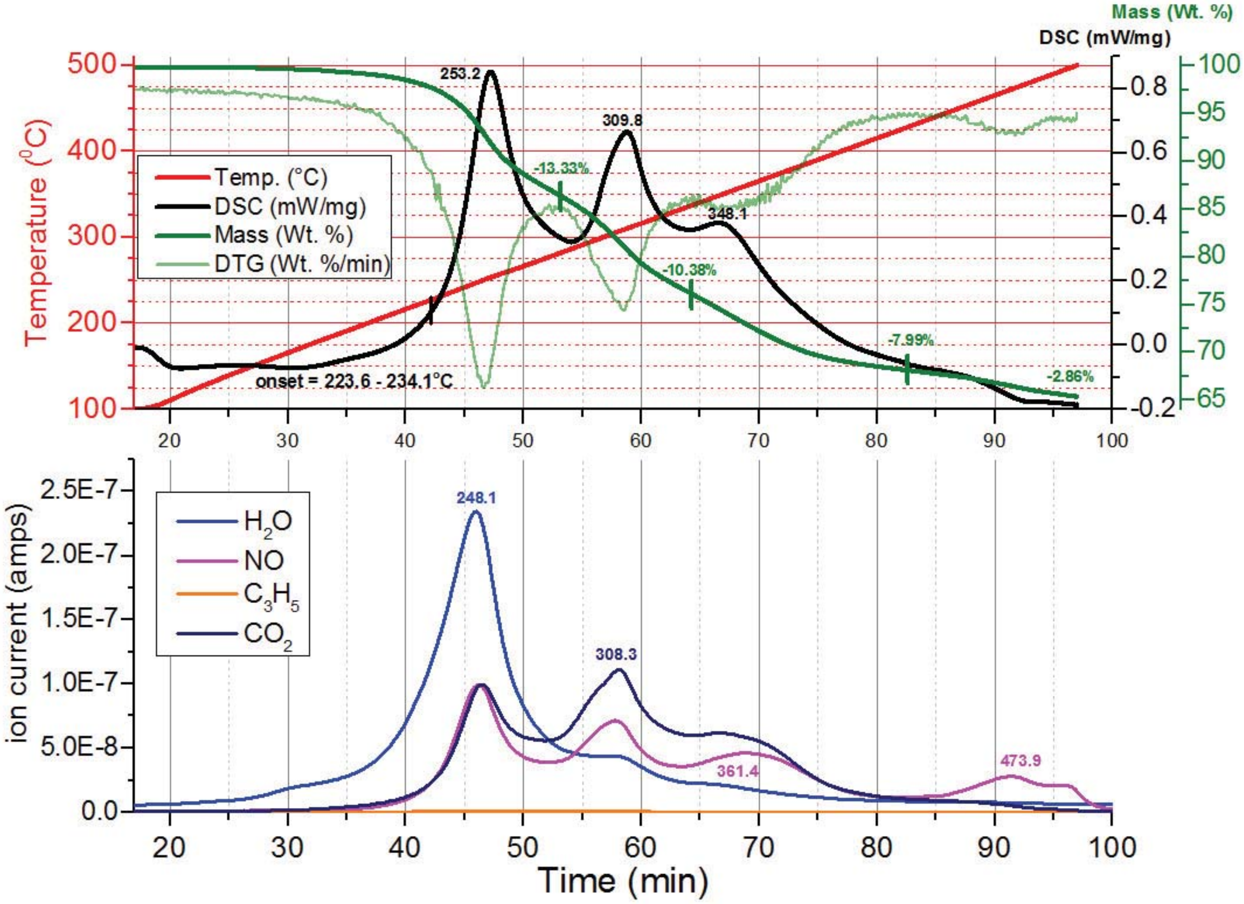


Figure 9: Residual salt monoliths from parent drum S855793, sample 69120. See Table 7 for explanation

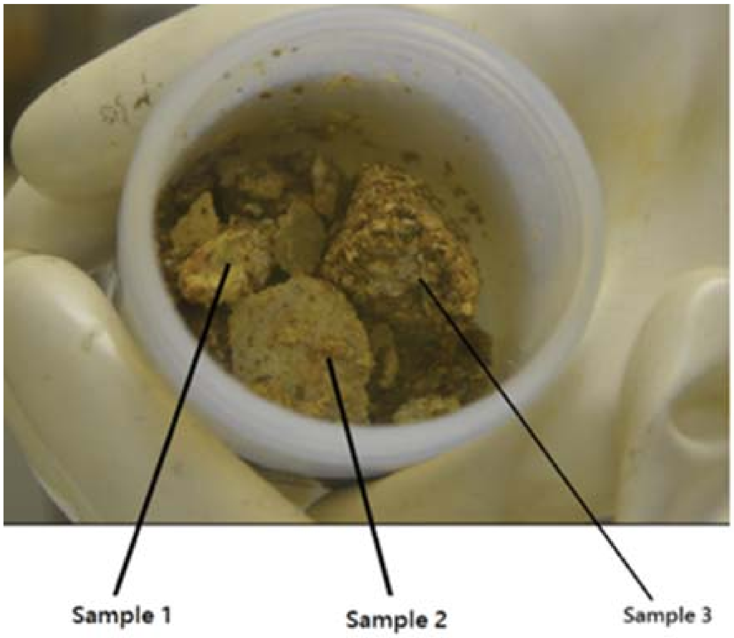
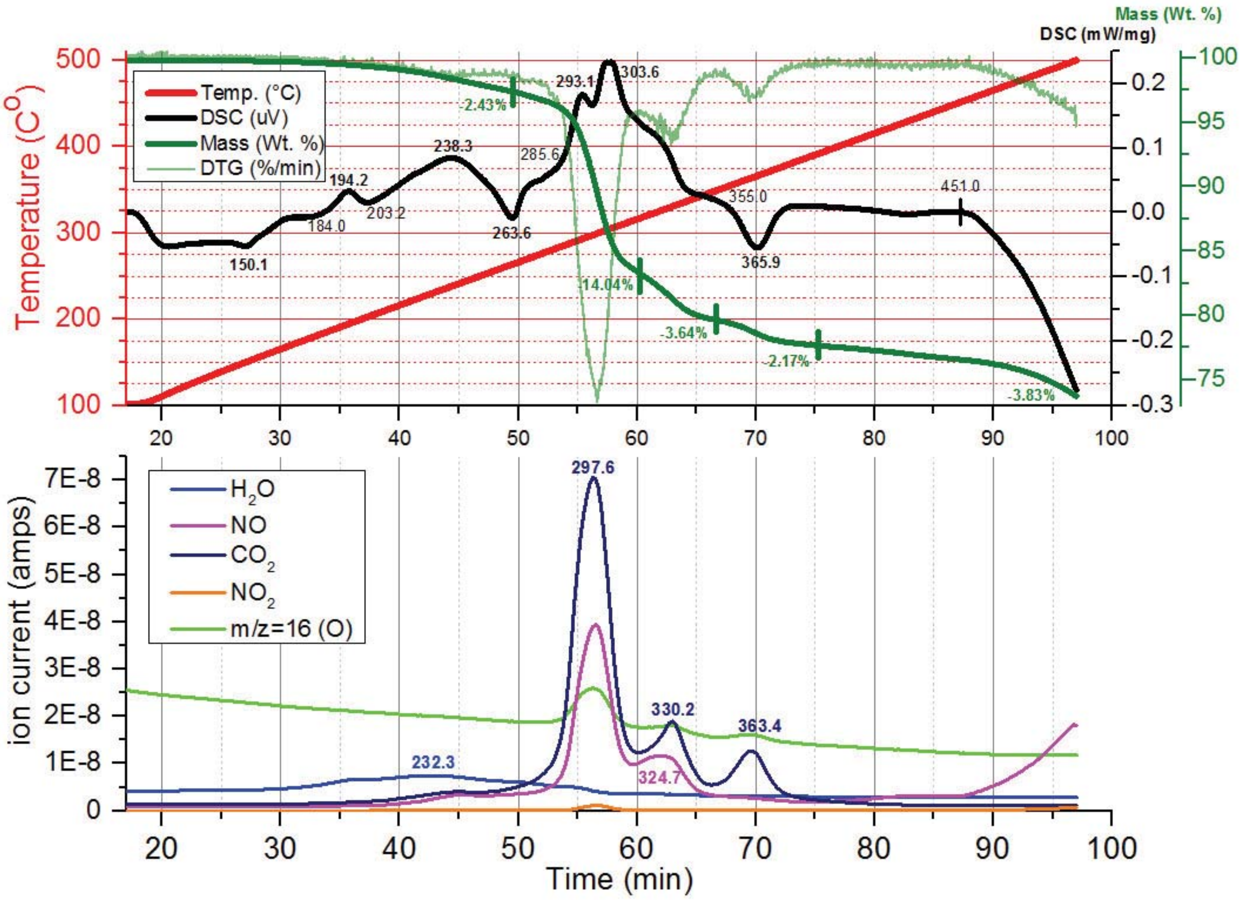
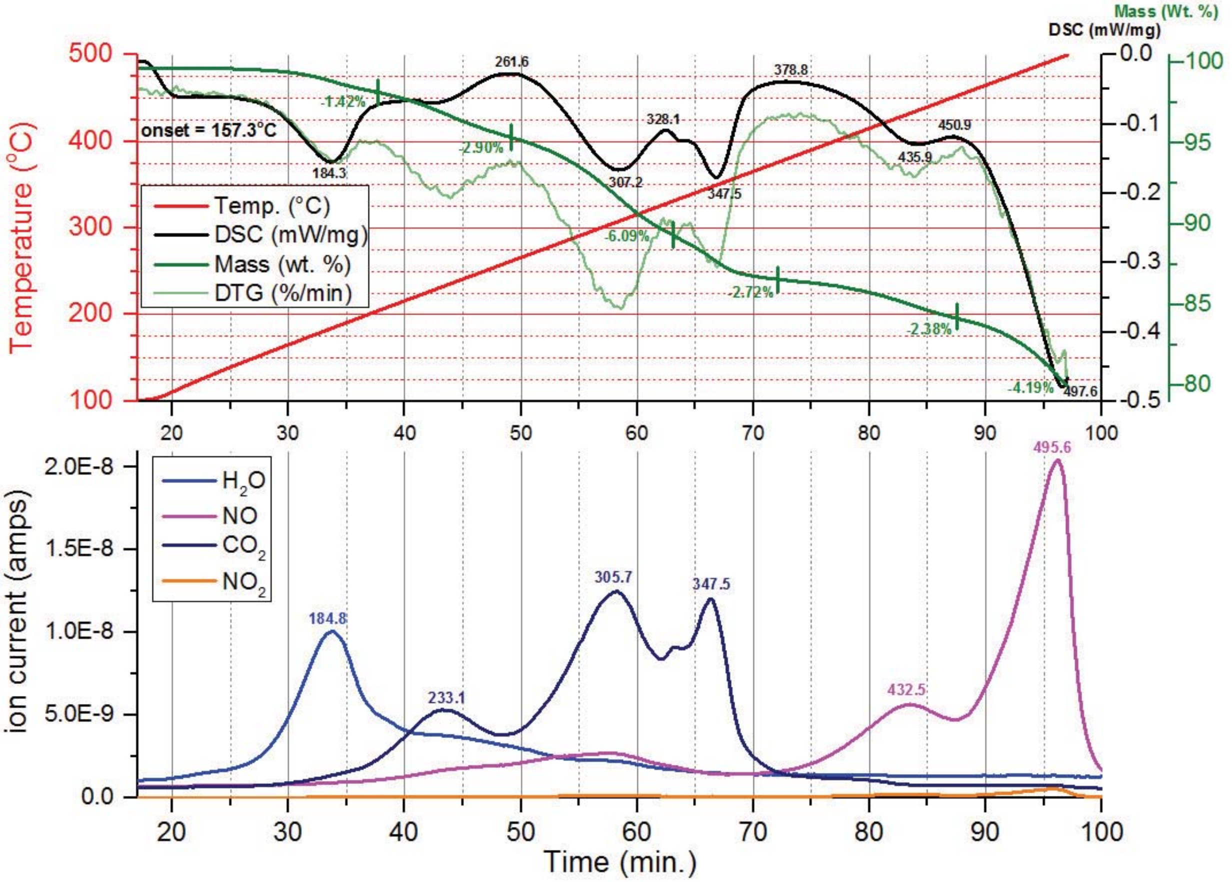


Figure 10: DSC-MS data (to 500 °C) for residual salt from LANL parent drum S855793, sample 69120-1 (#1). Peak temperatures in bold text, onset temperatures are in regular text.



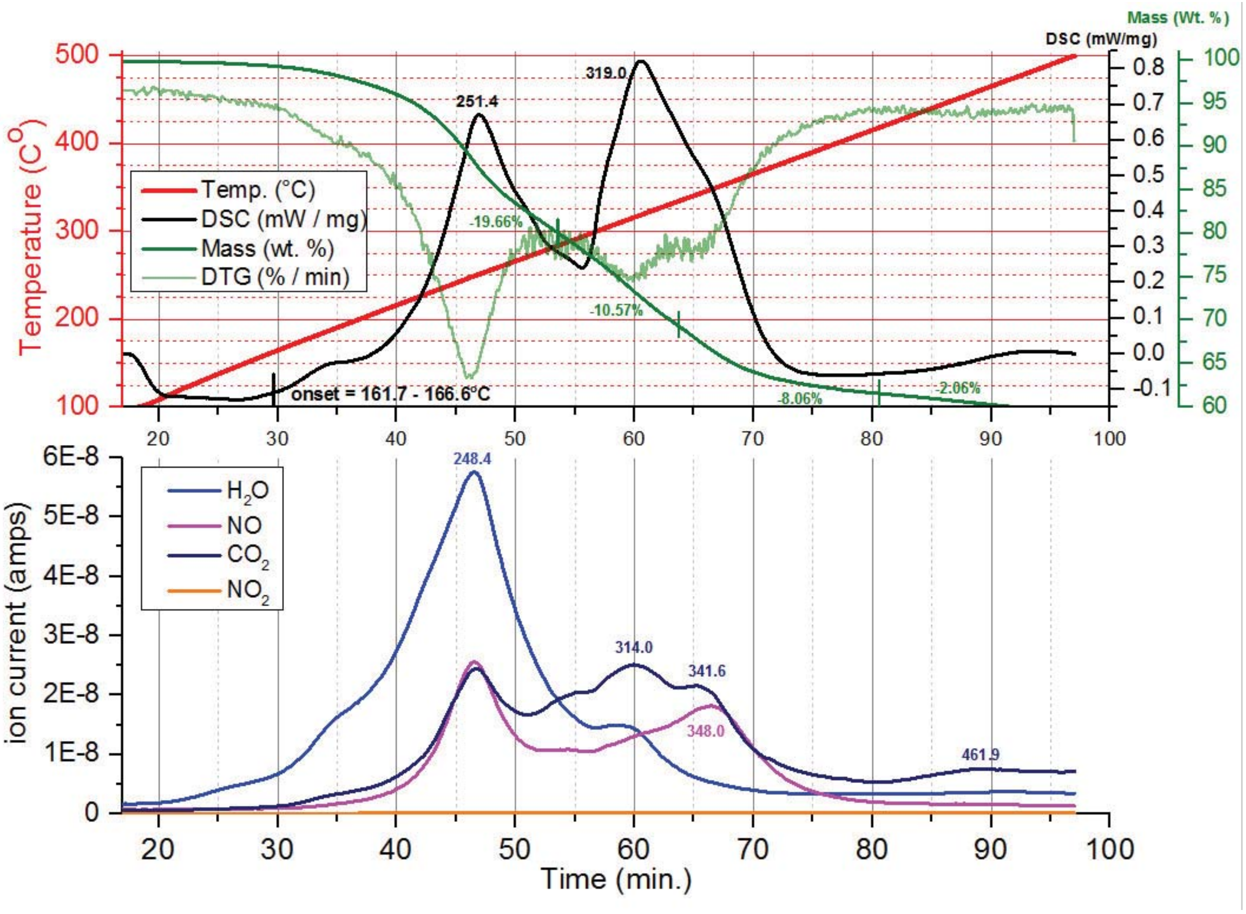
Monolith Sample 3 – 69120-3: XRD results (Martinez et al., 2015 and Table 6) indicate that 69120-3 has a similar mineralogy to 69120-1. However, DSC-MS analysis indicates that this sample's thermal and gas generation behavior between 100 °C to 500 °C is very distinct from that of 69120-1 and 69120-2. The DSC-MS results (Figure 13) from this sample are less complex than observed for 69120-1 and 69120-2, with only two large exothermic energy maxima. The mass loss and gas release spectra, however, are very complex and indicate multiple, coeval mass loss steps and gas evolution events with increasing temperature. From 100 °C to 500 °C, 69120-3 released far more gas and lost the most mass (total = -40.35 wt. % of 29.66 mg) of the three salt residue samples from S855793. Unlike 69120-1 and 69120-2, the two major exothermic reactions on the DSC curve (i.e., pointing upwards) correspond directly to the two major gas release events from the sample. No endothermic reactions were observed over the 100 °C to 500 °C analysis interval. The mass loss curve shows four discrete steps, from 100 °C to 283.3 °C, from 283.3 °C to 333.2 °C, from 333.2 °C to 422.0 °C, and from 422.0 °C to the end of the run.

Figure 11: DSC-MS data (to 500 °C) for residual salt from LANL parent drum S855793, sample 69120-2 (#2)



The first mass loss, between 100 °C to 283.3 °C (-19.66%) corresponds to a highly exothermic reaction which onsets between 161.7 and 166.6 °C, and peaks at 251.4 °C with a simultaneous release of copious amounts of H_2O (248.4 °C), CO_2 and NO / NO_2. The second large exothermic peak onsets at ~296.3 °C and peaks at 319.0 °C, though both the 1st derivative of the mass change curve (DTG on Figure 12) and the gas abundance data suggest that the second peak is comprised of two discrete mass change events. Between 283.3 °C and 333.2 °C, mass changes by -10.57%. The gas released is predominantly CO_2 (maximum at 309.0 °C) with lesser amounts of H_2O. As this reaction winds down, a new reaction occurs resulting in additional mass loss (-8.06 wt. %) between 333.2 °C and 422.0 °C, and liberating additional CO_2 (maximum at 341.6 °C) and NO (maximum at 348.0 °C). This reaction is also exothermic, but produces only a shoulder on the high-temperature side of the peak at 309.0 °C. To the end of the run, at 500.0 °C, further mass loss (-2.06 wt. %) is associated with the release of a small amount of CO_2.

Figure 12: DSC-MS data (to 500 °C) for residual salt from LANL parent drum S855793, sample 69120-3 (#3).



Discussion

Pure Nitrates vs. LANL Top Layer samples

Thermal analyses of pure Na, K, and Pb nitrates and the nitrate hydrates of Mg and Fe^{3+} revealed little in the way of reactions or potentially dangerous gas releases below 350 °C. Lead nitrate showed little reaction of any kind, while anhydrous $K(NO_3)$ and $Na(NO_3)$ undergo mild endothermic reactions which correspond to second order crystallographic transitions, followed by melting. As expected, the hydrous nitrates of Mg and Fe^{3+} respond quite differently. From room temperature to 350 °C, magnesium nitrate hexahydrate (Figure 13) dehydrates endothermically, starting well below 100 °C, in several discrete stages, eventually losing ~42 wt. % of its mass. A trace amount of NO is evolved at ~270.6 °C. Dehydration of $Fe(NO_3)_3{\cdot}9H_2O$ (Figure 2) onsets at ~46 °C, and the sample begins to de-gas NO^+ and NO_2^+ at ~110 °C. The H_2O peak reaches a maximum value between ~140 and 165 °C, while NO and NO_2 emissions reach a maximum simultaneously, at ~162-168 °C. Both reactions are largely endothermic in nature. No further reactions occur above ~200 °C, as the sample has reacted completely to a red-brown residue, likely Fe_2O_3 or a mixture of Fe^{3+} oxides and oxyhydroxides (Gadalla and Yu, 1990; Wieczorek-Ciurowa and Kozak, 1999), with a total mass loss of ~71 to 76 wt. %.

Top layer salts consist largely of Na and Mg nitrates. The DSC-MS data (Table 7) from one of the top layer salt samples, S802701 are identical to those obtained from pure $NaNO_3$ (Figure 3). This sample was completely soluble in H_2O and in 2% HNO_3, and its trace and minor constituents include 10s to >1000 mg/g of K, Fe, Ca, Pb, Mg, Ni, and Cr (Chamberlin and Martinez, 2014; Chamberlin et al, 2015). The radioactive element contents of this sample are relatively low: ^{235}U = 1600 μg/g; ^{238}U = 840 μg/g; and 1 μg/g or less of ^{239}Pu, ^{237}Np, and ^{241}Am (Chamberlin and Martinez, 2014; Chamberlin et al, 2015). Impurities of these elements at these levels appear to have no effect on the sample's thermal characteristics.

The other top layer salt sample, S813389, appears to be a hydrous mixture of Na and Mg nitrates with trace amounts (e.g., 10s to 100s of μg/g) of Al, K, Ca, Fe, and Ni (Chamberlin and Martinez, 2015) but only 9 μg/g of ^{238}U. Investigators noted the presence of brown flecks in the residue remaining after digestion in 2% HNO_3 (Chamberlin and Martinez, 2014; Chamberlin et al, 2015). XRF analyses of the residues revealed measurable quantities of several first-row transition elements, plus Si, Ca, Ba, Ta, W, Pb, and Pu (Chamberlin and Martinez, 2014; Chamberlin et al, 2015; Martinez et al., 2015). Heated to 500 °C (Figure 4) the sample dehydrates, emitting a single large pulse of H_2O (max. temp = 195.5 °C) followed by 'spiky' discharges of NO and NO_2 between 314 and 395 °C, possibly indicative of melting. Above 395 °C, the sample begins to break down completely, emitting copious amounts of gaseous NO and NO_2.

Though it is a mixture of Mg and Na nitrates, DSC-MS data from S813389 resembles neither those of pure $Mg(NO_3)_2{\cdot}6H_2O$ (Figure 2), nor pure $Na(NO_3)$. Like Fe nitrate hydrate, S813389 exhibits a single dehydration event above 100 °C. However, the concomitant losses of NO and NO_2 at low temperature seen in Fe nitrate hydrate are not seen in S813389. Instead, gaseous NO and NO_2 is liberated independently, at much higher temperatures. The energy flow during each of these reactions is largely endothermic. We observed only minor NO evolution from pure Mg nitrate hydrate up to 350 °C (Figure 13). However, the experimental results of Mu and Perlmutter (1982) indicate that anhydrous $Mg(NO_3)_2$ begins to denitrify above ~300 °C. Though there is no data in the literature on the thermal behavior and phase stability of mixed Na and Mg nitrate salts, Reddy et al. (2012) documented the formation of a hybrid potassium nitrate–magnesium nitrate compound, $[2KNO_3{\cdot}Mg(NO_3)_2]$, with unique thermal characteristics. It is possible that analogous compounds may also occur on the $NaNO_3$ - $Mg(NO_3)_2$ join.

Residual Salt Samples

Samples 68645, 68492, 69173, and 69139 were initially assumed to be mixtures of Pb and Na nitrates (Table 6) with possible oxalate content, and little organic content. The DSC-MS data from these samples (Figures 5-8) are characterized by multiple, overlapping dehydration, decarbonation and denitrification reactions; all exothermic in nature. Coeval gas evolution and exothermic energy events correlate to significant mass losses. These reactions may also be quite vigorous: during an early trial run of 68492 one such reaction resulted in a sudden combustion event at 255.7 °C. According to WCRRF personnel, routine operations following waste remediation included the addition of Swheat© to the parent drums, in order to immobilize small amounts of liquids that were left over. Thus, the possibility of contamination of the residual salts with organic components cannot be completely ruled out.

The thermal analysis results presented here are similar to those referenced in Clark and Funk (2015), Britt (2015) and Wilson et al. (2015). In addition, exothermic reactions were accompanied by the release of H_2O, CO_2, NO and NO_2 during the DSC-MS analysis of five of the seven salt residue samples taken from LANL parent drums. Heated to 500 °C, four of the residual salt samples—69120-3, 68492, 69173, and 69139—undergo exothermic reactions that peak between 253 °C and 258 °C (Table 8). Onset temperatures of these reactions vary from 161.7 °C to 223.6 °C, and the dominant species released is H_2O, accompanied by CO_2 and NO. Mass losses during this reaction vary from 13.33 wt. % to 27.42 wt.

%. The dehydration of 68645 (Figure 6) onsets at 160.4-174.4 °C and peaks at 216.3 °C, losing less than 10% of its total mass in the process (Table 7). Three of the residual salt samples (69120-3, 68645 and 69139) undergo a subsequent exothermic reaction that peaks between 305 and 320 °C and releases primarily CO_2, with some coeval NO and H_2O. Mass losses during this reaction vary from 10.38 to 13.43 wt. %. Sample 68492 shows no evidence of a reaction in this temperature region, while 69173 has a small CO_2 gas peak at 304.4 °C which corresponds to an endothermic peak at 302.4 °C. The latter may be indicative of cerussite decarbonation, which is an endothermic reaction known to occur at ~310 °C at low CO_2 pressures (Ball and Casson, 1975; Yamaguchi et al., 1980). Finally, three of the four samples (68492, 68645, 69139) undergo a high temperature reaction between 342 and 366 °C dominated by the release of CO_2, with lesser amounts of NO and little to no H_2O. 68492 and 68645 undergo significant mass loss during this reaction (Table 8), while 69139 loses ~8 wt. %. Minor losses of NO and CO_2, with little or no attendant mass loss, persist above 399 °C in both 69139 and 69173.

The response of residual salts to thermal energy is analogous to combustion reactions involving an organic component and a nitrate-based oxidizer in air (e.g., Wu and Zavarin, 1986; Biteau, 2009). Possible exothermic reactions could include the dehydration and oxidation of the organic constituent (starch), as well as the combustion of the organics via interaction with nitrate. The complete oxidation and breakdown of the organic component is sustained by ambient atmospheric O_2, as well as by O_2 derived from the breakdown of the nitrates (Biteau, 2009):

$$C_6H_{10}0_5 \cdot H_20 + KN0_3 \quad \rightarrow \quad 6\,C0_2 + 6\,H_20 + K_20 + N_2$$

$$C_6H_{10}0_5 \cdot H_20 + 6\,(0_2 + 3.76\,N_2) \quad \rightarrow \quad 6\,C0_2 + 6\,H_20 + 22.56\,N_2$$

The quadrupole MS cannot resolve CO^+ from N_2^+, thus it is impossible to tell if CO^+ is being produced by the incomplete oxidation via reactions analogous to:

$$C_6H_{10}0_5 \cdot H_20 + 12/5(KN0_3) \quad \rightarrow \quad 6\,C0 + 6\,H_20 + 6/5(K_20) + 6/5(N_2)$$

The above exothermic reactions, from Biteau (2009), involve only $K(NO_3)$. However, a different set of reactions, with different onset temperatures, products, and energy releases may occur when mixed nitrate salts of Na, Al, Pb, Fe, etc. are involved.

It is difficult to infer the amount of energy released during the reactions observed in the DSC-MS, though approximations using background values assumed *ad hoc* that bracket the exotherms suggest that the total amount of thermal energy generated is between 1000 and 1500 J/g. Previous studies indicate that thermally-activated exothermic reactions between nitrate salts and various organic materials including cellulose (Wu and Zavarin, 1986; Pourmortazavi et al., 2009), lactose / starch mixtures (Biteau, 2009), and cloth (Hartman et al., 2007; Scheele et al., 2005) typically onset between 150 and 200 °C and peak in the 200 – 350 °C range. The onset and peak temperatures appear to vary as a function of experimental heating rate (Pourmortazavi et al., 2009), oxidizer (nitrate)-to-fuel (organic) mass ratio (Scheele et al., 2007), and the age of the reacting material (Scheele et al., 2005).

Wu and Zavarin (1986) also found a positive correlation between the pH of the nitrate solution used to treat lignocellulose and the onset temperature of the oxidation exotherm. Wu and Zavarin (1986) and Hartman et al. (2007) also observed that reactions with nitrates of III and IV ions such as Fe^{3+} and Ce^{4+} led to markedly lower onset temperatures. Relatively few of these studies also analyzed the gases produced by these reactions, though Scheele et al. (2007) observed that non-explosive gases such as

CO_2 and H_2O were most abundant. Using FTIR spectroscopy, Biteau (2009) identified CO, aldehydes (CH_2O and $C_2H_4O_2$), formic acid (CH_2O_2), and low levels of N_2O and NO_2, in addition to H_2O and CO_2. Some of these gases persisted to higher temperatures, and N_2O appeared only above 400 °C. Re-analysis of the salt residues and the monolith samples for additional gas constituents (C_2H_3, C_2H_5, C_3H_5: M/z = 27, 29, and 41) revealed only trace releases at these M/z ratios coeval with the major gas release events.

Table 7: Summary of DSC-MS data from 100 °C to 500 °C. Endothermic reactions denoted by blue text.

Sample	Mass Change (wt. %)	**DSC** (onset/**peak °C: heat transfer**)	**Gas Peaks (°C; species; mass change)**
Top Layer Salts			
S802701	-0.3	Peak @ 274.6: endothermic	No corresponding peak
		Peak @315.5: endothermic	No corresponding peak
S813389	-36.06	n.a. / **111.1**: endothermic	No corresponding peak
		No peak	195.5; H_2O (-7.15 wt. %)
		n.a. / >395: endothermic	>500; NO, NO2 (-25.19 wt.%)
Residual Salts			
68492	-41.45	217.0-226.9 / **254.6**: exothermic	~250: H_2O>NO=CO_2 (-27.42 wt.%)
		n.a. / **342.6**: exothermic	347.0: CO_2>NO (-16.03 wt.%)
68645	-46.51	160.4-174.4 / **216.3**: exothermic	210.3: H_2O>NO=CO_2 (-9.31 wt.%)
		240.2 / **305.0**: exothermic	302.5: NO=CO_2=H_2O (-13.43 wt.%)
		348.0 / **365.5**: exothermic	~354: CO_2>>NO (-23.77 wt.%)
69173	-44.46	204.8 **/ 257.7**: exothermic	255.1: H_2O>>CO_2=NO (-19.78 wt.%)
		2 small endo. peaks @ 302.4, 399.2	Small CO_2 peaks @ 304.4, 396.2
69139	-34.56	223.6-234.1 / **253.2**: exothermic	248.1: H_2O>NO=CO_2 (-13.33 wt.%)
		296.3 / **309.8**: exothermic	308.3: CO_2>NO>H_2O (-10.38 wt.%)
		n.a. / **348.1**: exothermic	361.4: NO=CO_2 (-7.99 wt. %)
		No peak	473.9: NO only (-2.86 wt.%)
Monolith Samples from drum S855793			
69120-1	-26.11	184.0 / **194.2**: exothermic	No gas, no mass change
		203.2 / **238.3**: exothermic	232.3: H_2O (-2.43 wt.%)
		285.6 / **293.1-303.6**: exothermic	297.6: CO_2>NO, no H_2O (-14.04 wt.%)
		Shoulder in previous peak	325-330: CO_2>NO, no H_2O (-3.64 wt.%)
		355.0 / **365.9**: endothermic	363.4: CO_2 only (-2.17 wt.%)
		~451.0 / **>500**: endothermic	>500: NO only (-3.83 wt.%)
69120-2	-19.76	157.3 / **184.3**: endothermic	184.8: H_2O only (-1.42 wt.%)
		No peak	233.1: CO_2 only (-2.90 wt.%)
		234.6 / **261.6**: exothermic	No gas
		n.a. / **307.2**: endothermic	305.7: CO_2>NO, no H_2O (-6.09 wt.%)
		n.a. / **328.1**: exothermic	No gas
		340.5 / **347.5**: endothermic	347.5: CO_2 only (-2.72 wt.%)
		n.a. / **378.8**: exothermic	No gas
		n.a. / **435.9**: endothermic	432.5: NO only (-2.38 wt.%)
		467.8 / **497.6**: endothermic	495.6: NO only (-4.19 wt.%)
69120-3	-40.35	161.7-166.6 / **251.4**: exothermic	248.4: H_2O>NO=CO_2 (-19.66 wt.%)
		296.3 / **319.0**: exothermic	314.0: CO_2>NO (-10.57 wt.%)
		Shoulder in previous peak	348.0: NO=CO_2 (-8.06 wt.%)
		Slight rise <500: exothermic	461.9: CO_2 only (-2.06 wt.%)

Finally, onset temperatures may also vary with salt composition, as different combinations of nitrate salts will have eutectic compositions that liquefy well below the melting point of the pure end-members. Chemical analyses (Chamberlin and Martinez, 2015) indicate that the residual salts are primarily mixtures of Pb and Na nitrates, with smaller amounts of Mg, Al, and Ca nitrates (Table 8).

Table 8: Nitrate composition and exothermic onset temperatures for the four residual salt samples.

sample	Pb (Wt. %)	Na (Wt. %)	Mg (Wt. %)	Al (Wt. %)	Ca (Wt. %)	Onset (C°)
68492	21.7	2.73	0.18	0.33	0.26	217.0 - 226.9
68645	18.9	1.61	3.28	2.12	0.27	160.4 - 174.4
69173	42.0	1.39	0.74	0.74	0.16	204.8
69139	24.2	3.72	0.21	0.60	0.25	223.6 - 234.1

The sample with the lowest onset temperature, 68645, also has the most diverse nitrate salt compositions, with >1.0 wt. % each of Na, Mg, and Al nitrates and nitrate hydrates, and a lower Pb nitrate content. The melting points of Pb, Na, Mg, Al, and Ca nitrates (or nitrate hydrates) are, in °C: 270 (Pb), 308 (Na), 88.9 (Mg nitrate hexahydrate) or 129 (Mg nitrate dihydrate), 73.9 (Al nitrate nonahydrate), and 42.7 °C (Ca nitrate tetrahydrate), respectively. Though data on the eutectic compositions for the various binary, tertiary, and higher-order mixtures of these nitrate salts is lacking in the scientific literature, it seems likely that higher contents of the hydrated forms of the divalent and trivalent nitrate salts would tend to lead to lower eutectic temperatures. Nitrate salts, liquefied at lower temperatures, may then react more readily with the organic components in the residual salts. This could also lead to lower onset temperatures for the exothermic oxidation of the organics by nitrates.

In summary, the thermal behavior of the four residual salt samples (68645, 68492, 69173, 69139) is broadly similar, characterized by exothermic dehydration / decarbonation / denitrification reactions having onset temperatures between 160 and 235 °C. Subsequent higher temperature exothermic reactions have onset temperatures between 240 and 350 °C, and release primarily CO_2 and NO_x.

Residual Salt Monoliths from Parent Drum S855793

A third set of samples (69120-1, 69120-2, and 69120-3, see also Figure 10) has a much more diverse mineralogy than the other salt residue samples (Martinez et al, 2015). The DSC-MS data from these samples are also very complex. Sample 69120-1, though primarily composed of a mixture of Na and Pb nitrates, also contains lead nitrate oxalate, $Pb_2(NO_3)_2(C_2O_4)$ and lead oxide, PbO_2. Results from previous studies on the thermal behavior of the constituent compounds identified by XRF are summarized in Table 8. Results of the DSC-MS analysis reveal a complex succession of moderate endothermic and exothermic reactions an order of magnitude less vigorous than those observed during the analysis of the residual salts. Gas release is strongly dominated by CO_2 emission, with lesser amounts of NO and almost no H_2O above ~300 °C.

Thermal analysis of lead nitrate oxalate hydrate, (Boudaren et al., 2001) shows that—after dehydration below 150 °C—the nitrate oxalate breaks down into three phases: lead nitrate $Pb(NO_3)_2$, anhydrous lead oxalate PbC_2O_4, and amorphous, anhydrous $Pb_2(NO_3)_2(C_2O_4)$. Boudaren et al. (2001) postulate that the crystallization of anhydrous $Pb_2(NO_3)_2(C_2O_4)$ occurs between 162 °C and 180 °C, as evidenced by a small exothermic peak with no attendant mass change in this region. In sample 69120-1, a similar transformation could be responsible for a small exothermic peak which onsets at 182.0 °C and peaks at 194.2 °C. $Pb_2(NO_3)_2(C_2O_4)$ then breaks down below 200 °C into $Pb(NO_3)_2$ and PbC_2O_4, both of which, in turn, break down rapidly between ~260 °C and 280 °C, resulting in a significant mass loss. Significant mass losses occur in both monolith samples 69120-1 and 69102-2 over the same temperature range. Boudaren et al. (2001) further speculate that an exothermic peak at 315–320 °C accompanying the breakdown of lead oxalate into Pb_2OCO_3 may instead result from the oxidation of CO emitted by the sample. Both Pb nitrate and oxalate break down into ever more complex mixed oxynitrates and oxycarbonates which then break down into various Pb oxides (Boudaren et al., 2001). The intermediate compound, $Pb_6O_5(NO_3)_2$, for example, persists to ~335 °C, yielding a mixture Pb_2O_3 and PbO.

Table 9: Thermal properties of the mineral constituents of salt residues from parent drum S855793 (samples 69120-1, 69120-2, and 69120-3).

Constituent	**T_{melt} (C°)**	**T_{boil} (C°)**	**Characteristics**
$NaNO_3$	308	380	Melts, then decomposes to $NaNO_2$, releasing O_2
$NaNO_2$	271	--	Decomposes in air above 320 °C: $2NaNO_2 \rightarrow Na_2O + NO + NO_2$
Na_3ONO_2	--	--	No data – extremely hygroscopic, breaks down to $NaNO_3$, NaOH, and/or $Na_2(CO_3)$ upon exposure to air
$MgCO_3$	--	--	*Anhydrous*: decomposes above 350 °C: $MgCO_3 \rightarrow MgO + CO_2$ *Hydrous*: dehydrates between 157 °C and 179 °C depending on degree of hydration
$Pb(NO_3)_2$	270	--	Decomposes in air above ~300 °C: $2Pb(NO_3)_2 \rightarrow 2PbO + 4\ NO_2 + O_2$
$PbCO_3$	--	--	Decomposes in air above ~200 °C: $2Pb(CO_3) \rightarrow Pb_2OCO_3 + CO_2$ Further decomposition above~310 °C: $Pb_2OCO_3 \rightarrow 2PbO + CO_2$
$Pb_2(NO_3)_2(C_2O_4)\cdot 2H_2O$			Dehydrates 100-150 °C; then breaks down into $Pb(NO_3)_2 + PbC_2O_4$ + complex Pb nitrate & carbonate oxysalts. PbC_2O_4 decomposes between 310-315 °C
PbO_2	--	--	Decomposes above 290 °C: $PbO_2 \rightarrow Pb_{12}O_{19} \rightarrow Pb_{12}O_{17} \rightarrow Pb_3O_4 \rightarrow PbO$ First at 290 °C, second at 350 °C, third at 375 °C and fourth at 600 °C
Pb_2OCO_3			A thermal decomposition product of cerussite (e.g., Ware and Bayliss, 1962; Grisafe and White, 1964; Pring, et al., 1990), Pb_2OCO_3 decomposes above~310 °C: $Pb_2OCO_3 \rightarrow 2PbO + CO_2$

Sample 69120-1 displays a variety of thermochemical trends similar to those noted above. There is significant mass loss between 262.6 °C and 316.3 °C, accompanied by the emission of copious amounts of CO_2 and NO (but no H_2O). Two exothermic peaks occur within this temperature range, at 293.1 °C and 303.6 °C. The latter has a 'shoulder' on its high-temperature leg indicative of an additional reaction (Figure 10) which likely corresponds to the release of NO and CO_2 between 324 – 330 °C. These phenomena may mirror the combined effects of the decarbonation of lead oxalate and the breakdown of $Pb_6O_5(NO_3)_2$ observed by Boudaren et al. (2001). However, the signal at m/z=16 (Figure 11) rises in parallel with CO_2 between 262.6 °C and 316.3 °C. If the signal at m/z=16 is indicative of oxygen, even as a fragment ion of CO_2 or NO_x (in the near total absence of H_2O), it would follow that no oxygen-consuming reactions—such as the oxidation of CO—are taking place in this temperature interval. However, it is also possible that the amount of CO generated by the reaction is miniscule, or that the signal at m/z=16 could also indicate the evolution of isobars such as CH_4^+ or NH_2^+. Unfortunately, we can only speculate on the significance of $Pb_2(NO_3)_2(C_2O_4)$ breakdown *vis a vis* the thermochemistry of sample 69120-1 as quadrupole mass spectrometry offers no way to evaluate CO emission, which is completely obscured by ambient N_2. A final CO_2 peak at 363.4 °C corresponds to a mass loss of 2.17% (Figure 10), and may be related to the breakdown of PbC_2O_4 (Gavris et al., 2010) produced, in turn, by the breakdown of the mixed oxalate-nitrate. The sample begins to denitrify above 451 °C, much like the top layer salt sample S813389.

The DSC-MS results from sample 69120-2 (Figure 11) are similar in many respects to those of 69120-1, despite its more diverse mineralogy. XRD data revealed the presence of cerussite ($PbCO_3$), and sodium nitrite (+ amorphous material) as primary constituents, with minor amounts of $NaNO_3$, $Pb(NO_3)_2$, shannonite ($Pb_2O(CO_3)$), sodium nitrate oxide ($Na_3O(NO_2)$), and magnesite ($Mg(CO_3)$). Unlike 69120-1, the endothermic dehydration of 69120-2 at ~184 °C yields a copious amount of H_2O, though little to no water is released at higher temperatures. The CO_2 peaks at ~305.7 °C and 347.5 °C correspond to endothermic peaks and may represent the decarbonation of cerussite and magnesite, respectively, and possibly the breakdown of PbC_2O_4 (e.g., Gavris et al., 2010) though the latter may appear exothermic due to the oxidation of the emitted CO. The melting of $NaNO_3$ and/or the breakdown of sodium nitrite and lead nitrate may be responsible for the broad NO peak at ~310 °C. Experiments by Bauer et al. (2009) show that molten Na nitrate reacts with graphite, resulting in the emission of both NO and CO_2 above 300 °C. A similar phenomenon may facilitate the release of NO in this sample at ~310 °C, though the large NO peaks at 432.5 °C and 495.6 °C corresponding to endothermic reactions at 435.9 °C and 497.6 °C show no coeval CO_2 emissions. Thus it seems possible that the high-temperature NO peaks are related to the final breakdown of Pb and Na nitrates into their corresponding oxides.

Studies of $Pb(NO_3)_2$ by Vratny and Gugliotta (1963), and Cram and Davies (1976) indicate that its thermal decomposition proceeds in a stepwise manner over a very broad temperature range (<100 °C – 555 °C), with each reaction releasing NO, NO_2 and O_2 in varying proportions until all that remains is Pb_2O_3 and/or PbO_2. In particular, the 'Stage 5' and 'Stage 6' breakdown of Pb oxynitrates postulated by Cram and Davies (1976) fall close to the peak temperatures observed for high temperature NO emission from 69120-2. Conversely, both Jackson et al. (1995) and L'vov and Novichikhin (1995)—working with a small volume of $Pb(NO_3)_2$ solution deposited on graphite—observed a large NO peak at ~375 °C during $Pb(NO_3)_2$ breakdown, which was not seen in this study. Performing a similar experiment, with similar starting materials, Jackson et al. (1995) also observed smaller NO peaks at ~440 °C and ~480 °C. Hoshino et al (1981) and Radhakrishnan Nair (1988) show that the activation energy and onset temperatures of the thermal breakdown in both $NaNO_3$ and $Pb(NO_3)_2$ decrease in the presence of metal oxide catalysts such as TiO_2, Al_2O_3, Cu_2O, and MnO_2.

Unlike 69120-1 and 69120-2, monolith sample 69120-3 (Figure 12) displayed completely different properties in response to thermal analysis, in terms of reactivity, mass loss, and gas release. In short, despite its mineralogical similarities to 69120-1 (Na and Pb nitrates, plus small amounts of PbO_2 and $Pb_2(NO_3)_2(C_2O_4)$) its response was very similar to that observed in samples 68645, 68492, 69173, and 69139 (Table 7). This may simply indicate that 69120-3 contains a larger organic load than either 69120-1 or 69120-2, and its thermal profile is thus dominated by exothermic outgassing and combustion of an organic constituent in the presence of nitrates. The markedly lower onset temperature of the first exothermic combustion reaction, (161.7 – 166.6 °C) is similar to that observed in 68645. Unlike 69120-1 and 69120-2, which showed little CO_2 emission above ~375 °C and continued to outgas NO_x to 500 °C, 69120-3 showed significant CO_2 emission at high temperature, suggesting a greater organic content, overall. The lack of significant NO and NO_x outgassing above ~400 °C was also characteristic of three of the four residual salt samples: 69173, 68645, and 68492.

The Significance of Sodium Nitrite and Shannonite

It should be noted that several of the compounds identified by XRF (Martinez et al., 2015) in the monolith samples may be produced during various stages of the thermal decomposition of $NaNO_3$ and $PbCO_3$. Sodium nitrite ($NaNO_2$), lead dioxide (PbO_2), and the lead oxycarbonate shannonite (Pb_2OCO_3) were identified in the monolith samples. Sodium nitrite, also a possible primary constituent within the top layer salts, is a common product of the thermal decomposition of sodium nitrate (e.g., Kramer, et al. 1982), though the temperature of the nitrate-to-nitrite reaction is dependent on multiple factors.

In nature, lead dioxide and lead oxycarbonate are rare, but may form during the thermal decomposition of cerussite (e.g., Warne and Bayliss, 1962; Grisafe and White, 1964). Pring, et al. (1990) identified the mineral form of Pb_2OCO_3, shannonite, in loose fill material from a lead mine in New South Wales, Australia where it formed pseudomorphs after well-formed cerussite crystals. This fill material was heated to <390 °C during a well-documented underground mine fire early in 1906. If the cerussite in the waste drum formed as a result of the interaction between oxalic acid and the Pb metal drum shielding, the possibility of a precursor thermal event before the drum was emptied to create drum 68660, may warrant further inquiry.

CONCLUSIONS

In FY2015, MET-1 and NCO-4 were contracted to perform Differential Scanning Calorimetry coupled with quadrupole Mass Spectrometry (DSC-MS) analyses as part of the analytical chemistry effort in support of the investigation of the WIPP Waste Drum failure that occurred on Valentine's Day, 2014. Initially, we examined the thermal behavior of pure, reagent-grade nitrate salts (Na, Pb, K) and nitrate salt hydrates (Fe and Mg) in order to identify the possible waste constituents, which would be most likely to release NO_x at low temperatures. We followed this study up with systematic analyses of four samples sent to us by C-AAC: 2 top layer salts, four residual salts sampled from otherwise empty parent waste barrels that were related in terms of content and time to the salt wastes placed into the drum (68660) that heated and pressurized, and further set of 3 salt residue monolith samples from a single parent drum, S855793, which was a precursor to drum 68660.

The results of these analyses ultimately pose more questions than they answer. However, from the limited set of data available at this time, we can derive several pertinent conclusions:

1) Of the pure nitrate samples analyzed, only Fe^{3+} nitrate hydrate releases NO_x below 200 °C when heated.
2) The thermal behavior of $NaNO_3$, when mixed with low levels (^{235}U = 1600 μg/g; ^{238}U = 840 μg/g; and 1 μg/g or less of ^{239}Pu, ^{237}Np, and ^{241}Am) of radioactive constituents, appears to be identical to that of the pure, unadulterated salt.
3) The thermal response of mixed nitrate salts (e.g., Na + Mg nitrate) is very different from that of the pure end-members.
4) Chemical transformations in nitrate salts and calcium oxalate are endothermic. However, the reactions observed in 5 of the 7 salt residue samples were strongly exothermic, with onset temperatures >160 °C.
5) Exothermic reactions observed in the salt residues release H_2O, CO_2 and NO_x, and strongly resemble those commonly observed in combustion reactions involving mixtures of nitrates and organics.

6) The mineralogy and, to a lesser extent, the thermal behavior of two samples, 69120-1 and 69120-2, suggests that they may have experienced heating and decarbonation prior to the initiation of thermal analysis.

Samples for thermal analysis by DSC-MS were first dried to 110 °C and ground into fine powders to reduce sampling bias. Thus, this effort was inherently not as effective as hoped in terms of identifying which waste phase could have self-heated or ignited at ambient temperatures. However, the conclusions listed above lay the groundwork for follow-on studies.

ACKNOWLEDGEMENT

Thanks to Daniel Garcia, Judy Roybal, Leonard Lujan, and Chastity Kolar (NCO-4) for their assistance during the course of the analytical work that comprises the bulk of this report. A debt of gratitude is also owed to Drs. Joshua White (MST-7), Kirk Veirs (MET-1), Kirk Weisbrod (AET-5), Rebecca Chamberlin (C-AAC), Patrick Martinez (C-AAC), Philip Leonard (M-7), and Paul Schumann (EM) for thorough and insightful technical reviews. The input of each of these individuals significantly improved the content of this report.

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ADDENDUM

DSC-MS Analysis of Mixtures of Tri- and Divalent Nitrate Hydrate Salts and Oxalic Acid

Following the compilation and synthesis of the DSC-MS data for pure nitrates, top layer salts, residual salts, and residual salt monoliths from unremediated LANL parent drums, we decided to explore the thermal response of oxalate / nitrate mixtures to determine if the carbon contributed by the oxalate (as crystalline oxalic acid hydrate) would react exothermically with gaseous NO_x emitted during the thermal breakdown of trivalent and divalent nitrate hydrates of Al and Fe(III). This would presumably involve the cleavage of the C-C bond in oxalic acid, a C2 compound, to form CO_2. Little work has been documented on the nature of reactions between oxalates (or oxalic acid) and nitrate salts at high temperature. Kubota (1982) reports the Mn-catalyzed breakdown of oxalate ions in HNO_3 solutions below 100 °C.

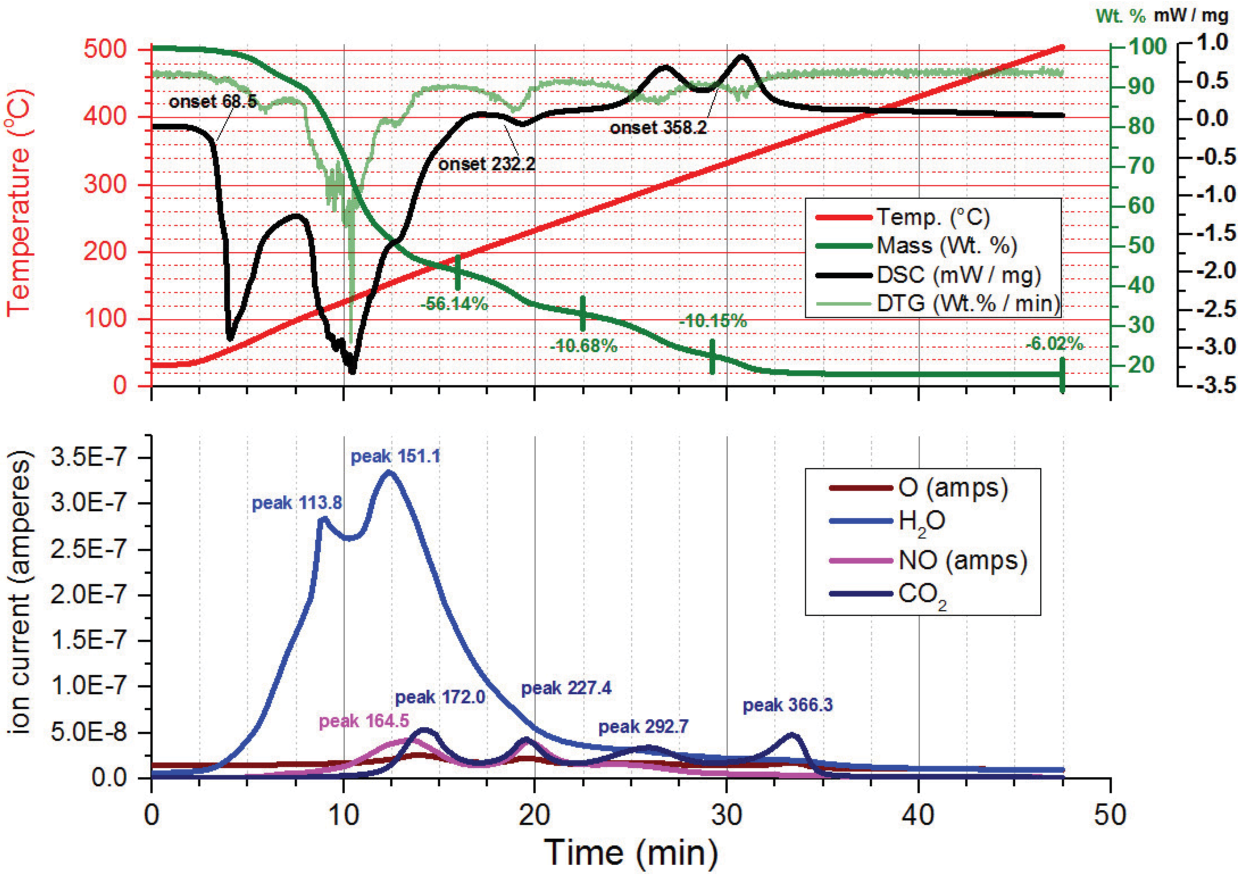
Oxalic acid is commonly manufactured by the oxidation of carbohydrates with nitric acid. Pure, anhydrous oxalic acid melts and decomposes at 187 °C, though it begins to sublimate slightly below 100 °C. The dihydrate, if heated rapidly, melts at 101.5 °C. If heated slowly, it dehydrates to the anhydrous form at the same temperature. Upon heating, oxalic acid decomposes to formic acid, carbon monoxide, carbon dioxide, and water (Sawada and Murakami, 2000).

Pure reagent-grade oxalic acid hydrate, and Al and Fe(III) nitrate hydrates were obtained from LANL's M-7 group. Samples were prepared by weighing equimolar masses of each nitrate hydrate and oxalic acid hydrate and grinding each pair to a fine powder using a clean agate mortar and pestle. Additionally, two samples utilizing a 10:1 mixture of Al nitrate hydrate and oxalic acid were analyzed. A small quantity (10 – 40 mg) of each mixture was transferred, using a stainless steel spatula, into a clean platinum-rhodium (Pt-Rh) STA pan, placed on the DSC carrier and covered with a pierced Pt-Rh lid. When mixed with oxalic acid, Fe(III) nitrate hydrate spontaneously sorbed water and turned into a canary-yellow paste. The DSC-MS run described here used the hydrated paste as the starting material. In the dry air glovebox environment, the remaining unanalyzed paste dehydrated and turned into a dull yellow powder in a matter of hours.

During analysis, ambient glovebox conditions were: temperature (T) = 25 to 30 ^{o}C, relative humidity (%RH) = 0.0 – 1.0%. Pre-run baseline values varied from approximately -5.5 to -4 μV. For all nitrate / oxalic acid mixtures, temperature Profile 4 was used (Table 3) and the run was stopped manually at 500 °C. Unless otherwise indicated, all other analytical parameters for the nitrate/oxalate acid samples were identical to those used for the analysis of the samples described in the main body of this report.

$Al(NO_3)_3{\cdot}9H_2O$ / $C_2H_2O_4{\cdot}2H_2O$ – 10:1 molar ratio: The thermal behavior of 10:1 molar mixtures of Al nitrate nonahydrate and oxalic acid dihydrate are (Figure 13), as one would expect, dominated by the thermal behavior of the nitrate itself. In the first sample, run immediately after mixing, endothermic melting (onset = 45.3 °C) and dehydration (onset = 107.3 °C) reactions dominate below ~210 °C; the latter releasing copious amounts of H_2O (peak = 123.8 °C) and minor amounts of NO. These reactions run to completion at ~190 °C, at which point the mixture has lost 55.72% of its initial mass. Three subsequent reactions, one mildly endothermic and two mildly exothermic release mostly NO and CO_2 in varying amounts. The first onsets at 212.9 °C, releasing both NO (peak = 217.2 °C) and CO_2 (peak = 214.9 °C), and corresponds to a further mass loss of 10.53 wt. %. The two exothermic peaks onset at 282.3 and 326.2 °C and both release CO_2. These reactions result in additional mass losses of 10.62 wt. % and 5.10 wt. %, respectively.

Figure 13: DSC-MS data (to 500 °C) for 10:1 molar mixture of $Al(NO_3)_3·9H_2O$ and oxalic acid.

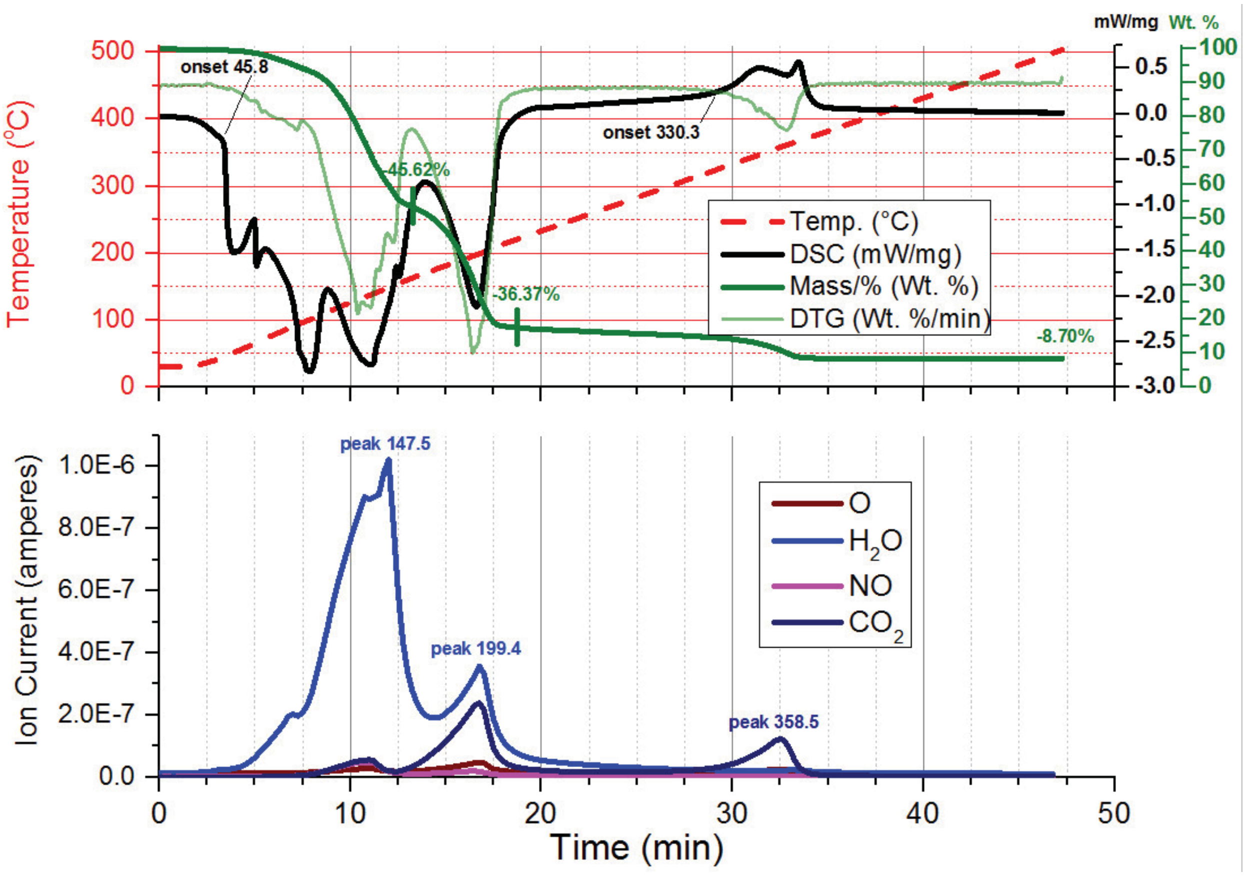


A duplicate sample run several hours later (Figure 13) yields results that are slightly different in several respects. Melting onsets at a higher temperature (68.5 °C), and dehydration occurs in two discrete H_2O pulses which peak at 113.8 °C and 151.1 °C. The small endothermic peak also onsets at a slightly higher temperature (232.2 °C) and is barely visible, as do the two CO_2-emitting exothermic reactions. Gas releases follow a similar sequence, however, with minor NO emission coeval with dehydration, followed by three discrete pulses of CO_2, the first of which is accompanied by significant NO emission. Mass losses in this sample were nearly identical: -56.14 wt. %; -10.68 wt. %; -10.15 wt. %; and 6.02 wt. %.

$Al(NO_3)_3·9H_2O$ / $C_2H_2O_4·2H_2O$ – 1:1 molar ratio: When the oxalic acid content is increased to achieve an equal molar ratio with the aluminum nitrate nonahydrate, the behavior of the sample at temperatures below ~200 °C is, again, dominated by the thermal response of $Al(NO_3)_3·9H_2O$ to increasing temperature (Figure 14). Endothermic melting onsets at 45.8 °C, followed by multiple dehydration events (H_2O peaks at 147.5 °C and 199.4 °C), the first of which releases both NO and CO_2. A subsequent dehydration releases a greater amount of CO_2, indicating that the oxalic acid is also beginning to break down. Though there may be as many as four discrete H_2O peaks, mass loss proceeds in 2 easily-resolvable steps: -46.52 wt.% complete at 161.8 °C and a further -36.37 wt. % complete at 230.1 °C. Above ~230 °C, the enthalpy plateaus close to 0 to ~300 °C, where an exothermic reaction releasing only CO_2 onsets at 330.3 °C. This final CO_2 release corresponds to a mass loss of 8.70 wt. %. A second exothermic peak that follows the first may be indicative of CO oxidation, as the oxygen concentration of the offgas drops precipitously, immediately prior to the development of this peak (Figure A2).

A duplicate sample run several hours later produced somewhat different results, though melting onsets at a higher temperature (63.5 °C), and dehydration occurs in two discrete H_2O emissions which peak at 117.8 °C and 207.7 °C, each accompanied by a discrete endothermic reaction. Mass loss after the first dehydration is 15.63 wt. %, and a further 68.68 wt. % is lost after the second dehydration, which is complete at ~236 °C. Unlike the initial run, significant amounts of CO_2 and NO are also released during the second dehydration reaction. Additional CO_2 and O are evolved during a significant exothermic reaction which onsets at 353.3 °C. The final CO_2 release corresponds to a mass loss of 9.04 wt. %, and lacks the ancillary CO oxidation peak.

Figure 14: DSC-MS data (to 500 °C) for 1:1 molar mixture of $Al(NO_3)_3{\cdot}9H_2O$ and oxalic acid.

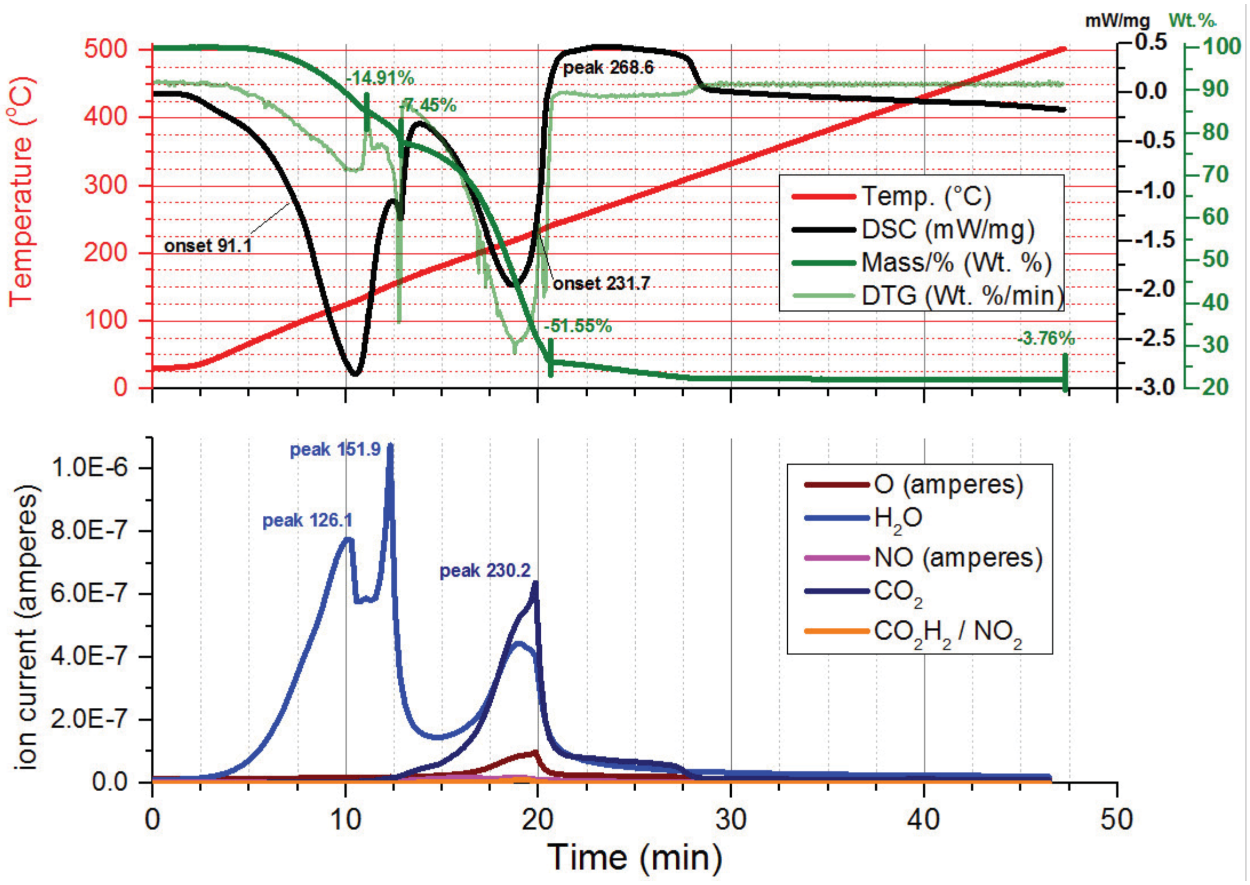


$Fe(NO_3)_3{\cdot}9H_2O$ / $C_2H_2O_4{\cdot}2H_2O$ – 1:1 molar ratio: The equimolar mixture of oxalic acid dihydrate and ferric nitrate nonahydrate produces results which are quite similar to those seen in the equimolar mixture with aluminum nitrate nonahydrate. Below ~230 °C, the most significant features consist of the endothermic melting and multi-step dehydration of the ferric nitrate (Figure 15). These reactions are immediately followed by a broad, plateau-like exothermic reaction which onsets at 231.7 °C and peaks at 268.6 °C. This reaction is complete at ~324.8 °C, and is dominated by CO_2 release. The release of formic acid (CO_2H_2) between ~210 and 230 °C is evident by the decoupling of the NO peak from the peak at m/Z = 46, which corresponds to NO_2 for most of these analyses. Here, the rise of m/Z=46 tracks the evolution of H_2O, H_2, and CO_2 produced by the possible breakdown of ferrous oxalate, rather than that of NO (Figure 16).

Discussion: Oxalic – Nitrate mixtures

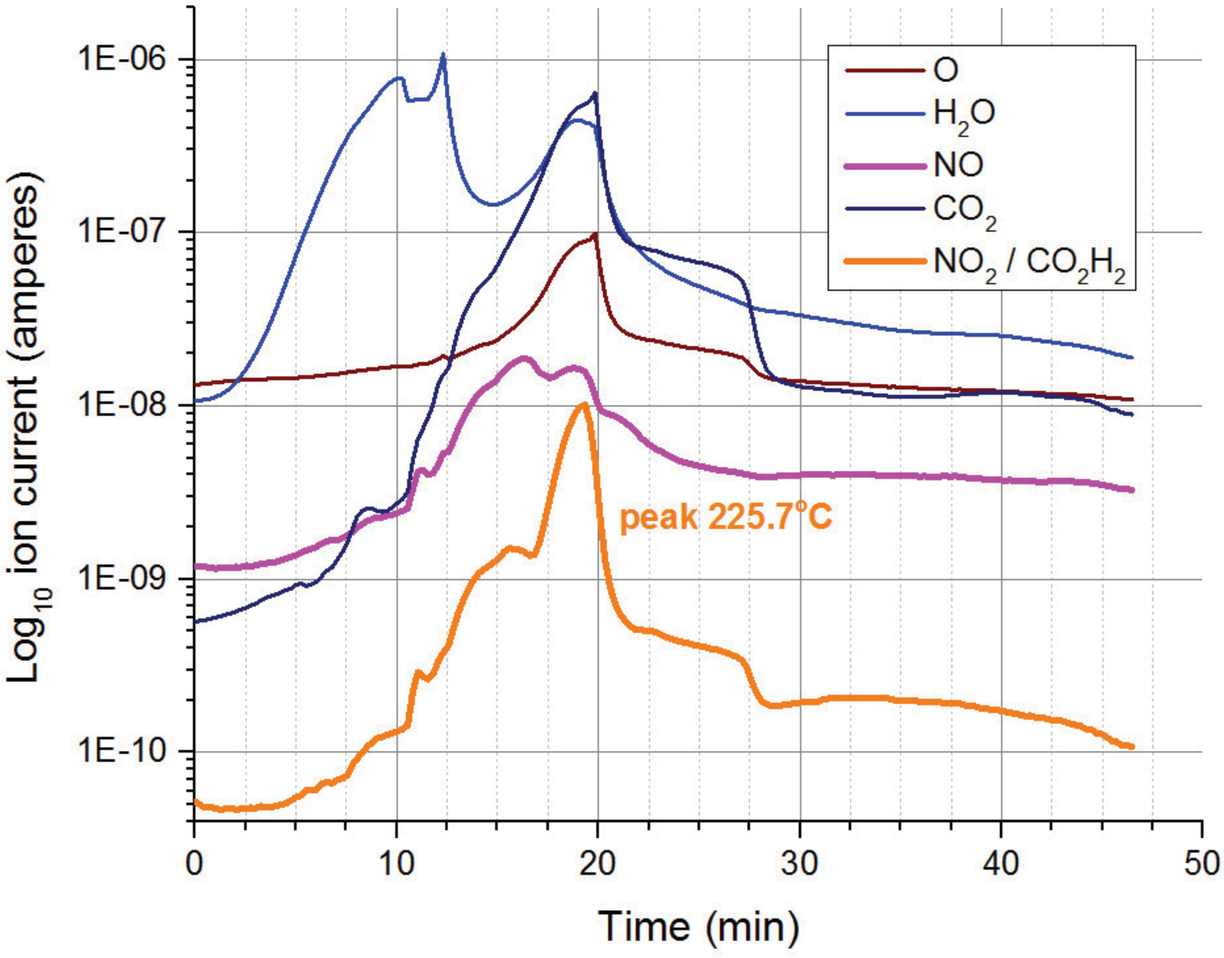
The few experiments run using mixed trivalent nitrate salts and oxalic acid indicate that oxalic acid is not oxidized exothermically by NO_x released as the nitrate breaks down. Instead, the oxalic acid breaks down exothermically at higher temperatures, releasing formic acid, CO_2, CO, and H_2O. As with pure nitrate salts, the enthalpy profile of these mixtures below ~210 °C is dominated by endothermic phase changes, dehydration / denitrification reactions and the formation of metal oxalates.

Figure 15: DSC-MS data (to 500 °C) for 1:1 molar mixture of $Fe(NO_3)_3{\cdot}9H_2O$ and oxalic acid.



The persistence of oxalate in some form in the Al nitrate/oxalic acid is suggested by the exothermic reactions which onset at ~330-360 °C. Though pure oxalic acid breaks down below 200 °C, Palaniappan (1995) also noted a the occurrence of a broad exotherm above 210 °C in mixtures of oxalic acid and polyaniline salts which was attributed to the breakdown of the oxalic acid. Broadbent, et al (1967) observed exothermic peaks just below 300 °C in ferrous oxalate heated in air. When heated in nitrogen, the same compound yielded a flat-topped plateau in the same temperature range that resembles the one seen in Figure 15, above. Other transition metal oxalates decompose to CO_2, CO, H_2O and the corresponding metal oxide in air between ~240 and 460 °C (Mohamed et al, 2005). For ferrous oxalate hydrate, Mohamed et al. (2005) observed an exothermic peak at 244 °C, which coincided to the compound's breakdown into iron oxides. In the case of the aluminum nitrate / oxalic acid mixtures, the formation of aluminum oxalate during the breakdown of the nitrate and oxalic acid seems likely, as other investigators (e.g., Kumar Saha and Pramanik, 1994) have noted that aluminum oxalate reacts exothermically, breaking down at ~375 °C.

Figure 16: Decoupling of m/Z=46 from m/Z=30, indicative of formic acid evolution during Fe3+ oxalate breakdown at ~220 °C in the $Fe(NO_3)_3 \cdot 9H_2O$ / oxalic acid mixture.



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ATTACHMENT A - Chain of Custody (page 1 of 1)

LOS ALAMOS NATIONAL LABORATORY ANALYTICAL CHEMISTRY	CHAIN OF CUSTODY

Sample Management: Pat Martinez | Z#: | Signature: Pat Martinez | Date: 3/8/16

LabWare Sample No.	Sample Submitter Name/ Sample No.	Custody Seal/TID Intact (Y/N)	Container Intact (Y/N)	Discrepancy Record Initiated (Y/N)	Comments
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	SFWB8-15-1 3/7/2016	↓	↓		
	SFWB8-15-1 3/7/2016	↓	↓		

Relinquished by (Print name and sign)		Received by (Print name and sign)	
Print Name: Hongzhao Tian	Z#: 230045	Print Name: Donivon Porterfield	Z#: 113720
Signature: Hongzhao Tian	Date/Time: 3/8/16 1:25pm	Signature:	Date/Time: 3/8/2016 1:25pm
Print Name: Donivon Porterfield	Z#: 113720	Print Name: David M. Wayne	Z#: 113674
Signature:	Date/Time: 3/8/2016 1:56pm	Signature:	Date/Time: 3-8-16 1:57 PM
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:

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Samples intro-ed into GB Line @ 2:15 PM 3-8-16

ATTACHMENT A - Chain of Custody (page 2 of 2)

LabWare Sample No: ____________________

Relinquished by (Print name and sign)	Received by (Print name and sign)
Print Name: ______ Z#: ______	Print Name: ______ Z#: ______
Signature: ______ Date/Time: ______	Signature: ______ Date/Time: ______
Print Name: ______ Z#: ______	Print Name: ______ Z#: ______
Signature: ______ Date/Time: ______	Signature: ______ Date/Time: ______
Print Name: ______ Z#: ______	Print Name: ______ Z#: ______
Signature: ______ Date/Time: ______	Signature: ______ Date/Time: ______
Print Name: ______ Z#: ______	Print Name: ______ Z#: ______
Signature: ______ Date/Time: ______	Signature: ______ Date/Time: ______
Print Name: ______ Z#: ______	Print Name: ______ Z#: ______
Signature: ______ Date/Time: ______	Signature: ______ Date/Time: ______
Print Name: ______ Z#: ______	Print Name: ______ Z#: ______
Signature: ______ Date/Time: ______	Signature: ______ Date/Time: ______

Remarks: ______________________________

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Disposed to Residue by:

Print Name: ______ Z#: ______ Signature: ______

Residue Number: ______ Date/Time: ______

ATTACHMENT A - Chain of Custody (page 1 of 1)

LOS ALAMOS NATIONAL LABORATORY **ANALYTICAL CHEMISTRY**	**CHAIN OF CUSTODY**

Sample Management: Pat Martinez 120558 3/15/16

Print Name **Z#** **Signature** **Date**

LabWare Sample No.	Sample Submitter Name/ Sample No.	Custody Seal/TID Intact (Y/N)	Container Intact (Y/N)	Discrepancy Record Initiated (Y/N)	Comments
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	SFWBB-15-4 3/14/2016	Y	Y	N	
	SF WBB-15-4 3/14/2016	Y	Y	N	
N/A	SFWBB-15-4 3/14/2016	Y	Y	N	

Relinquished by (Print name and sign)	**Received by (Print name and sign)**
Print Name: Geoffrey N. Brown Z#: 118813	Print Name: 184010 Z#:
Signature: N. Brown Date/Time: 3/15/16 2 pm	Signature: F DICKSON Date/Time: 3.15.16 2PM
Print Name: 184010 Z#:	Print Name: David Wayne Z#: 113674
Signature: Date/Time: 3.15.16 3PM	Signature: Date/Time: 3-15-16 15:00 hrs
Print Name: David M Wayne Z#: 113674	opened & Print Name: Z#:
Signature: Date/Time: 3-15-16 15:35 -	Into, into Glovebox Line Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:

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ATTACHMENT A - Chain of Custody (page 2 of 2)

LabWare Sample No: ______________________

Relinquished by (Print name and sign)	**Received by (Print name and sign)**
Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______	Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______
Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______	Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______
Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______	Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______
Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______	Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______
Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______	Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______
Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______	Print Name:____________ Z#:_______ Signature:____________ Date/Time:_______

Remarks: ____

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Disposed to Residue by:

Print Name:______________ Z#:__________ Signature:______________________

Residue Number:______________________ Date/Time:______________________

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ATTACHMENT A - Chain of Custody (page 1 of 1)

LOS ALAMOS NATIONAL LABORATORY **ANALYTICAL CHEMISTRY**	**CHAIN OF CUSTODY**

Sample Management: Donivan Porterfield 2113720

Print Name **Z#** **Signature** **Date**

LabWare Sample No.	Sample Submitter Name/ Sample No. DP 113720 3/22/2016 8	Custody Seal/TID Intact (Y/N)	Container Intact (Y/N)	Discrepancy Record Initiated (Y/N)	Comments
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	SFWB8-15-2 3/21/2016	↓	↓		
	SFWB8-15-2 3/21/2016	↓	↓		
	SFWB8-15-1 3/21/2016	↓	↓		
	SFWB8-15-4 3/21/2016	↓	↓		

Relinquished by (Print name and sign)	Received by (Print name and sign)
Print Name: Geoff Brown Z#: 118813	Print Name: Donivan Porterfield Z#: 113720 1:56pm
Signature: Date/Time: 3/22/16 1:56	Signature: Date/Time: 3/22/2016
Print Name: Donivan Porterfield Z#: 113720 2:38pm	Print Name: Dave Wayne Z#: 113674
Signature: Date/Time: 3/22/2016	Signature: Date/Time: 3-22-16 2:38
Print Name: Dave Wayne Z#: 113674	Print Name: Leonard Lujan Z#: 120527
Signature: Date/Time: 3-22-16 2:55 pm	Signature: Date/Time: 3.22.16 2:55 pm
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:

QA-38, Att. A, Form 1, R1 (November 2015) Page 1 of 2

ATTACHMENT A - Chain of Custody (page 2 of 2)

LabWare Sample No: ____________________

Relinquished by (Print name and sign)	**Received by (Print name and sign)**
Print Name:__________ Z#:______	Print Name:__________ Z#:______
Signature:__________ Date/Time:______	Signature:__________ Date/Time:______
Print Name:__________ Z#:______	Print Name:__________ Z#:______
Signature:__________ Date/Time:______	Signature:__________ Date/Time:______
Print Name:__________ Z#:______	Print Name:__________ Z#:______
Signature:__________ Date/Time:______	Signature:__________ Date/Time:______
Print Name:__________ Z#:______	Print Name:__________ Z#:______
Signature:__________ Date/Time:______	Signature:__________ Date/Time:______
Print Name:__________ Z#:______	Print Name:__________ Z#:______
Signature:__________ Date/Time:______	Signature:__________ Date/Time:______
Print Name:__________ Z#:______	Print Name:__________ Z#:______
Signature:__________ Date/Time:______	Signature:__________ Date/Time:______

Remarks: ____

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Disposed to Residue by:

Print Name:__________ Z#:__________ Signature:____________________

Residue Number:____________________ Date/Time:____________________

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ATTACHMENT A - Chain of Custody (page 1 of 1)

LOS ALAMOS NATIONAL LABORATORY ANALYTICAL CHEMISTRY	**CHAIN OF CUSTODY**

Sample Management: Pat Martinez 120558 3/29/16

Print Name **Z#** **Signature** **Date**

LabWare Sample No.	Sample Submitter Name/ Sample No.	Custody Seal/TID Intact (Y/N)	Container Intact (Y/N)	Discrepancy Record Initiated (Y/N)	Comments
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	SFWB8-15-4 3/27/16				1.3g
	SFWB8-15-1 3/27/16				1.3g

Relinquished by (Print name and sign)		Received by (Print name and sign)	
Print Name: Geoff Brown	Z#: 118813	Print Name: Doniven Porterfield	Z#: 113720 11am
Signature:	Date/Time: 3/29/16 11am	Signature:	Date/Time: 3/29/16
Print Name: Doniven Porterfield	Z#: 113720 11:47am	Print Name: David M Wayne	Z#: 113674
Signature:	Date/Time: 3/29/16	Signature:	Date/Time: 3-29-16 11:47 AM
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:

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opened & samples put in line RM 208 @ 15:40
3-29-16

ATTACHMENT A - Chain of Custody (page 2 of 2)

LabWare Sample No: ____________________

Relinquished by (Print name and sign)		**Received by (Print name and sign)**	
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:
Print Name:	Z#:	Print Name:	Z#:
Signature:	Date/Time:	Signature:	Date/Time:

Remarks: ____________________

Disposed to Residue by:

Print Name: __________ Z#: ______ Signature: __________

Residue Number: __________ Date/Time: __________

QA-38, Att. A, Form 1, R1 (November 2015) Page 2 of 2

ATTACHMENT A - Chain of Custody (page 1 of 1)

LOS ALAMOS NATIONAL LABORATORY ANALYTICAL CHEMISTRY	CHAIN OF CUSTODY

Sample Management: Doniven Porterfield 113720 4/7/2016

Print Name **Z#** **Signature** **Date**

LabWare Sample No.	Sample Submitter Name/ Sample No.	Custody Seal/TID Intact (Y/N)	Container Intact (Y/N)	Discrepancy Record Initiated (Y/N)	Comments
~~040616 SFWB8-15-1~~	040616 SFWB8-15-1	Y	Y		
	040616 SFWB8-15-2	Y	Y		
	040616 SFWB8-15-4	Y	Y		

DPP 4/7/16 Z113720

Relinquished by (Print name and sign)	Received by (Print name and sign)
Print Name: Geoff Brown Z#: 118413	Print Name: Doniven Porterfield Z#: 113720 9:07 am
Signature: Date/Time: 4/7/16 9:07	Signature: Date/Time: 4/7/16
Print Name: Doniven Porterfield Z#: 113720 9:47 am	Print Name: David M. Wayne Z#: 113674
Signature: Date/Time: 4/7/2016	Signature: Date/Time: 9:47 am 4-7-16
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:

Sample bag opened - vials introed into GB line @10:13 AM 4-7-16

ATTACHMENT A - Chain of Custody (page 2 of 2)

LabWare Sample No: ____________________

Relinquished by (Print name and sign)	Received by (Print name and sign)
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:

Remarks: ____________________

Disposed to Residue by:

Print Name: ____________ Z#: ____________ Signature: ____________

Residue Number: ____________ Date/Time: ____________

QA-38, Att. A, Form 1, R1 (November 2015) Page 2 of 2

ATTACHMENT A - Chain of Custody (page 1 of 1)

LOS ALAMOS NATIONAL LABORATORY ANALYTICAL CHEMISTRY	CHAIN OF CUSTODY

Sample Management: Doniven Porterfield 113720 4/12/2016

Print Name Z# Signature Date

LabWare Sample No.	Sample Submitter Name/ Sample No.	Custody Seal/TID Intact (Y/N)	Container Intact (Y/N)	Discrepancy Record Initiated (Y/N)	Comments
	SFWB8-15-1 041116		Y		
	SFWB8-15-2 041116		Y		
	SFWB8-15-4 041116		Y		

Relinquished by (Print name and sign)	Received by (Print name and sign)
Print Name: Geoff Brown Z#: 118813	Print Name: Doniven Porterfield Z#: 113720
Signature: Date/Time: 4/12/16 8:55	Signature: Date/Time: 4/12/2016 8:55
Print Name: ~~David McWayne~~ Doniven Porterfield Z#: ~~113~~ Z113720 9:50 am	Print Name: David McWayne Z#: 113674
Signature: Date/Time: 4/12/2016	Signature: Date/Time: 4-12-16 9:50 AM
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:
Print Name: Z#:	Print Name: Z#:
Signature: Date/Time:	Signature: Date/Time:

QA-38, Att. A, Form 1, R1 (November 2015) Page 1 of 2

samples placed into GB line @ 10:37 AM
4-12-16

ATTACHMENT A - Chain of Custody (page 2 of 2)

LabWare Sample No: ____________________

Relinquished by (Print name and sign)	Received by (Print name and sign)
Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______	Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______
Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______	Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______
Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______	Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______
Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______	Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______
Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______	Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______
Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______	Print Name: ____________ Z#: ______ Signature: ____________ Date/Time: ______

Remarks: ____

____

____

____

____

Disposed to Residue by:

Print Name: ____________ Z#: ____________ Signature: ____________________

Residue Number: ____________________ Date/Time: ____________________

QA-38, Att. A, Form 1, R1 (November 2015) Page 2 of 2

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Werksbescheinigung / *Manufacturer´s Certification*

nach / *in accordance with*

DIN 51007, ASTM E 967 / DIN EN 10 204 - 2.1

DTA/DSC-Kalibrierset für Pt Tiegel
DTA/DSC Calibration Set for Pt crucibles

Ausstelldatum / *Date of Issue:*	21.10.2014
Bestell-Nr. / *Order - No:*	6.223.5-91.2.01
Hersteller-Serien-Nr. / *Series-No:*	14670
Kommission / *Commission:*	216042070
Temperaturbereich / *Temperature range*	69,2°C

Kalibriersubstanz / *Calibration substance 500 mg*

Bezeichnung *Material*	chem. Zeichen *Formula*	Reinheit *Purity* [%]	Theoretische Umwandl.-Temperatur *Theoretical Transform.-temperature T [°C]*	Theoretische Umwandl.-enthalpie *Theoretical Transform.-enthalpy* ΔH [J/g]	Chargen - Nr. *Lot No.*	Mindest-Haltbarkeit *Min. Durability* *1	Quellen-angabe *Reference*
Biphenyl / Biphenyl	C12H10	99,5	69,2	-120,5	12905BHV	10/2019	T=[13]; ΔH=[14]

*1 bei Veränderung der angegebenen Werte oder Verdacht auf Kontamination bitte sofort erneuern!
renew please immediately on change of the indicated values or suspicion on contamination!

Literatur:

[13] U.Schley, PTB-Mitteilung 89 (1979)13-21
[14] U. Ueberreiter und H.-J. Orthmann, Z. Naturforschung Teil A, 5 (1950) 101 – 108

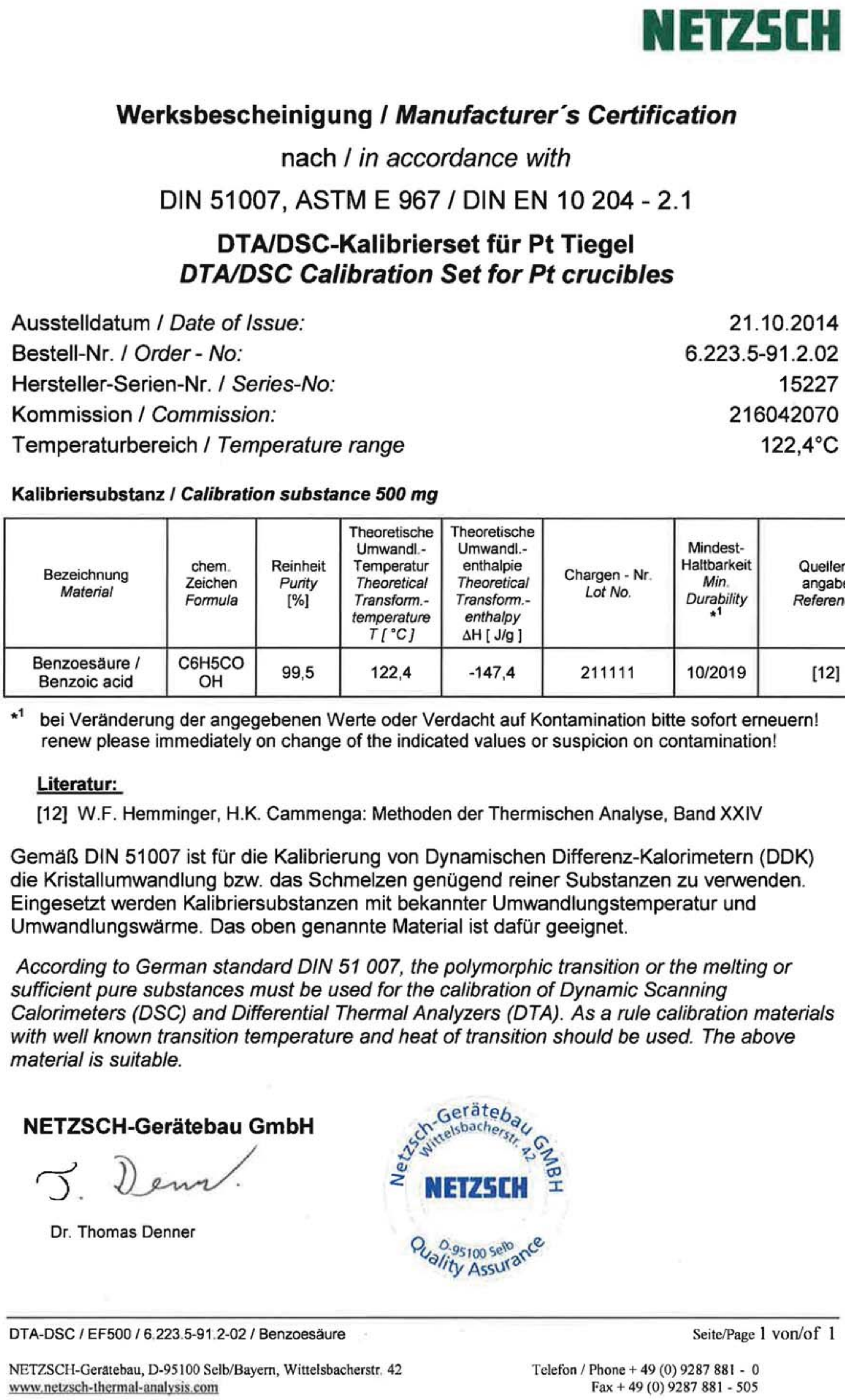
Gemäß DIN 51007 ist für die Kalibrierung von Dynamischen Differenz-Kalorimetern (DDK) die Kristallumwandlung bzw. das Schmelzen genügend reiner Substanzen zu verwenden. Eingesetzt werden Kalibriersubstanzen mit bekannter Umwandlungstemperatur und Umwandlungswärme. Das oben genannte Material ist dafür geeignet.

According to German standard DIN 51 007, the polymorphic transition or the melting or sufficient pure substances must be used for the calibration of Dynamic Scanning Calorimeters (DSC) and Differential Thermal Analyzers (DTA). As a rule calibration materials with well known transition temperature and heat of transition should be used. The above material is suitable.

NETZSCH-Gerätebau GmbH

T. Denn

Dr. Thomas Denner



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Werksbescheinigung / *Manufacturer´s Certification*

nach / *in accordance with*

DIN 51007, ASTM E 967 / DIN EN 10 204 - 2.1

DTA/DSC-Kalibrierset für Pt Tiegel
DTA/DSC Calibration Set for Pt crucibles

Ausstelldatum / *Date of Issue:*	21.10.2014
Bestell-Nr. / *Order - No:*	6.223.5-91.2.02
Hersteller-Serien-Nr. / *Series-No:*	15227
Kommission / *Commission:*	216042070
Temperaturbereich / *Temperature range*	122,4°C

Kalibriersubstanz / *Calibration substance 500 mg*

Bezeichnung *Material*	chem. Zeichen *Formula*	Reinheit *Purity* [%]	Theoretische Umwandl.-Temperatur *Theoretical Transform.-temperature T [°C]*	Theoretische Umwandl.-enthalpie *Theoretical Transform.-enthalpy* ΔH [J/g]	Chargen - Nr. *Lot No.*	Mindest-Haltbarkeit *Min. Durability* *1	Quellen-angabe *Reference*
Benzoesäure / Benzoic acid	C6H5COOH	99,5	122,4	-147,4	211111	10/2019	[12]

*1 bei Veränderung der angegebenen Werte oder Verdacht auf Kontamination bitte sofort erneuern!
renew please immediately on change of the indicated values or suspicion on contamination!

Literatur:

[12] W.F. Hemminger, H.K. Cammenga: Methoden der Thermischen Analyse, Band XXIV

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NETZSCH-Gerätebau GmbH

Dr. Thomas Denner

NETZSCH-Gerätebau, D-95100 Selb/Bayern, Wittelsbacherstr. 42
www.netzsch-thermal-analysis.com
Telefon / Phone + 49 (0) 9287 881 - 0
Fax + 49 (0) 9287 881 - 505

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Werksbescheinigung / *Manufacturer´s Certification*

nach / *in accordance with*

DIN 51007, ASTM E 967 / DIN EN 10 204 - 2.1

DTA/DSC-Kalibrierset für Pt Tiegel
DTA/DSC Calibration Set for Pt crucibles

Ausstelldatum / *Date of Issue:*	21.10.2014
Bestell-Nr. / *Order - No:*	6.223.5-91.2.03
Hersteller-Serien-Nr. / *Series-No:*	14859
Kommission / *Commission:*	216042070
Temperaturbereich / *Temperature range*	164,2°C

Kalibriersubstanz / *Calibration substance 500 mg*

Bezeichnung *Material*	chem. Zeichen *Formula*	Reinheit *Purity* [%]	Theoretische Umwandl.-Temperatur *Theoretical Transform.-temperature T [°C]*	Theoretische Umwandl.-enthalpie *Theoretical Transform.-enthalpy* ΔH [J/g]	Chargen - Nr. *Lot No.*	Mindest-Haltbarkeit *Min. Durability* *1	Quellen-angabe *Reference*
Rubidiumnitrat / Rubidium nitrate	RbNO3	99,99	164,2	n. A. *2	180811	10/2017	[15]

*1 bei Veränderung der angegebenen Werte oder Verdacht auf Kontamination bitte sofort erneuern!
renew please immediately on change of the indicated values or suspicion on contamination!

Literatur:

[7] W. Eysel, K.-H. Breuer: Analytical Calorimetry. Plenum Publ. Corp., 1984
[15] NETZSCH-Gerätebau GmbH

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NETZSCH-Gerätebau GmbH

T. Denn

Dr. Thomas Denner

Netzsch-Gerätebau GmbH Wittelsbacherstr. 42 NETZSCH D-95100 Selb Quality Assurance

Werksbescheinigung / *Manufacturer´s Certification*

nach / *in accordance with*

DIN 51007, ASTM E 967 / DIN EN 10 204 - 2.1

DTA/DSC-Kalibrierset für Pt Tiegel
DTA/DSC Calibration Set for Pt crucibles

Ausstelldatum / *Date of Issue:*	21.10.2014
Bestell-Nr. / *Order - No:*	6.223.5-91.2.08
Hersteller-Serien-Nr. / *Series-No:*	15194
Kommission / *Commission:*	216042070
Temperaturbereich / *Temperature range*	426,4°C

Kalibriersubstanz / *Calibration substance 500 mg*

Bezeichnung *Material*	chem. Zeichen *Formula*	Reinheit *Purity* [%]	Theoretische Umwandl.-Temperatur *Theoretical Transform.-temperature T [°C]*	Theoretische Umwandl.-enthalpie *Theoretical Transform.-enthalpy* ΔH [J/g]	Chargen - Nr. *Lot No.*	Mindest-Haltbarkeit *Min. Durability* *1	Quellen-angabe *Reference*
Silbersulfat / Silver sulfate	Ag2SO4	99,999	426,4	-51,9	220210	10/2019	[12]

*1 bei Veränderung der angegebenen Werte oder Verdacht auf Kontamination bitte sofort erneuern!
renew please immediately on change of the indicated values or suspicion on contamination!

Literatur:

[12] W.F. Hemminger, H.K. Cammenga: Methoden der Thermischen Analyse, Band XXIV

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NETZSCH-Gerätebau GmbH

Dr. Thomas Denner

NETZSCH

Werksbescheinigung / *Manufacturer´s Certification*

nach / *in accordance with*

DIN 51007, ASTM E 967 / DIN EN 10 204 - 2.1

DTA/DSC-Kalibrierset für Pt Tiegel
DTA/DSC Calibration Set for Pt crucibles

Ausstelldatum / *Date of Issue:*	21.10.2014
Bestell-Nr. / *Order - No:*	6.223.5-91.2.05
Hersteller-Serien-Nr. / *Series-No:*	15482
Kommission / *Commission:*	216042070
Temperaturbereich / *Temperature range*	476,0°C

Kalibriersubstanz / *Calibration substance 500 mg*

Bezeichnung *Material*	chem. Zeichen *Formula*	Reinheit *Purity* [%]	Theoretische Umwandl.-Temperatur *Theoretical Transform.-temperature T [°C]*	Theoretische Umwandl.-enthalpie *Theoretical Transform.-enthalpy* ΔH [J/g]	Chargen - Nr. *Lot No.*	Mindest-Haltbarkeit *Min. Durability* *1	Quellen-angabe *Reference*
Cäsiumchlorid / Caesium chloride	CsCl	99,999	476,0	-17,2	160709	10/2017	[12]

*1 bei Veränderung der angegebenen Werte oder Verdacht auf Kontamination bitte sofort erneuern!
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Literatur:

[12] W.F. Hemminger, H.K. Cammenga: Methoden der Thermischen Analyse, Band XXIV

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NETZSCH-Gerätebau GmbH

Dr. Thomas Denner

Netzsch-Gerätebau GmbH
Wittelsbacherstr. 42
NETZSCH
D-95100 Selb
Quality Assurance

NETZSCH

Werksbescheinigung / *Manufacturer´s Certification*

nach / *in accordance with*

DIN 51007, ASTM E 967 / DIN EN 10 204 - 2.1

DTA/DSC-Kalibrierset für Pt Tiegel
DTA/DSC Calibration Set for Pt crucibles

Ausstelldatum / *Date of Issue:*	21.10.2014
Bestell-Nr. / *Order - No:*	6.223.5-91.2.06
Hersteller-Serien-Nr. / *Series-No:*	14940
Kommission / *Commission:*	216042070
Temperaturbereich / *Temperature range*	668°C

Kalibriersubstanz / *Calibration substance 500 mg*

Bezeichnung *Material*	chem. Zeichen *Formula*	Reinheit *Purity* [%]	Theoretische Umwandl.-Temperatur *Theoretical Transform.-temperature T [°C]*	Theoretische Umwandl.-enthalpie *Theoretical Transform.-enthalpy* ΔH [J/g]	Chargen - Nr. *Lot No.*	Mindest-Haltbarkeit *Min. Durability* *1	Quellen-angabe *Reference*
Kaliumchromat / Potassium chromate	K2CrO4	≥99	668,0	-37,0	12008EH	10/2019	T=[12]; ΔH=[15]

*1 bei Veränderung der angegebenen Werte oder Verdacht auf Kontamination bitte sofort erneuern!
renew please immediately on change of the indicated values or suspicion on contamination!

Literatur:

[7] W. Eysel, K.-H. Breuer: Analytical Calorimetry. Plenum Publ. Corp., 1984
[15] NETZSCH-Gerätebau GmbH

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NETZSCH-Gerätebau GmbH

Dr. Thomas Denner

NETZSCH

Werksbescheinigung / *Manufacturer´s Certification*

nach / *in accordance with*

DIN 51007, ASTM E 967 / DIN EN 10 204 - 2.1

DTA/DSC-Kalibrierset für Pt Tiegel
DTA/DSC Calibration Set for Pt crucibles

Ausstelldatum / *Date of Issue:*	21.10.2014
Bestell-Nr. / *Order - No:*	6.223.5-91.2.07
Hersteller-Serien-Nr. / *Series-No:*	15778
Kommission / *Commission:*	216042070
Temperaturbereich / *Temperature range*	808,0°C

Kalibriersubstanz / *Calibration substance 500 mg*

Bezeichnung *Material*	chem. Zeichen *Formula*	Reinheit *Purity* [%]	Theoretische Umwandl.-Temperatur *Theoretical Transform.-temperature T [°C]*	Theoretische Umwandl.-enthalpie *Theoretical Transform.-enthalpy* ΔH [J/g]	Chargen - Nr. *Lot No.*	Mindest-Haltbarkeit *Min. Durability* *1	Quellen-angabe *Reference*
Bariumcarbonat / Barium carbonate	BaCO3	99,98	808	-94,9	140512	10/2017	T=[7]; ΔH=[2]

*1 bei Veränderung der angegebenen Werte oder Verdacht auf Kontamination bitte sofort erneuern!
renew please immediately on change of the indicated values or suspicion on contamination!

Literatur:

[2] R. Riesen, G. Widmann: Thermoanalyse. A. Hüthig Verlag, Heidelberg, 1984
[7] W. Eysel, K.-H. Breuer: Analytical Calorimetry. Plenum Publ. Corp., 1984

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NETZSCH-Gerätebau GmbH

Dr. Thomas Denner

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: WIPP 350C Baseline 030816.ngb-bsv
Date/Time: 3/8/2016 9:39:37 AM (UTC-7)
End Date/Time: 3/8/2016 10:11:36 AM (UTC-7)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: Correction
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: 350C baseline 030816
Sample name: 350C baseline 030816
Sample Mass: 0 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: baseline
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: WIPP 350 C baseline - 1st attempt

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

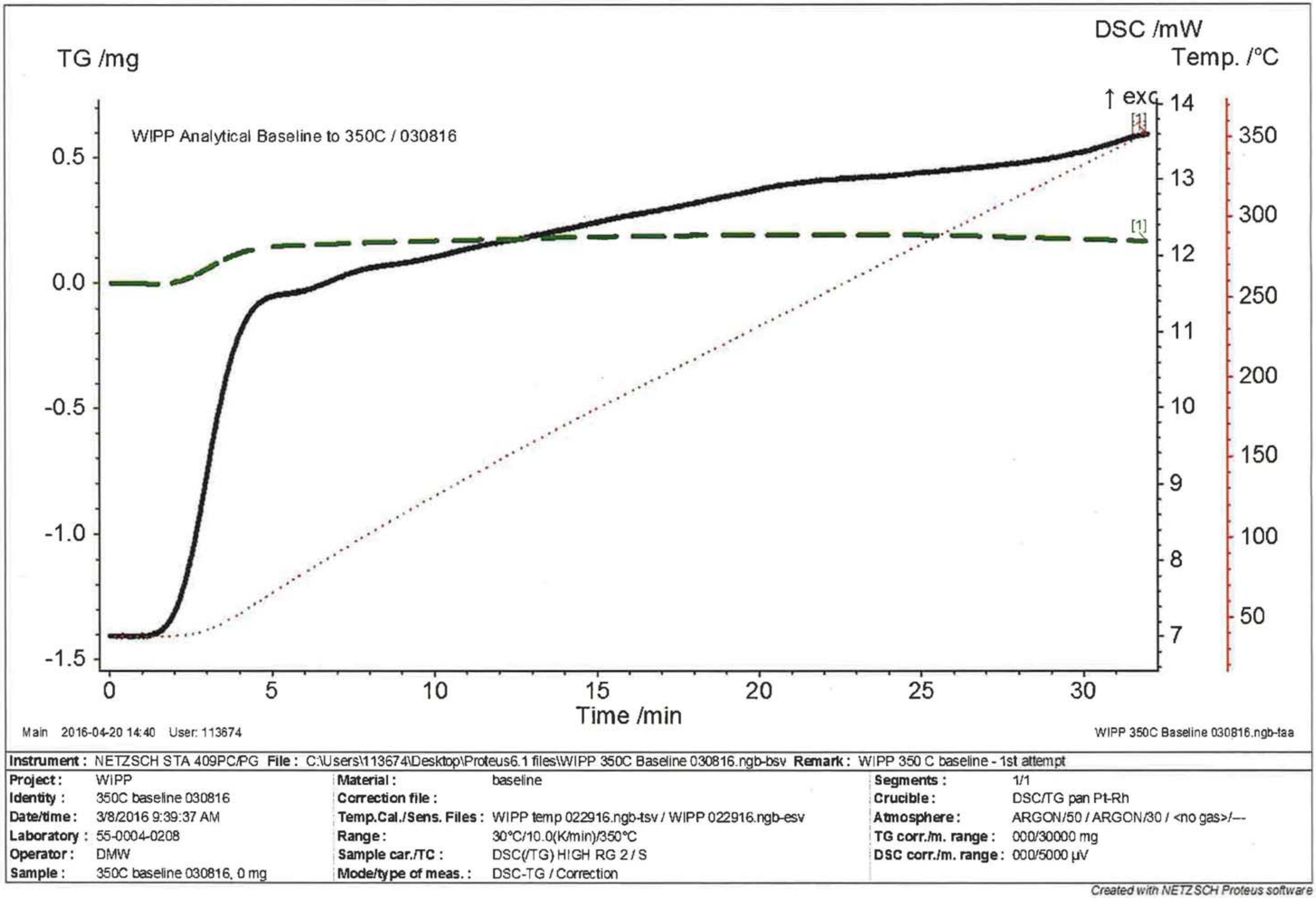
Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0



Main 2016-04-20 14:40 User: 113674

WIPP 350C Baseline 030816.ngb-taa

Instrument : NETZSCH STA 409PC/PG **File :** C:\Users\113674\Desktop\Proteus6.1 files\WIPP 350C Baseline 030816.ngb-bsv **Remark :** WIPP 350 C baseline - 1st attempt

Project :	WIPP	**Material :**	baseline	**Segments :**	1/1
Identity :	350C baseline 030816	**Correction file :**		**Crucible :**	DSC/TG pan Pt-Rh
Date/time :	3/8/2016 9:39:37 AM	**Temp.Cal./Sens. Files :**	WIPP temp 022916.ngb-tsv / WIPP 022916.ngb-esv	**Atmosphere :**	ARGON/50 / ARGON/30 / <no gas>/---
Laboratory :	55-0004-0208	**Range :**	30°C/10.0(K/min)/350°C	**TG corr./m. range :**	000/30000 mg
Operator :	DMW	**Sample car./TC :**	DSC(/TG) HIGH RG 2 / S	**DSC corr./m. range :**	000/5000 µV
Sample :	350C baseline 030816, 0 mg	**Mode/type of meas. :**	DSC-TG / Correction		

Created with NETZSCH Proteus software

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	Pu-Am spike 200 uL dried.ngb-dsv	**Sample identity:**	Pu-Am spike 200 uL dried
Date/Time:	4/15/2016 11:35:13 AM (UTC-6)	**Sample name:**	Pu-Am spike 200 uL dried
End Date/Time:	4/15/2016 12:07:14 PM (UTC-6)	**Sample Mass:**	0.7 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	Pu-Am Nitrate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: Pu-Am spike 200 uL dried in Pt-Rh capsule / weight approximate

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

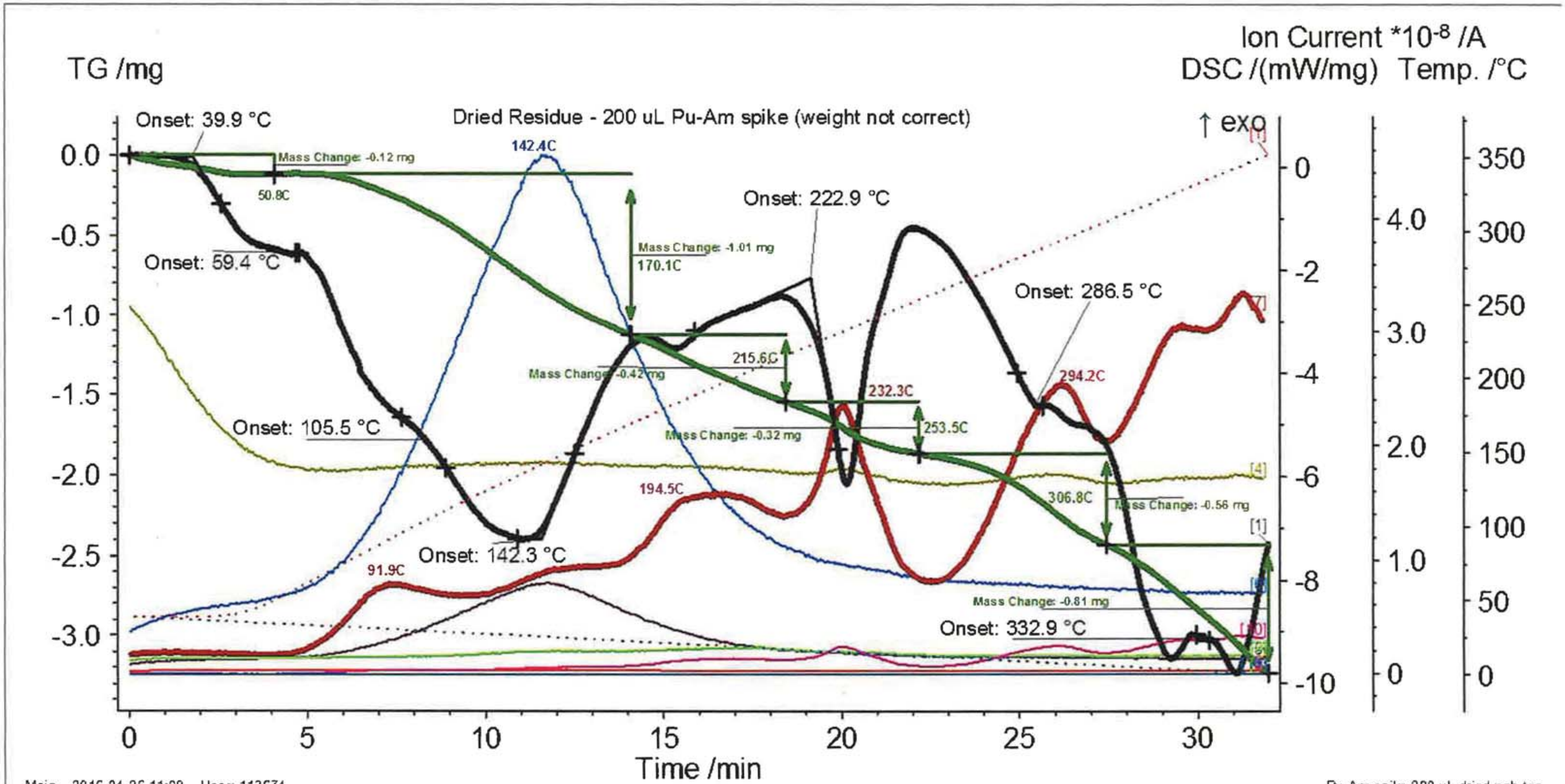
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	Pu-Am spike 200 uL dried.ngb-dsv
Date/Time:	4/15/2016 11:35:13 AM (UTC-6)
End Date/Time:	4/15/2016 12:07:14 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	Pu-Am spike 200 uL dried
Sample name:	Pu-Am spike 200 uL dried
Sample Mass:	0.7 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	Pu-Am Nitrate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: Pu-Am spike 200 uL dried in Pt-Rh capsule / weight approximate

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Mass Change (TG)	-0.12 mg	0.0 min	4.1 min
Mass Change (TG)	-1.01 mg	4.1 min	14.1 min
Mass Change (TG)	-0.42 mg	14.1 min	18.4 min
Mass Change (TG)	-0.32 mg	18.4 min	22.2 min
Mass Change (TG)	-0.56 mg	22.2 min	27.4 min
Mass Change (TG)	-0.81 mg	27.4 min	32.0 min
Onset (DSC)	39.9 °C	0.0 min	4.7 min
Onset (DSC)	59.4 °C	4.7 min	7.7 min
Onset (DSC)	105.5 °C	7.7 min	12.4 min
Value (Temp.)	7.3 min	92.0 °C	
Onset (DSC)	142.3 °C	10.9 min	15.1 min
Onset (DSC)	222.9 °C	15.9 min	20.4 min
Onset (DSC)	286.5 °C	24.9 min	26.1 min
Onset (DSC)	332.9 °C	30.0 min	30.8 min



Main 2016-04-26 11:09 User: 113674

Pu-Am spike 200 uL dried.ngb-taa

[#] Instrument	File	Date	Identity	Sample	M..	S...	Range	Atmosphere	Corr.
[1] STA 409PC/...	Pu-Am spike 200 uL dried.ngb-dsv	2016-04-15	Pu-Am spike 200 uL dri...	Pu-Am spike 200 uL dri...	0.7	1/1	30°C/10.0(K/min)/350...	ARGON/50 / ARGON/30 / <no gas>/...	DSC:020, TG:0...
[2] QMS 403	pu-am spike 200 ul dried_m2.00.imp	2016-04-15	pu-am spike 200 ul dried	Mass 2.00		1/1	40°C/1.2(K/min)/1°C		---
[3] QMS 403	pu-am spike 200 ul dried_m11.98.i...	2016-04-15	pu-am spike 200 ul dried	Mass 11.98		1/1	40°C/1.2(K/min)/1°C		---
[4] QMS 403	pu-am spike 200 ul dried_m15.97.i...	2016-04-15	pu-am spike 200 ul dried	Mass 15.97		1/1	40°C/1.2(K/min)/1°C		---
[5] QMS 403	pu-am spike 200 ul dried_m16.97.i...	2016-04-15	pu-am spike 200 ul dried	Mass 16.97		1/1	40°C/1.2(K/min)/1°C		---
[6] QMS 403	pu-am spike 200 ul dried_m17.97.i...	2016-04-15	pu-am spike 200 ul dried	Mass 17.97		1/1	40°C/1.2(K/min)/1°C		---
[7] QMS 403	pu-am spike 200 ul dried_m30.03.i...	2016-04-15	pu-am spike 200 ul dried	Mass 30.03		1/1	40°C/1.2(K/min)/1°C		---
[9] QMS 403	pu-am spike 200 ul dried_m43.91.i...	2016-04-15	pu-am spike 200 ul dried	Mass 43.91		1/1	40°C/1.2(K/min)/1°C		---
[10] QMS 403	pu-am spike 200 ul dried_m45.91.i...	2016-04-15	pu-am spike 200 ul dried	Mass 45.91		1/1	40°C/1.2(K/min)/1°C		---
[11] QMS 403	pu-am spike 200 ul dried_m47.91.i...	2016-04-15	pu-am spike 200 ul dried	Mass 47.91		1/1	40°C/1.2(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: PuAm Spike 200uL Dried **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-15-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPP TEMP 022916/WIPP 022916	N/A	02-28-17	DMW Initials 113674 Z No. 4-15-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-15-16 Date	
Date & Time Sample Vial Opened	Date: 04/13/16 *(mm / dd / yy)* Time: 09:43 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/29.0	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-15-16 Date	Initials 120527 Z No. 4/15/16 Date

MATL NAME: PuAmSpike 200mL dried **Location:** G220

Sample Weights (Section 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
Date & Time Sample Weighed	**Date:** N/A **Time:** N/A	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-15-16 Date	
Crucible / Pan Tare Wt.	N/A	grams	N/A	DMW Initials 113674 Z No. 4-15-16 Date	Initials 120527 Z No. 4/15/16 Date
Net Sample Weight	Assumed 0.7 mg		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-15-16 Date	Initials 120527 Z No. 4/15/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10:57	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350C Baseline 030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: Pu-Am Spike 200uL dried **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-15-16 Date	Initials 120527 Z No. 4/15/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 04/15/16 Time Started: 11:40 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 40.2	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: Pu-Am Spike 200uL dried.ngb-dsv **ThermoStar** Filename: Pu-AmSpike 200uL dried.MDC	N/A	N/A	DMW Initials 113674 Z No. 4-15-16 Date	
Proteus Data 5) -0.56 253.5-306.8°C 6) -0.81 306.8-350°C	NOTE - in mg, not % **Mass Changes:** 1) -0.12 Temp. Range (RT- 50.8°C) 2) -1.01 Temp. Range (50.8-170.1°C) 3) -0.42 Temp. Range (170.1-215.6°C) 4) -0.32 Temp. Range (215.6-253.5°C) **Total mass Change:** -3.24 mg	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-15-16 Date	Initials 120527 Z No. 4/15/16 Date

DMW 113674 4-15-16

Notes:

MATL NAME: Pu-Am Spike 200mL dried **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H2O/OH Peak T (142.4 °C) 2) NO/NO2 Peak T (91.9, 194.5, 237.3, 294.2 °C) 3) CO2 Peak T (none) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-15-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): N/A = 100 x ____ ÷ ____ 4-15-16 Std. Error Slope	113674 Wt. %	< 0.32	DMW Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-15-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HwM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments: Mass Change entered as mg, not wt %

non standard sample - 200 mL of spike dried in
Pt-Rh DSC pan for ~2 days. Forgot to record tare
weight of pan before adding spike - gross wt. was 0.17678
used 0.7 mg - but was likely closer to 8.0 mg.
DMW 113674 4-15-16

N/A summary
1 - UPC instrument - Cal File # not required
2 - Calib. Thermometer not required
3 - Seal %RH/Temp not required (no seal)
4 - Date & Time Sample weighed - not recorded - in error
5 - Crucible tare wt. not recorded
6 - Moisture Content Eval not required
7 - Profile not required to run to 1100°C
DMW 113674 - 4/15/16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-1 030716A.ngb-dsv	**Sample identity:**	SFWB8-15-1 030716A
Date/Time:	3/9/2016 9:21:55 AM (UTC-7)	**Sample name:**	SFWB8-15-1 030716A
End Date/Time:	3/9/2016 9:53:57 AM (UTC-7)	**Sample Mass:**	38.47 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SURROGATE BY ITSELF - 1ST RUN

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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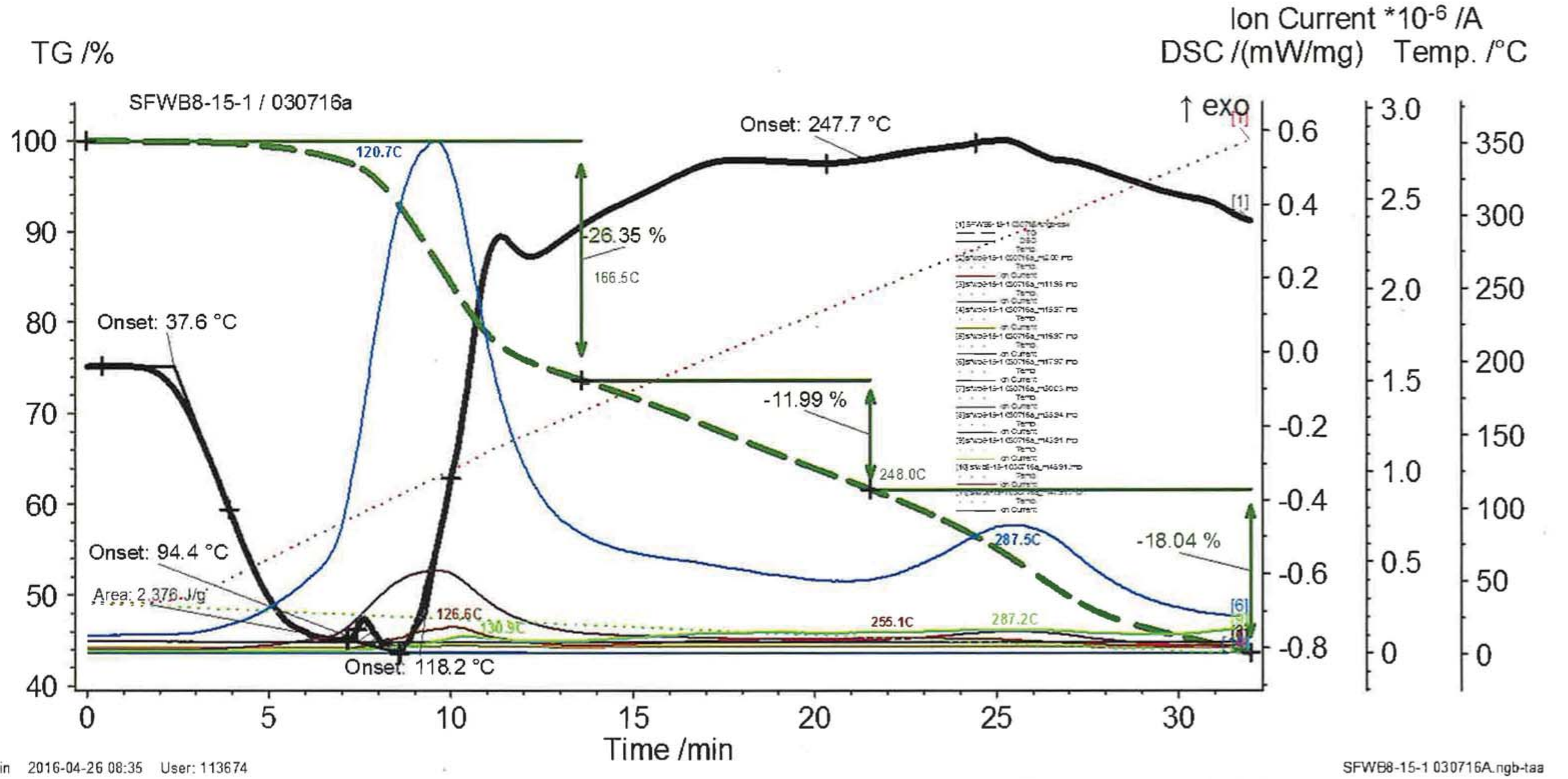
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-1 030716A.ngb-dsv
Date/Time:	3/9/2016 9:21:55 AM (UTC-7)
End Date/Time:	3/9/2016 9:53:57 AM (UTC-7)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-1 030716A
Sample name:	SFWB8-15-1 030716A
Sample Mass:	38.47 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SURROGATE BY ITSELF - 1ST RUN

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	37.6 °C	0.4 min	5.9 min
Onset (DSC)	94.4 °C	7.2 min	7.8 min
Area (DSC),o	2.376 J/g	7.2 min	8.6 min
Onset (DSC)	118.2 °C	8.6 min	10.1 min
Onset (DSC)	247.7 °C	20.3 min	27.6 min



Main 2016-04-26 08:35 User: 113674

SFWB8-15-1 030716A.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 030716A.ngb-dsv	2016-03-09	SFWB8-15-1 030716A	SFWB8-15-1 030716A	38.47	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 030716a_m2.00.imp	2016-03-09	sfwb8-15-1 030716a	Mass 2.00		1/1	36°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 030716a_m11.98.imp	2016-03-09	sfwb8-15-1 030716a	Mass 11.98		1/1	36°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 030716a_m15.97.imp	2016-03-09	sfwb8-15-1 030716a	Mass 15.97		1/1	36°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 030716a_m16.97.imp	2016-03-09	sfwb8-15-1 030716a	Mass 16.97		1/1	36°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 030716a_m17.97.imp	2016-03-09	sfwb8-15-1 030716a	Mass 17.97		1/1	36°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 030716a_m30.03.imp	2016-03-09	sfwb8-15-1 030716a	Mass 30.03		1/1	36°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 030716a_m35.94.imp	2016-03-09	sfwb8-15-1 030716a	Mass 35.94		1/1	36°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 030716a_m43.91.imp	2016-03-09	sfwb8-15-1 030716a	Mass 43.91		1/1	36°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 030716a_m45.91.imp	2016-03-09	sfwb8-15-1 030716a	Mass 45.91		1/1	36°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 030716a_m47.91.imp	2016-03-09	sfwb8-15-1 030716a	Mass 47.91		1/1	36°C/1.1(K/min)/1°C		---

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MATL NAME: SFWB8-15-1 A **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113679 Z No. 3-9-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTemp022916/WIPP022916	N/A	02-28-17	DMW Initials 113674 Z No. 3-9-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113679 Z No. 3-9-16 Date
Date & Time Sample Vial Opened	**Date:** 03/09/16 *(mm / dd / yy)* **Time:** 08:53 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.5/29.9°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113679 Z No. 3.9.16 Date Initials 120527 Z No. 3/9/16 Date

MATL NAME: SFWB8-15-1 A **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-9-16 Time: 08:56	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Crucible / Pan Tare Wt.	0.16909	grams	N/A	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
Net Sample Weight	0.03847		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 8 : 59	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350C Baseline 030816.ngb-bsv Filename: ____ *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 A **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-9-16 Time Started: 09:21 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 35.4	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP SFWB8-15-1 030716A.ngb-dsV **Netzsch Measure** Filename: SFWB8-15-1 030716A.MDC **ThermoStar** Filename:	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Proteus Data	**Mass Changes:** DMW 113674 4-28-16 1) -26.35 ~~-26.53~~ Temp. Range (RT-166.5°C) 2) -11.99 Temp. Range (166.5-248.0°C) 3) -18.04 Temp. Range (248.0-350°C) 4) ______ Temp. Range (__________) **Total mass Change:** ~~-56.56 %~~ DMW 113674 4-28-16 -56.38 %	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date

Notes:

MATL NAME: SFWB8-15-1A **Location:** G270

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (120.7, 287.5°C) 2) CO_2 Peak T (130.9, 287.2°C) 3) NO/NO_2 Peak T (126.6, 255.1°C) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-9-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): N/A = 100 x ____ ÷ ____ Std. Error Slope DMW 113674 3-9-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-9-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1- Cal. File # not required for OPC instruments.
2. Calib. Thermometer for water bath is not required
3. Profile not required to go to 1100°C
4- Seal %RH & Temp measurement not required
5- Moisture Content not required
DMW 113674 3-9-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-1 030716B.ngb-dsv	**Sample identity:**	SFWB8-15-1 030716B
Date/Time:	3/9/2016 11:22:05 AM (UTC-7)	**Sample name:**	SFWB8-15-1 030716B
End Date/Time:	3/9/2016 11:54:08 AM (UTC-7)	**Sample Mass:**	7.98 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-1 030716B - 2nd run

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument:	NETZSCH STA 409PC/PG	**TG TG Range:**	30000 mg
Project:	WIPP	**Sample identity:**	SFWB8-15-1 030716B
Filename:	SFWB8-15-1 030716B.ngb-dsv	**Sample name:**	SFWB8-15-1 030716B
Date/Time:	3/9/2016 11:22:05 AM (UTC-7)	**Sample Mass:**	7.98 mg
End Date/Time:	3/9/2016 11:54:08 AM (UTC-7)	**Crucible:**	DSC/TG pan Pt-Rh
Laboratory:	55-0004-0208	**Crucible Mass:**	0 mg
Operator:	DMW	**Reference name:**	empty
Mode:	DSC-TG	**Reference Mass:**	0 mg
Measurement Type:	sample with correction	**Reference Crucible Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Material:**	WIPP surrogate
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Sample determination mode:**	Manual
Sensitivity:	WIPP 022916.ngb-esv	**Residuum measurement:**	Not possible
Crucible:	DSC/TG pan Pt-Rh	**Atmosphere:**	ARGON/50 / ARGON/30 / <no gas>/---
DSC DSC Range:	5000 µV		

Remark: SFWB8-15-1 030716B - 2nd run

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	42.2 °C	0.9 min	6.7 min
Onset (DSC)	84.3 °C	6.4 min	7.7 min
Area (DSC),o	8.426 J/g	6.4 min	7.5 min
Onset (DSC)	105.9 °C	8.0 min	8.8 min
Peak (DSC)	135.4 °C/0.8955 mW/mg	9.3 min	12.2 min
Onset (DSC)	161.7 °C	12.1 min	16.3 min

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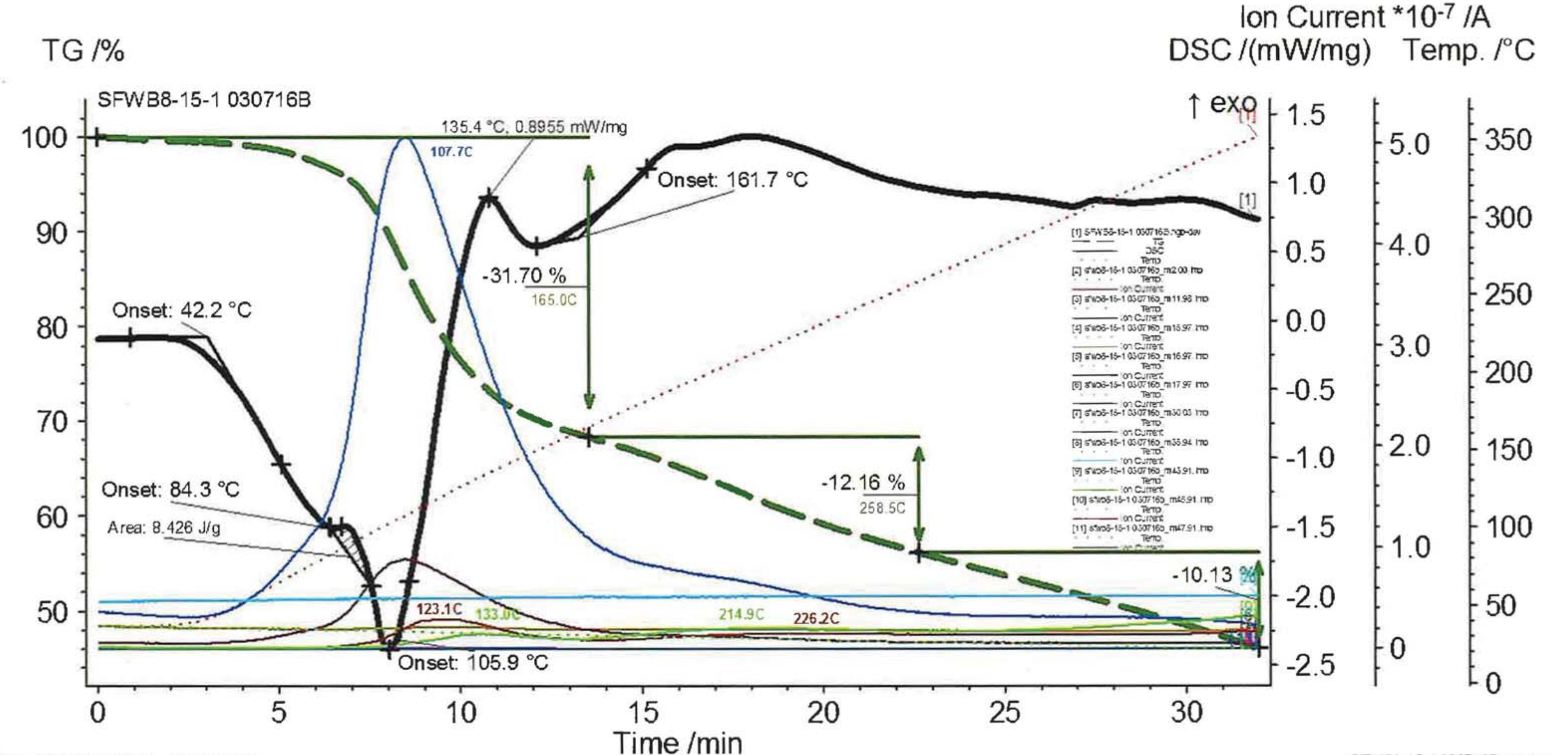

Main 2016-04-26 08:47 User: 113674

SFWB8-15-1 030716B.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mas...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 030716B.ngb-dsv	2016-03-09	SFWB8-15-1 030716B	SFWB8-15-1 030716B	7.98	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 030716b_m2.00.imp	2016-03-09	sfwb8-15-1 030716b	Mass 2.00		1/1	38°C/0.5(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 030716b_m11.98.imp	2016-03-09	sfwb8-15-1 030716b	Mass 11.98		1/1	38°C/0.5(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 030716b_m15.97.imp	2016-03-09	sfwb8-15-1 030716b	Mass 15.97		1/1	38°C/0.5(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 030716b_m16.97.imp	2016-03-09	sfwb8-15-1 030716b	Mass 16.97		1/1	38°C/0.5(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 030716b_m17.97.imp	2016-03-09	sfwb8-15-1 030716b	Mass 17.97		1/1	38°C/0.5(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 030716b_m30.03.imp	2016-03-09	sfwb8-15-1 030716b	Mass 30.03		1/1	38°C/0.5(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 030716b_m35.94.imp	2016-03-09	sfwb8-15-1 030716b	Mass 35.94		1/1	38°C/0.5(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 030716b_m43.91.imp	2016-03-09	sfwb8-15-1 030716b	Mass 43.91		1/1	38°C/0.5(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 030716b_m45.91.imp	2016-03-09	sfwb8-15-1 030716b	Mass 45.91		1/1	38°C/0.5(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 030716b_m47.91.imp	2016-03-09	sfwb8-15-1 030716b	Mass 47.91		1/1	38°C/0.5(K/min)/1°C		---

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MATL NAME: SFWB8-15-1 B **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-9-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** wipptemp022916/wspp022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-9-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Date & Time Sample Vial Opened	Date: 03/09/16 *(mm / dd / yy)* Time: 11:09 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.5/25.9°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/14 Date

MATL NAME: SFWB8-15-1 B **Location:** G120

Sample Weights (Section 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
Date & Time Sample Weighed	**Date:** 03/09/16 **Time:** 11:11	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Crucible / Pan Tare Wt.	0.16801	grams	N/A	DMW Initials 113674 Z No. 3-5-16 Date	Initials 120527 Z No. 3/9/16 Date
Net Sample Weight	0.00798		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 08 : 59	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350C Baseline 030816.ngb-bsr Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 B **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-9-16 Time Started: 11:22 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 37.5	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-1 030716B.ngb-dsv **ThermoStar** Filename: SFWB8-15-1 030716B.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Proteus Data	**Mass Changes:** 1) -31.70 Temp. Range (RT-165°C) 2) -12.16 Temp. Range (165-258.5°C) 3) -10.13 Temp. Range (258.5-350°C) 4) ______ Temp. Range (__________) **Total mass Change:** -53.99%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date

Notes:

MATL NAME: SFWB8-15-1 B **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H_2O Peak T (107.7°C) 2) CO_2 Peak T (133.0, 213.9°C) 3) NO/NO_2 Peak T (123.1, 226.2°C) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-9-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __/ 113674 3-9-16 mm dd yy % Error (RSD, 1s): DMW N/A = 100 x ____ ÷ ____ Std. Error Slope	Wt. %	< 0.32	~~DMW~~ Initials ~~H~~ DMW Z No. ____ Date	DMW 113674 3.9.16 Initials 113674 3.9.16 Z No. ____ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-9-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1- Cal File # for UPC not required
2- Calibrated Thermometer for water Chiller not required
3- Temp. Profile not required to go to 1100°C
4- Seal %RH/Temp. measurement not required
5- Moisture Content not required
DMW 13674 3-9-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-1 030716C.ngb-dsv	**Sample identity:**	SFWB8-15-1 030716C
Date/Time:	3/9/2016 2:22:56 PM (UTC-7)	**Sample name:**	SFWB8-15-1 030716C
End Date/Time:	3/9/2016 2:54:57 PM (UTC-7)	**Sample Mass:**	31.69 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: WIPP Surrogate SFWB8-15-1 030716 C

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

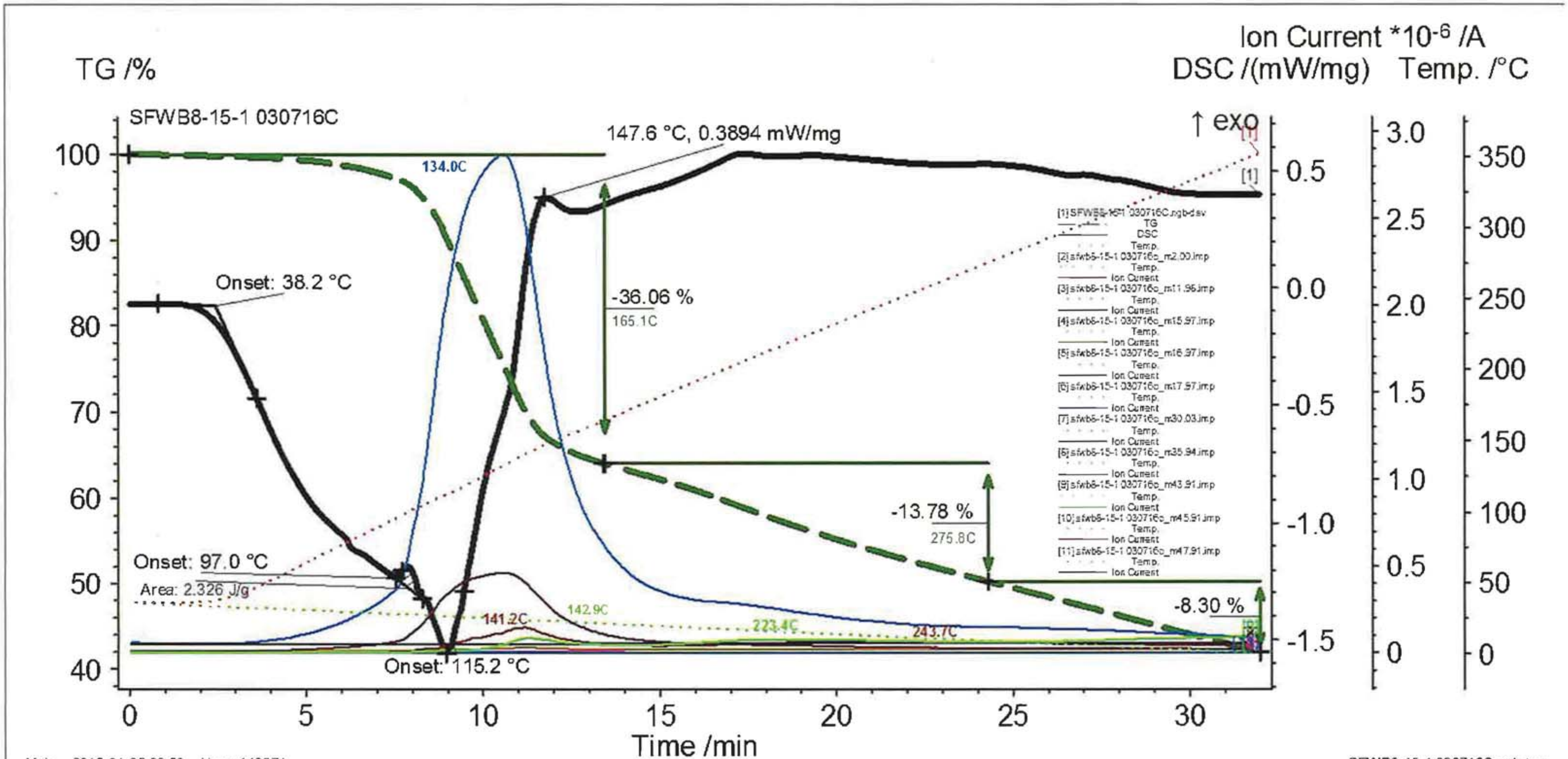
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-1 030716C.ngb-dsv
Date/Time: 3/9/2016 2:22:56 PM (UTC-7)
End Date/Time: 3/9/2016 2:54:57 PM (UTC-7)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-1 030716C
Sample name: SFWB8-15-1 030716C
Sample Mass: 31.69 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: WIPP Surrogate SFWB8-15-1 030716 C

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Area (DSC),o	2.326 J/g	7.5 min	8.3 min
Onset (DSC)	38.2 °C	0.8 min	5.8 min
Onset (DSC)	97.0 °C	7.5 min	8.1 min
Onset (DSC)	115.2 °C	9.0 min	10.5 min
Peak (DSC)	147.6 °C/0.3894 mW/mg	8.9 min	12.9 min



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SFWB8-15-1 030716C.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 030716C.ngb-dsv	2016-03-09	SFWB8-15-1 030716C	SFWB8-15-1 030716C	31.69	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 030716c_m2.00.imp	2016-03-09	sfwb8-15-1 030716c	Mass 2.00		1/1	36°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 030716c_m11.98.imp	2016-03-09	sfwb8-15-1 030716c	Mass 11.98		1/1	36°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 030716c_m15.97.imp	2016-03-09	sfwb8-15-1 030716c	Mass 15.97		1/1	36°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 030716c_m16.97.imp	2016-03-09	sfwb8-15-1 030716c	Mass 16.97		1/1	36°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 030716c_m17.97.imp	2016-03-09	sfwb8-15-1 030716c	Mass 17.97		1/1	36°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 030716c_m30.03.imp	2016-03-09	sfwb8-15-1 030716c	Mass 30.03		1/1	36°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 030716c_m35.94.imp	2016-03-09	sfwb8-15-1 030716c	Mass 35.94		1/1	36°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 030716c_m43.91.imp	2016-03-09	sfwb8-15-1 030716c	Mass 43.91		1/1	36°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 030716c_m45.91.imp	2016-03-09	sfwb8-15-1 030716c	Mass 45.91		1/1	36°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 030716c_m47.91.imp	2016-03-09	sfwb8-15-1 030716c	Mass 47.91		1/1	36°C/1.1(K/min)/1°C		---

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MATL NAME: SFWB8-15-1C **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-9-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPP TEMP 022916 / WIPP022916	N/A	02-28-17	DMW Initials 113674 Z No. 3-9-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / Pt [✓ DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / [✓ No] N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Date & Time Sample Vial Opened	Date: 03-09-16 *(mm / dd / yy)* Time: 14:04 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.5 / 25.9 °C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date

MATL NAME: SFWB8-15-1 C **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	**Date:** 03/09/16 **Time:** 14:06	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Crucible / Pan Tare Wt.	0.16748	grams	N/A	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
Net Sample Weight	0.03169		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 08 : 59	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350C Baseline030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 C **Location:** 6220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 03-09-16 Time Started: 14:22 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.3°C	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-1 030716C.ngb-dsv **ThermoStar** Filename: SFWB8-15-1 030716C.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-9-16 Date	
Proteus Data	**Mass Changes:** 1) -36.06 Temp. Range (RT-165.1°) 2) -13.78 Temp. Range (165.1-275.8°)C 3) -8.30 Temp. Range (275.8-350°C) 4) ______ Temp. Range (__________) **Total mass Change:** -58.14%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-9-16 Date	Initials 120527 Z No. 3/9/16 Date

Notes:

MATL NAME: SFWB8-15-1C **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H₂O Peak T (134.0°C) 2) CO_2 Peak T (142.9/223.4°C 3) NO/NO_2 Peak T (141.2/243.7°C) 4) ______ Peak T ( ) 5) ______ Peak T ( )	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-9-16 Date	
Total Moisture (H_2O) N/A	Total Moisture = ______ mg ______ Wt % 3-9-16 Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): DMW 113674 ______ = 100 x ______ ÷ ______ Std. Error Slope	Wt. %	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-9-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A summary
1- Cal. File # not required for UPC instrumentation
2- Calibrated Thermometer for Chiller Water not required
3- Temp. Profile not required to go to 1100°C
4- Seal %RH/Temp meas. not required
5- Moisture content NOT REQUIRED
DMW 113674 3-9-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-1 ACID A.ngb-dsv	**Sample identity:**	SFWB8-15-1 ACID A
Date/Time:	3/10/2016 9:42:24 AM (UTC-7)	**Sample name:**	SFWB8-15-1 ACID A
End Date/Time:	3/10/2016 10:14:25 AM (UTC-7)	**Sample Mass:**	26.63 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: surrogate + acid: first run

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-1 ACID A.ngb-dsv
Date/Time:	3/10/2016 9:42:24 AM (UTC-7)
End Date/Time:	3/10/2016 10:14:25 AM (UTC-7)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-1 ACID A
Sample name:	SFWB8-15-1 ACID A
Sample Mass:	26.63 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: surrogate + acid: first run

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	37.9 °C	0.4 min	5.6 min
Onset (DSC)	90.5 °C	6.6 min	7.9 min
Area (DSC),o	8.594 J/g	7.0 min	8.1 min
Onset (DSC)	108.4 °C	8.4 min	8.9 min
Peak (DSC)	141.8 °C/0.4595 mW/mg	9.2 min	12.4 min

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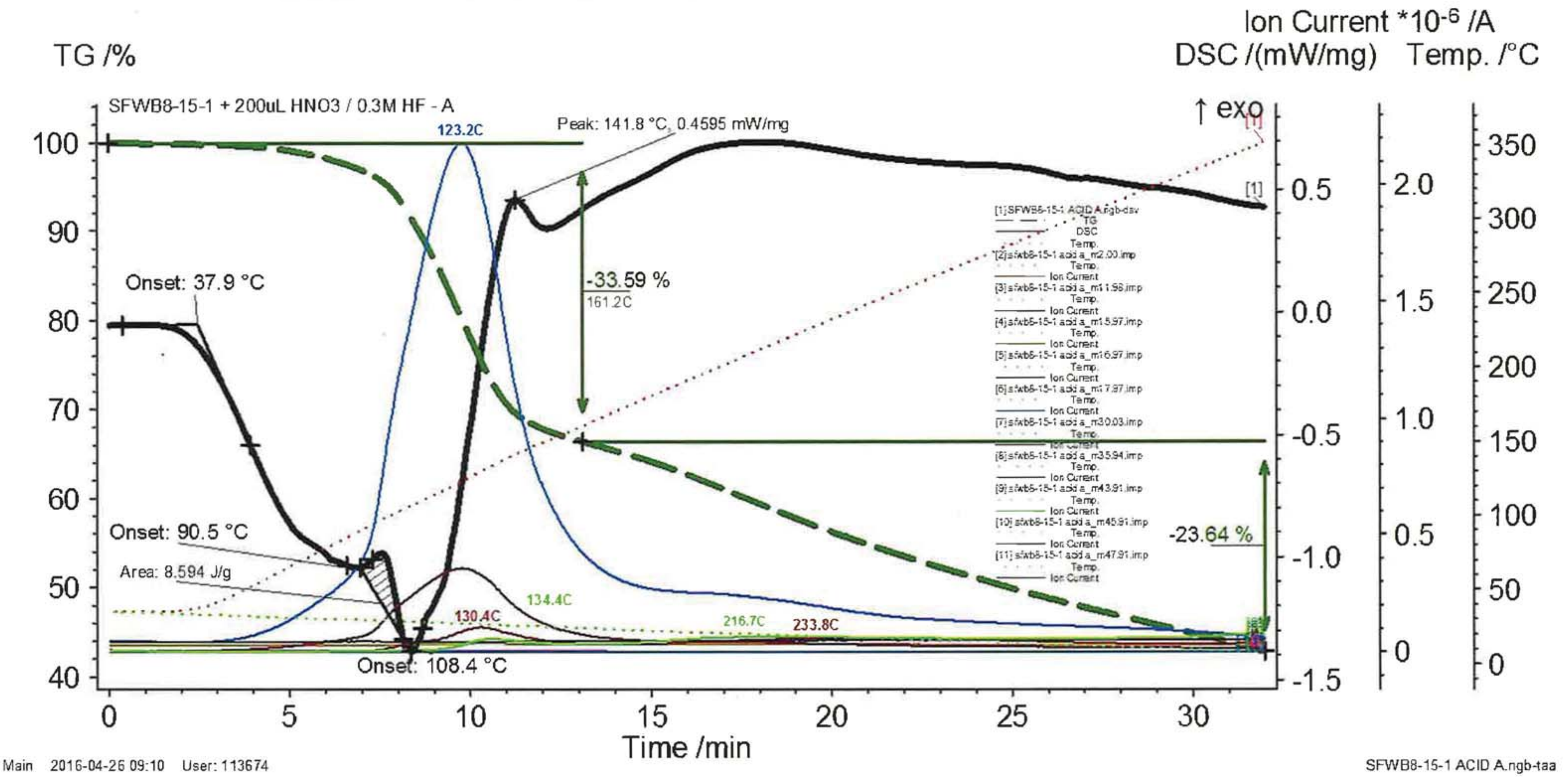

Main 2016-04-26 09:10 User: 113674

SFWB8-15-1 ACID A.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 ACID A.ngb-dsv	2016-03-10	SFWB8-15-1 ACID A	SFWB8-15-1 ACID A	26.63	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 acid a_m2.00.imp	2016-03-10	sfwb8-15-1 acid a	Mass 2.00		1/1	36°C/0.8(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 acid a_m11.98.imp	2016-03-10	sfwb8-15-1 acid a	Mass 11.98		1/1	36°C/0.8(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 acid a_m15.97.imp	2016-03-10	sfwb8-15-1 acid a	Mass 15.97		1/1	36°C/0.8(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 acid a_m16.97.imp	2016-03-10	sfwb8-15-1 acid a	Mass 16.97		1/1	36°C/0.8(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 acid a_m17.97.imp	2016-03-10	sfwb8-15-1 acid a	Mass 17.97		1/1	36°C/0.8(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 acid a_m30.03.imp	2016-03-10	sfwb8-15-1 acid a	Mass 30.03		1/1	36°C/0.8(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 acid a_m35.94.imp	2016-03-10	sfwb8-15-1 acid a	Mass 35.94		1/1	36°C/0.8(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 acid a_m43.91.imp	2016-03-10	sfwb8-15-1 acid a	Mass 43.91		1/1	36°C/0.8(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 acid a_m45.91.imp	2016-03-10	sfwb8-15-1 acid a	Mass 45.91		1/1	36°C/0.8(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 acid a_m47.91.imp	2016-03-10	sfwb8-15-1 acid a	Mass 47.91		1/1	36°C/0.8(K/min)/1°C		---

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MATL NAME: SFW B8-15-1 ACID A **Location:** G220

Thermal Analysis Data Sheet

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DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-10-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WSPPTEMP022916/WSPP022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-10-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	
Date & Time Sample Vial Opened	Date: 03/10/16 *(mm / dd / yy)* Time: 09:24 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/28.5°	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date

MATL NAME: SFWB8-15-1 ACID A **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-10-16 Time: 09:27	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-10-16 Date	
Crucible / Pan Tare Wt.	0.16648	grams	N/A	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date
Net Sample Weight	0.02663		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 09:20	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP 350C Baseline 030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 ACID A **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 03-10-16 Time Started: 09:46 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 35.5	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-1 ACID A.ngb-dsv **ThermoStar** Filename: SFWB8-15-1 ACIDA.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	
Proteus Data	**Mass Changes:** 1) -33.59 Temp. Range (RT-161.2°C) 2) -23.64 Temp. Range (161.2-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -57.23%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date

Notes:

MATL NAME: SFWB 8-15-1 ACID A **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (123.2°C) 2) CO_2 Peak T (134.4, 216.7°C) 3) NO/NO_2 Peak T (130.4, 233.8°C) 4) ______ Peak T (__________) 5) ______ Peak T (__________)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-10-16 Date	
Total Moisture (H_2O)	Total Moisture = __________ mg __________ Wt % Date of most recent calibration: ___/___/___ 3-10-16 mm dd yy 113674 % Error (RSD, 1s): DMW = 100 x ________ ÷ N/A ________ Std. Error Slope	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-10-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 395012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary

1- Cal. File # for VPG instrument is not required

2- Calib. Thermometer for water chiller not required

3- Temp. Profile not required to go to 1100°C

4- Seal %RH/Temp measurement not required

5- Moisture content not required

DMW 113674 3-10-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-1 ACID B.ngb-dsv
Date/Time: 3/10/2016 11:23:30 AM (UTC-7)
End Date/Time: 3/10/2016 11:55:30 AM (UTC-7)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-1 ACID B
Sample name: SFWB8-15-1 ACID B
Sample Mass: 14.18 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-1 ACID B

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

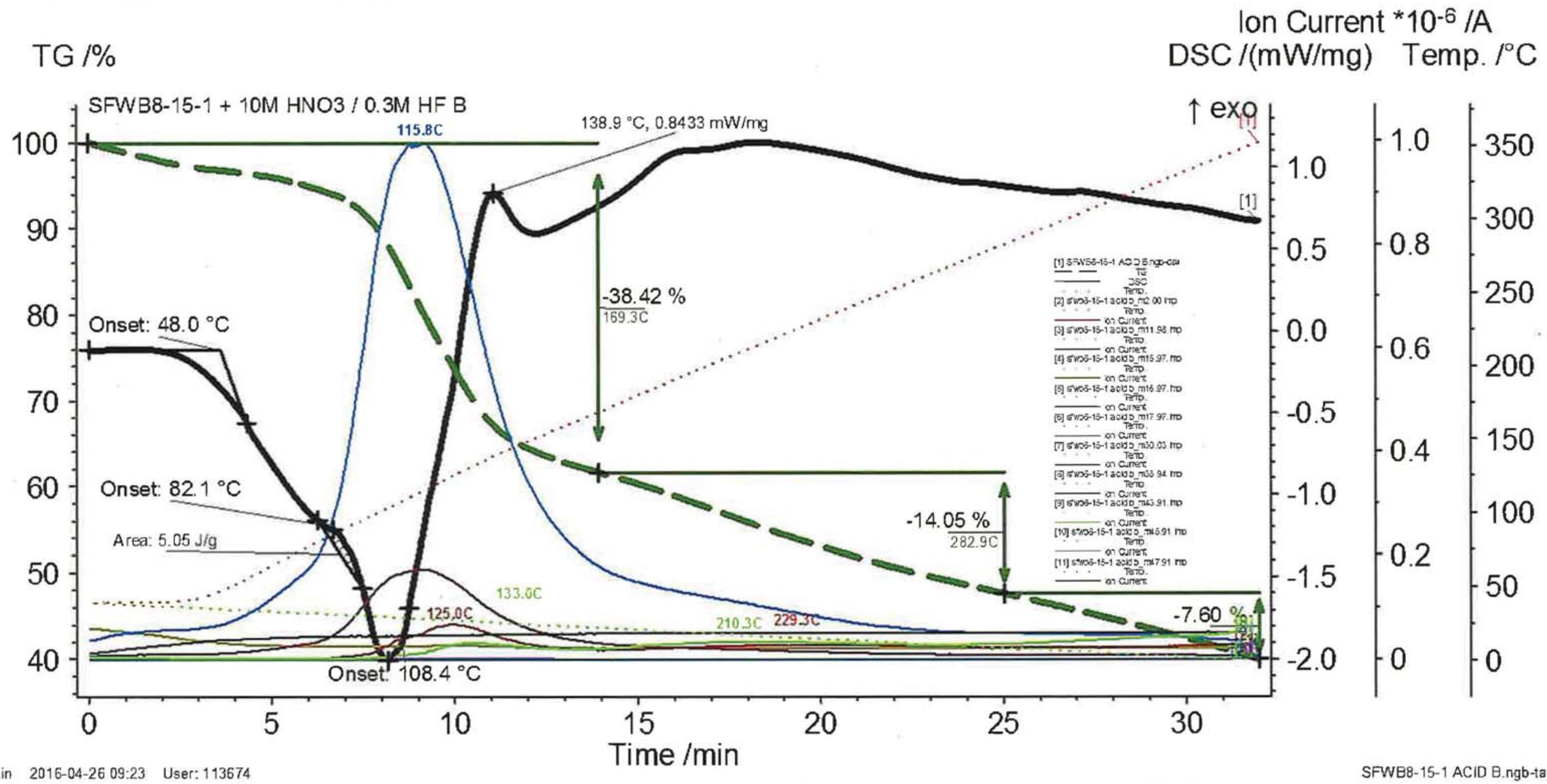
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-1 ACID B.ngb-dsv
Date/Time:	3/10/2016 11:23:30 AM (UTC-7)
End Date/Time:	3/10/2016 11:55:30 AM (UTC-7)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-1 ACID B
Sample name:	SFWB8-15-1 ACID B
Sample Mass:	14.18 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-1 ACID B

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	48.0 °C	0.0 min	6.7 min
Onset (DSC)	82.1 °C	6.3 min	7.0 min
Area (DSC),o	5.05 J/g	6.3 min	7.5 min
Onset (DSC)	108.4 °C	8.2 min	9.0 min
Peak (DSC)	138.9 °C/0.8433 mW/mg	9.6 min	12.1 min



Main 2016-04-26 09:23 User: 113674

SFWB8-15-1 ACID B.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 ACID B.ngb-dsv	2016-03-10	SFWB8-15-1 ACID B	SFWB8-15-1 ACID B	14.18	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 acid b_m2.00.imp	2016-03-10	sfwb8-15-1 acid b	Mass 2.00		1/1	40°C/1.2(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 acid b_m11.98.imp	2016-03-10	sfwb8-15-1 acid b	Mass 11.98		1/1	40°C/1.2(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 acid b_m15.97.imp	2016-03-10	sfwb8-15-1 acid b	Mass 15.97		1/1	40°C/1.2(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 acid b_m16.97.imp	2016-03-10	sfwb8-15-1 acid b	Mass 16.97		1/1	40°C/1.2(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 acid b_m17.97.imp	2016-03-10	sfwb8-15-1 acid b	Mass 17.97		1/1	40°C/1.2(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 acid b_m30.03.imp	2016-03-10	sfwb8-15-1 acid b	Mass 30.03		1/1	40°C/1.2(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 acid b_m35.94.imp	2016-03-10	sfwb8-15-1 acid b	Mass 35.94		1/1	40°C/1.2(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 acid b_m43.91.imp	2016-03-10	sfwb8-15-1 acid b	Mass 43.91		1/1	40°C/1.2(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 acid b_m45.91.imp	2016-03-10	sfwb8-15-1 acid b	Mass 45.91		1/1	40°C/1.2(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 acid b_m47.91.imp	2016-03-10	sfwb8-15-1 acid b	Mass 47.91		1/1	40°C/1.2(K/min)/1°C		---

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MATL NAME: SFW88-15-1 ACID B **Location:** G220

Thermal Analysis Data Sheet

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DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-10-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WippTemp022916/WIPP022916	N/A	02-28-17	DMW Initials 113674 Z No. 3-10-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date
Date & Time Sample Vial Opened	Date: 03/10/16 *(mm / dd / yy)* Time: 11:19 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6%/28.5°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-10-16 Date Initials 120527 Z No. 3/10/16 Date

MATL NAME: SFWB8-15-1 ACID B **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
Date & Time Sample Weighed	**Date:** 03-10-16 **Time:** 11:19	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-10-16 Date
Crucible / Pan Tare Wt.	0.16960	grams	N/A	DMW Initials 113674 Z No. 3-10-16 Date
Net Sample Weight	0.02418		>3 g, < 18 g	Initials 120527 Z No. 3/10/16 Date

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 DMW 113674 3-10-16 Gas Flow 2: ~~80~~ 30 Time gas flows turned on: 09 : 20	hh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350C Baseline030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 ACID B **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials	Initials
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 03-10-16 Time Started: 11:23 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A	113674 Z No. 3-10-16 Date	120527 Z No. 3/10/16 Date
ThermoStar Reference	Sample Temp. at Start: 39.8	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-1 ACID B.ngb-dsv **ThermoStar** Filename: SFWB8-15-1 ACIDB.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	
Proteus Data	**Mass Changes:** 1) -38.42 Temp. Range (RT-169.3°C) 2) -14.05 Temp. Range (169.3-282.9 °C) 3) -7.60 Temp. Range (282.9-350°C) 4) ______ Temp. Range (__________) **Total mass Change:** -60.07%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date

Notes:

MATL NAME: SFWB8-15-1 ACID B **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (115.8°C) 2) CO_2 Peak T (133.0/210.3°C) 3) NO/NO_2 Peak T (125.0/229.3°C) 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-10-16 Date	
Total Moisture (H_2O) N/A	Total Moisture = ______ mg ______ Wt % 3-10-16 Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): 113674 DMW ______ = 100 x ______ ÷ ______ Std. Error Slope	Wt. %	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-10-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/21/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary

1- Cal File# for UPC instrument is not required

2- Calib. Thermometer not required for water chiller

3- Profile not required to go to 1100°C

4- %RH - Temp of Seal - not required

5- Moisture content not required

DMW

113674 3-10-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-1 ACID C.ngb-dsv	**Sample identity:**	SFWB8-15-1 ACID C
Date/Time:	3/10/2016 2:11:42 PM (UTC-7)	**Sample name:**	SFWB8-15-1 ACID C
End Date/Time:	3/10/2016 2:43:43 PM (UTC-7)	**Sample Mass:**	20.39 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SURROGATE WITH ACID 3RD RUN

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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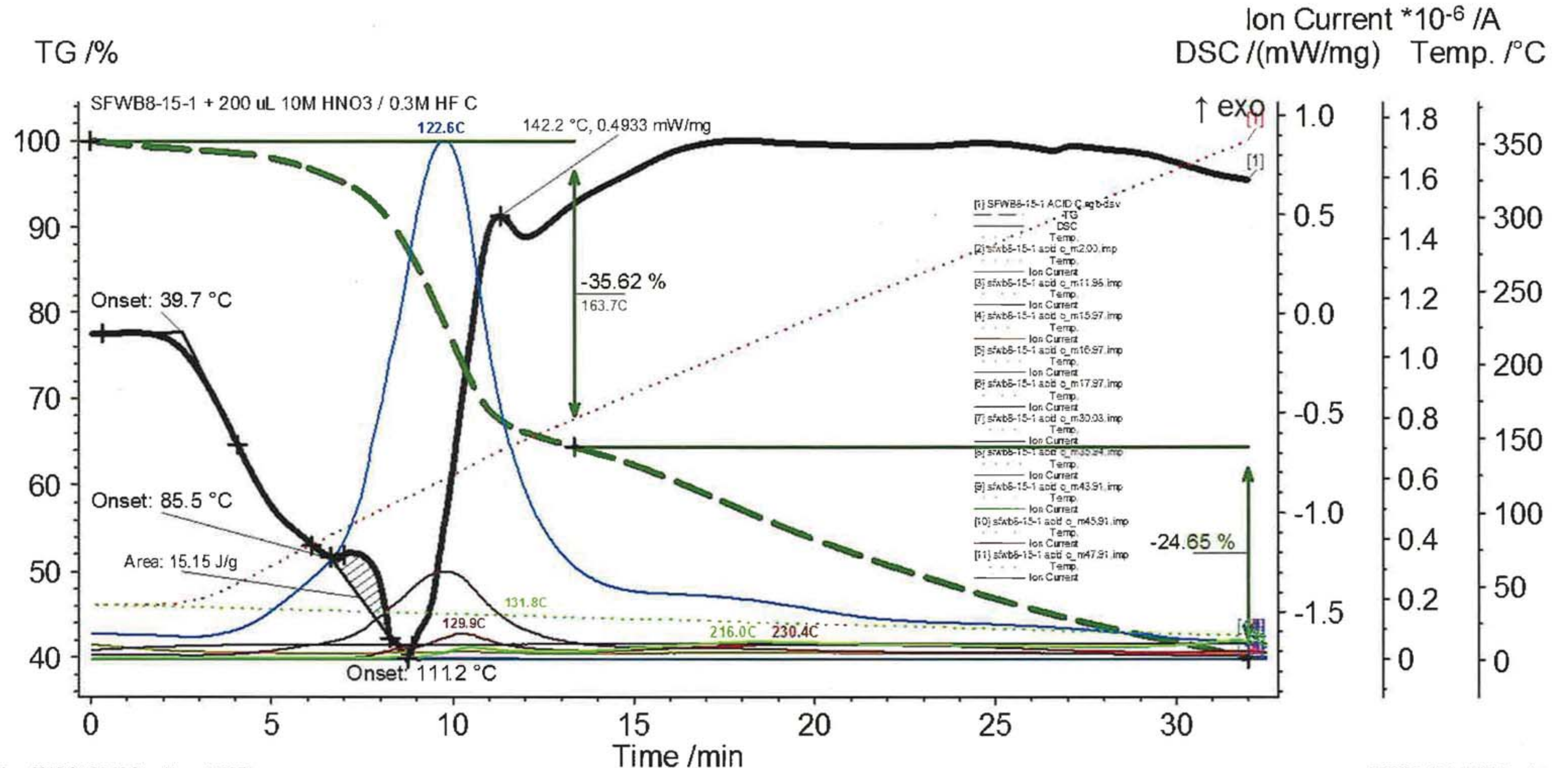
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-1 ACID C.ngb-dsv
Date/Time:	3/10/2016 2:11:42 PM (UTC-7)
End Date/Time:	3/10/2016 2:43:43 PM (UTC-7)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-1 ACID C
Sample name:	SFWB8-15-1 ACID C
Sample Mass:	20.39 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SURROGATE WITH ACID 3RD RUN

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	39.7 °C	0.3 min	5.6 min
Onset (DSC)	85.5 °C	6.1 min	8.2 min
Area (DSC),o	15.15 J/g	6.6 min	8.3 min
Onset (DSC)	111.2 °C	8.8 min	9.2 min
Peak (DSC)	142.2 °C/0.4933 mW/mg	10.0 min	12.0 min



Main 2016-04-26 09:34 User: 113674

SFWB8-15-1 ACID C.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 ACID C.ngb-dsv	2016-03-10	SFWB8-15-1 ACID C	SFWB8-15-1 ACID C	20.39	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 acid c_m2.00.imp	2016-03-10	sfwb8-15-1 acid c	Mass 2.00		1/1	38°C/0.6(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 acid c_m11.98.imp	2016-03-10	sfwb8-15-1 acid c	Mass 11.98		1/1	38°C/0.6(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 acid c_m15.97.imp	2016-03-10	sfwb8-15-1 acid c	Mass 15.97		1/1	38°C/0.6(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 acid c_m16.97.imp	2016-03-10	sfwb8-15-1 acid c	Mass 16.97		1/1	38°C/0.6(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 acid c_m17.97.imp	2016-03-10	sfwb8-15-1 acid c	Mass 17.97		1/1	38°C/0.6(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 acid c_m30.03.imp	2016-03-10	sfwb8-15-1 acid c	Mass 30.03		1/1	38°C/0.6(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 acid c_m35.94.imp	2016-03-10	sfwb8-15-1 acid c	Mass 35.94		1/1	38°C/0.6(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 acid c_m43.91.imp	2016-03-10	sfwb8-15-1 acid c	Mass 43.91		1/1	38°C/0.6(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 acid c_m45.91.imp	2016-03-10	sfwb8-15-1 acid c	Mass 45.91		1/1	38°C/0.6(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 acid c_m47.91.imp	2016-03-10	sfwb8-15-1 acid c	Mass 47.91		1/1	38°C/0.6(K/min)/1°C		---

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MATL NAME: SFWBB-15-1 ACIDC **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-10-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPP TEMP 022916 / WIPP022916	N/A	2-28-17	DMW Initials 113674 Z No. 3-10-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	
Date & Time Sample Vial Opened	**Date:** 03/10/16 *(mm / dd / yy)* **Time:** 14:05 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/33.1°c	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date

MATL NAME: SFWB8-15-1 ACIDC **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 03-10-16 Time: 14:07	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-10-16 Date	
Crucible / Pan Tare Wt.	0.16890 ~~0.0~~ DMW 113674 3-10-16	grams	N/A	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date
Net Sample Weight	0.02039		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 09 :20	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350C Baseline 030816.ngb-bsv Filename: ____ *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 ACID C **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 03-10-16 Time Started: 14:15 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 38.3	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP SFWB8-15-1 ACIDC.ngb-dsv **Netzsch Measure** Filename: SFWB8-15-1 ACIDC.MDC **ThermoStar** Filename:	N/A	N/A	DMW Initials 113674 Z No. 3-10-16 Date	
Proteus Data	**Mass Changes:** 1) -35.62 Temp. Range (RT-163.7°C) 2) -24.65 Temp. Range (163.7-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -60.27%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-10-16 Date	Initials 120527 Z No. 3/10/16 Date

Notes:

MATL NAME: SFWB8-15-1 ACIDC **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (172.6°c) 2) CO_2 Peak T (131.8°c, 216.0°c) 3) NO/NO_2 Peak T (129.9°c, 230.4°c) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-10-16 Date	
Total Moisture (H_2O) N/A	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __ / __ / __ (mm dd yy) % Error (RSD, 1s): 113674 DMW = 100 x ____ ÷ ____ Std. Error Slope	3-10-16 Wt. %	< 0.32	____ Initials ____ Z No. ____ Date	____ Initials ____ Z No. ____ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-10-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1 - UPC instrument not required to have Cal. File #
2 - Calib. thermometer not required for water chiller
3 - Temp profile not required to go to 1100 °C
4 - Seal %RH & Temp not required
5 - Moisture content eval. not required

DMW 113674 3-10-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-1 PuAm 1.ngb-dsv
Date/Time: 4/7/2016 1:43:36 PM (UTC-6)
End Date/Time: 4/7/2016 2:15:38 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-1 PuAm 1
Sample name: SFWB8-15-1 PuAm 1
Sample Mass: 22.364 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: Surrogate 04-07-16 with 200 ul spike SFWB8-15-1

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

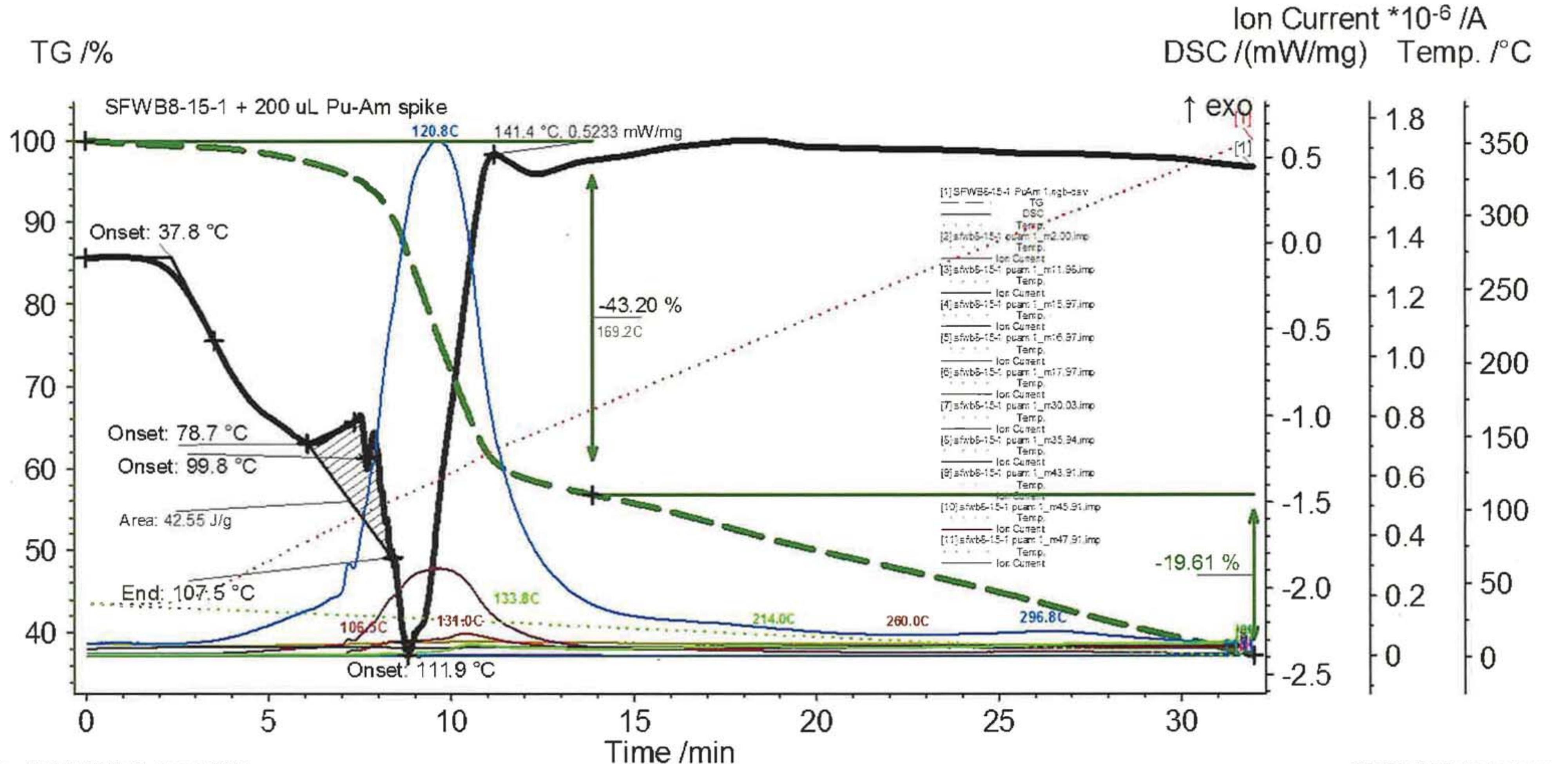
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-1 PuAm 1.ngb-dsv
Date/Time: 4/7/2016 1:43:36 PM (UTC-6)
End Date/Time: 4/7/2016 2:15:38 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-1 PuAm 1
Sample name: SFWB8-15-1 PuAm 1
Sample Mass: 22.364 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: Surrogate 04-07-16 with 200 ul spike SFWB8-15-1

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	37.8 °C	0.0 min	5.2 min
Onset (DSC)	78.7 °C	6.0 min	7.6 min
Onset (DSC)	99.8 °C	7.7 min	8.1 min
Onset (DSC)	111.9 °C	8.8 min	9.1 min
Peak (DSC)	141.4 °C/0.5233 mW/mg	9.9 min	12.5 min
End (DSC)	107.5 °C	8.3 min	8.7 min
Area (DSC),o	42.55 J/g	6.1 min	8.4 min



Main 2016-04-26 09:43 User: 113674

SFWB8-15-1 PuAm 1.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 PuAm 1.ngb-dsv	2016-04-07	SFWB8-15-1 PuAm 1	SFWB8-15-1 PuAm 1	22.364	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 puam 1_m2.00.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 2.00		1/1	36°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 puam 1_m11.98.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 11.98		1/1	36°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 puam 1_m15.97.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 15.97		1/1	36°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 puam 1_m16.97.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 16.97		1/1	36°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 puam 1_m17.97.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 17.97		1/1	36°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 puam 1_m30.03.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 30.03		1/1	36°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 puam 1_m35.94.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 35.94		1/1	36°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 puam 1_m43.91.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 43.91		1/1	36°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 puam 1_m45.91.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 45.91		1/1	36°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 puam 1_m47.91.imp	2016-04-07	sfwb8-15-1 puam 1	Mass 47.91		1/1	36°C/1.1(K/min)/1°C		---

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MATL NAME: SFWB8-15-1 Pu Am 1 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-7-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916/WIPP022916	N/A	02/28/17	DMW Initials 113674 Z No. 4-7-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-7-16 Date
Date & Time Sample Vial Opened	Date: 04-07-16 *(mm / dd / yy)* Time: 13:36 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.4°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-7-16 Date Initials 120527 Z No. 4/7/16 Date

MATL NAME: SFWBR-15-1 ~~ACID A~~ PuAm1 DMW 113674 4-7-16 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 04-07-16 Time: 13:39	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-7-16 Date	
Crucible / Pan Tare Wt.	0.16888	grams	N/A	DMW Initials 113674 Z No. 4-7-16 Date	Initials 120527 Z No. 4/7/16 Date
Net Sample Weight	0.02272		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-7-16 Date	Initials 120527 Z No. 4/7/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 15	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350C BASELINC030816, ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 PuAm1 **Location:** 6-220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-7-16 Date	Initials 120527 Z No. 4/7/16 Date
	Maximum Temp.: 350 °C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 4-7-16 Time Started: 13;47 Total Analysis Time: 00;32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.2	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-1 PuAm1.ngb-dsv **ThermoStar** Filename: SFWB8-15-1 PuAm1.MDC	N/A	N/A	DMW Initials 113674 Z No. 4-7-16 Date	
Proteus Data	**Mass Changes:** 1) -43.20 Temp. Range (RT-169.2°C) 2) -19.61 Temp. Range (169.2-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -62.81%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-7-16 Date	Initials 120527 Z No. 4/7/16 Date

Notes:

MATL NAME: SFWB8-15-1 Pu Am 1 **Location:** 0220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
DMW 113674 4-7-16 ThermoStar Data	Volatile Species: 1) H₂O - OH ~~43,20~~ Peak T (120.8/296.8°C) 2) CO_2 Peak T (133.8/214.0°C) 3) NO/NO_2 Peak T (106.5/131.0°C/) 260.0°C 4) ______ Peak T (__________) 5) ______ Peak T (__________)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-7-16 Date	
Total Moisture (H_2O) N/A	Total Moisture = __________ mg __________ Wt % Date of most recent calibration: ___/___/___ mm dd yy % Error (RSD, 1s): __________ = 100 x __________ ÷ __________ Std. Error Slope DMW 113674 4-7-16	Wt. %	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-7-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		Hum QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1- UPC instrumentation not required to have Cal File #
2- Calib. Thermometer not required for Water Chiller
3- Temp Profile not required to run to 1100°C
4- Seal %RH-Temp. not required
5. Moisture Content Eval. not required
DMW 113674 4-7-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-1 PuAm 2.ngb-dsv
Date/Time: 4/8/2016 11:33:55 AM (UTC-6)
End Date/Time: 4/8/2016 12:05:57 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-1 PuAm 2
Sample name: SFWB8-15-1 PuAm 2
Sample Mass: 19.960 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-1 PuAm 2 - 2nd spiked run

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-1 PuAm 2.ngb-dsv
Date/Time: 4/8/2016 11:33:55 AM (UTC-6)
End Date/Time: 4/8/2016 12:05:57 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-1 PuAm 2
Sample name: SFWB8-15-1 PuAm 2
Sample Mass: 19.960 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-1 PuAm 2 - 2nd spiked run

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	38.8 °C	0.0 min	5.8 min
Onset (DSC)	83.1 °C	6.0 min	7.2 min
Onset (DSC)	100.5 °C	7.8 min	8.1 min
End (DSC)	106.1 °C	7.9 min	8.4 min
Area (DSC),o	28.93 J/g	5.6 min	8.4 min
Onset (DSC)	115.0 °C	8.9 min	11.3 min
Peak (DSC)	138.0 °C/0.6445 mW/mg	9.7 min	12.0 min

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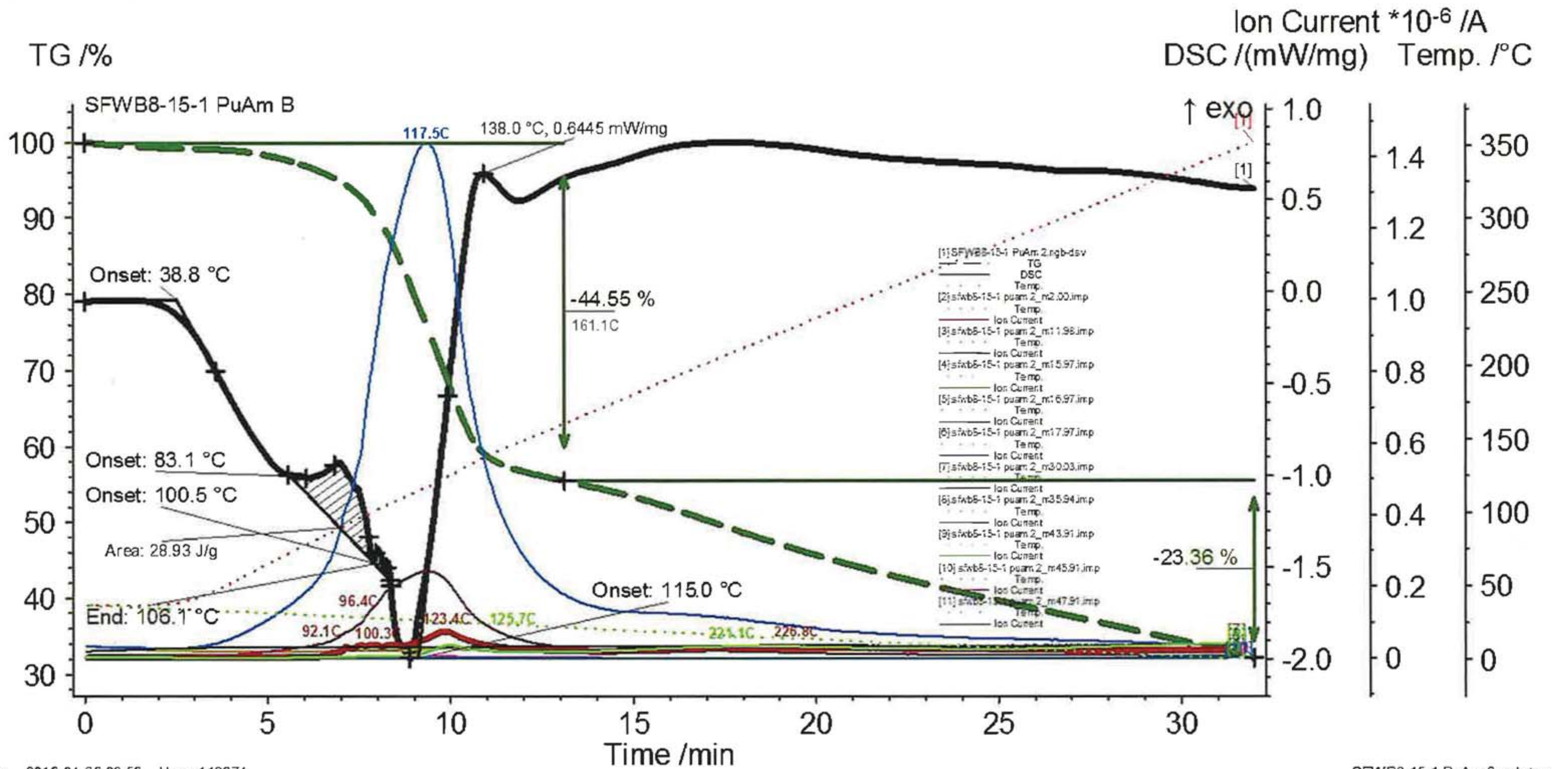

Main 2016-04-26 09:56 User: 113674

SFWB8-15-1 PuAm 2.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 PuAm 2.ngb-dsv	2016-04-08	SFWB8-15-1 PuAm 2	SFWB8-15-1 PuAm 2	19.960	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 puam 2_m2.00.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 2.00		1/1	37°C/1.2(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 puam 2_m11.98.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 11.98		1/1	37°C/1.2(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 puam 2_m15.97.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 15.97		1/1	37°C/1.2(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 puam 2_m16.97.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 16.97		1/1	37°C/1.2(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 puam 2_m17.97.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 17.97		1/1	37°C/1.2(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 puam 2_m30.03.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 30.03		1/1	37°C/1.2(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 puam 2_m35.94.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 35.94		1/1	37°C/1.2(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 puam 2_m43.91.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 43.91		1/1	37°C/1.2(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 puam 2_m45.91.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 45.91		1/1	37°C/1.2(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 puam 2_m47.91.imp	2016-04-08	sfwb8-15-1 puam 2	Mass 47.91		1/1	37°C/1.2(K/min)/1°C		---

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MATL NAME: SFWB8-15-1 PuAm2 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-8-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WSPPTEMP022916/WSPP022916	N/A	02-28-17	DMW Initials 113674 Z No. 4-8-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08-03-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-8-16 Date	
Date & Time Sample Vial Opened	Date: 04/08/16 *(mm / dd / yy)* Time: 11:24 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.0°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date

MATL NAME: SFWB8-15-1 PuAm2 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 04-08-16 Time: 11:31	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-8-16 Date	
Crucible / Pan Tare Wt.	0.17943	grams	N/A	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
Net Sample Weight	0.019960		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 09 :20	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350C Baseline 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 PuAm2 **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 04/08/16 Time Started: 11:34 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 37.1	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-1-PuAM2.ngb-dsv **ThermoStar** Filename: SFWB8-15-1 PuAM2.MDC	N/A	N/A	DMW Initials 113674 Z No. 4.8.16 Date	
Proteus Data	**Mass Changes:** 1) -44.55 Temp. Range (RT-161.1°C) 2) -23.36 Temp. Range (161.1-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -67.91%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date

Notes:

MATL NAME: SFWB8-1S-1 PuAm2 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (117.5°C) 2) CO_2 Peak T (125.7, 221.1°C) 3) NO/NO_2 Peak T (92.1, 96.4, 100.3), 123.4, 226.8°C 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-8-16 Date	
Total Moisture (H_2O) DMW	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): 2-8-16 113674 ______ = 100 x ______ ÷ ______ Std. Error Slope	N/A Wt. %	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-8-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HwM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1- UPC instrument: Cal File # not required
2- Calib. thermometer for Chiller water not required
3- Temp Profile not required to run to 1100°C
4- Seal %RH/Temp meas. not required
5- Moisture Content eval. not required

DMW 113674 4/8/16

Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-1 PuAm3.ngb-dsv
Date/Time:	4/12/2016 2:06:12 PM (UTC-6)
End Date/Time:	4/12/2016 2:38:13 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh

DSC DSC Range:	5000 µV
TG TG Range:	30000 mg
Sample identity:	SFWB8-15-1 PuAm3
Sample name:	SFWB8-15-1 PuAm3
Sample Mass:	17.338 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible

Remark: SFWB8-15-1 PuAm3 third SNM spiked run

Furnace:	STD SiC(PC)
Sample carrier:	DSC(/TG) HIGH RG 2
Measurement End:	Normal end

Furnace TC:	S
Sample TC:	S

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-1 PuAm3.ngb-dsv
Date/Time: 4/12/2016 2:06:12 PM (UTC-6)
End Date/Time: 4/12/2016 2:38:13 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-1 PuAm3
Sample name: SFWB8-15-1 PuAm3
Sample Mass: 17.338 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-1 PuAm3 third SNM spiked run

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	38.1 °C	0.0 min	4.9 min
Onset (DSC)	98.6 °C	7.6 min	8.1 min
Onset (DSC)	106.7 °C	8.2 min	8.9 min
End (DSC)	113.9 °C	8.6 min	9.0 min
Area (DSC),o	20.12 J/g	7.6 min	9.0 min
Onset (DSC)	119.9 °C	9.3 min	11.5 min
Peak (DSC)	141.7 °C/0.601 mW/mg	9.5 min	12.0 min

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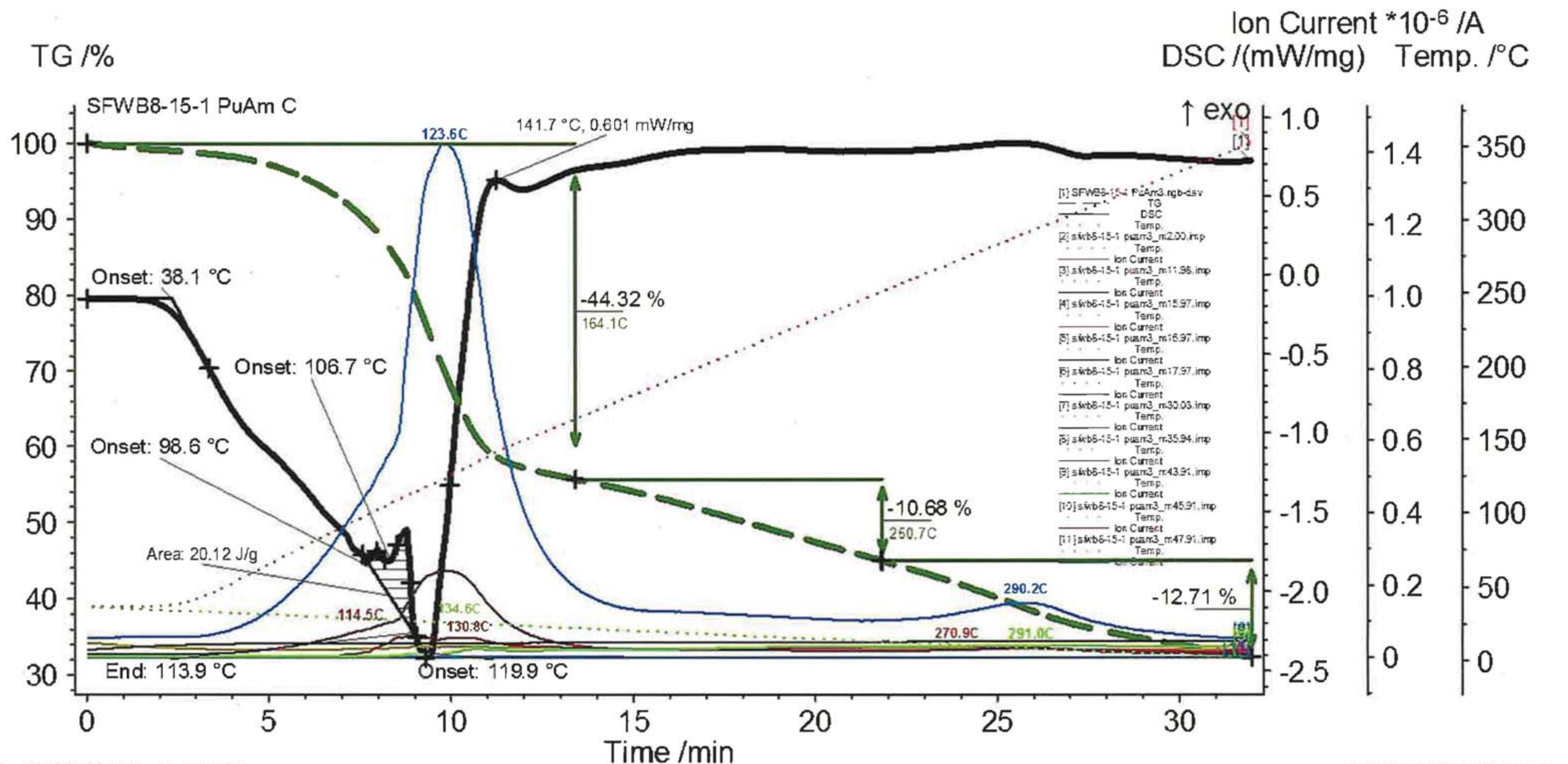

Main 2016-04-26 10:30 User: 113674

SFWB8-15-1 PuAm 3.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 PuAm3.ngb-dsv	2016-04-12	SFWB8-15-1 PuAm3	SFWB8-15-1 PuAm3	17.338	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 puam3_m2.00.imp	2016-04-12	sfwb8-15-1 puam3	Mass 2.00		1/1	37°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 puam3_m11.98.imp	2016-04-12	sfwb8-15-1 puam3	Mass 11.98		1/1	37°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 puam3_m15.97.imp	2016-04-12	sfwb8-15-1 puam3	Mass 15.97		1/1	37°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 puam3_m16.97.imp	2016-04-12	sfwb8-15-1 puam3	Mass 16.97		1/1	37°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 puam3_m17.97.imp	2016-04-12	sfwb8-15-1 puam3	Mass 17.97		1/1	37°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 puam3_m30.03.imp	2016-04-12	sfwb8-15-1 puam3	Mass 30.03		1/1	37°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 puam3_m35.94.imp	2016-04-12	sfwb8-15-1 puam3	Mass 35.94		1/1	37°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 puam3_m43.91.imp	2016-04-12	sfwb8-15-1 puam3	Mass 43.91		1/1	37°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 puam3_m45.91.imp	2016-04-12	sfwb8-15-1 puam3	Mass 45.91		1/1	37°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 puam3_m47.91.imp	2016-04-12	sfwb8-15-1 puam3	Mass 47.91		1/1	37°C/1.1(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-1 Pu Am3 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-12-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WSPPTEMP022916/WSPP022916	N/A	2-28-17	DMW Initials 113674 Z No. 4-12-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-12-16 Date	
Date & Time Sample Vial Opened	Date: 04/12/16 *(mm / dd / yy)* Time: 14:00 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/32.6	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date

MATL NAME: SFWB8-15-1 PuAm3 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 04-12-16 Time: 14:02	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-12-16 Date	
Crucible / Pan Tare Wt.	0.17030	grams	N/A	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date
Net Sample Weight	0.01815		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 41	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WTPP 350C Baseline 030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 PuAm3 **Location:** G270

Run Parameters (Sections 5.4 & 5.6)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 04/12/16 Time Started: 14:06 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.7°C	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-1 PuAm3.ngb-dsv **ThermoStar** Filename: SFWB8-15-1 PuAm3.MDC	N/A	N/A	DMW Initials 113674 Z No. 4-12-16 Date	
Proteus Data	**Mass Changes:** 1) -44.32 Temp. Range (RT-164.1°C) 2) -10.68 Temp. Range (164.1-250.7°C) 3) -12.71 Temp. Range (250.7-350°C) 4) ____ Temp. Range (____) **Total mass Change:** -67.71%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date

Notes:

MATL NAME: SFWB8-15-1 PuAm 3 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H_2O Peak T (123.6, 290.2°C) 2) CO_2 Peak T (134.6, 291.0°C) 3) NO/NO_2 Peak T (114.5, 130.8, 270.7°C 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-12-16 Date	
Total Moisture (H_2O) N/A	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): ______ = 100 x ______ ÷ ______ Std. Error Slope DMW 113674 4-12-16	Wt. %	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-12-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary

1- UPC instrument - Cal. File # not required

2- Calib. Thermometer not required for Chiller water

3- Temp. Profile not required to run to 1100°C

4- Seal %RH & Temp. meas. not required

5- Moisture content eval. not required

DMW 113674 4-12-16

DMW 113674
4-28-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-1 ~~400~~ 300 PuAm.ngb-dsv	**Sample identity:**	SFWB8-15-1 400 PuAm
Date/Time:	4/14/2016 3:24:37 PM (UTC-6)	**Sample name:**	SFWB8-15-1 400 PuAm
End Date/Time:	4/14/2016 3:56:40 PM (UTC-6)	**Sample Mass:**	20.23 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-1 400 PuAm - 400 uL spiked sample

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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DMW 113674 4-28-16

Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-1 ~~400~~ 40u PuAm.ngb-dsv
Date/Time:	4/14/2016 3:24:37 PM (UTC-6)
End Date/Time:	4/14/2016 3:56:40 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-1 400 PuAm
Sample name:	SFWB8-15-1 400 PuAm
Sample Mass:	20.23 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-1 400 PuAm - 400 uL spiked sample

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	101.7 °C	7.8 min	8.5 min
Onset (DSC)	111.1 °C	8.5 min	8.8 min
Onset (DSC)	125.9 °C	9.8 min	10.2 min
Area (DSC),o	33.87 J/g	7.8 min	9.3 min
End (DSC)	117.1 °C	8.7 min	9.2 min
Peak (DSC)	147.7 °C/0.4128 mW/mg	10.4 min	12.5 min

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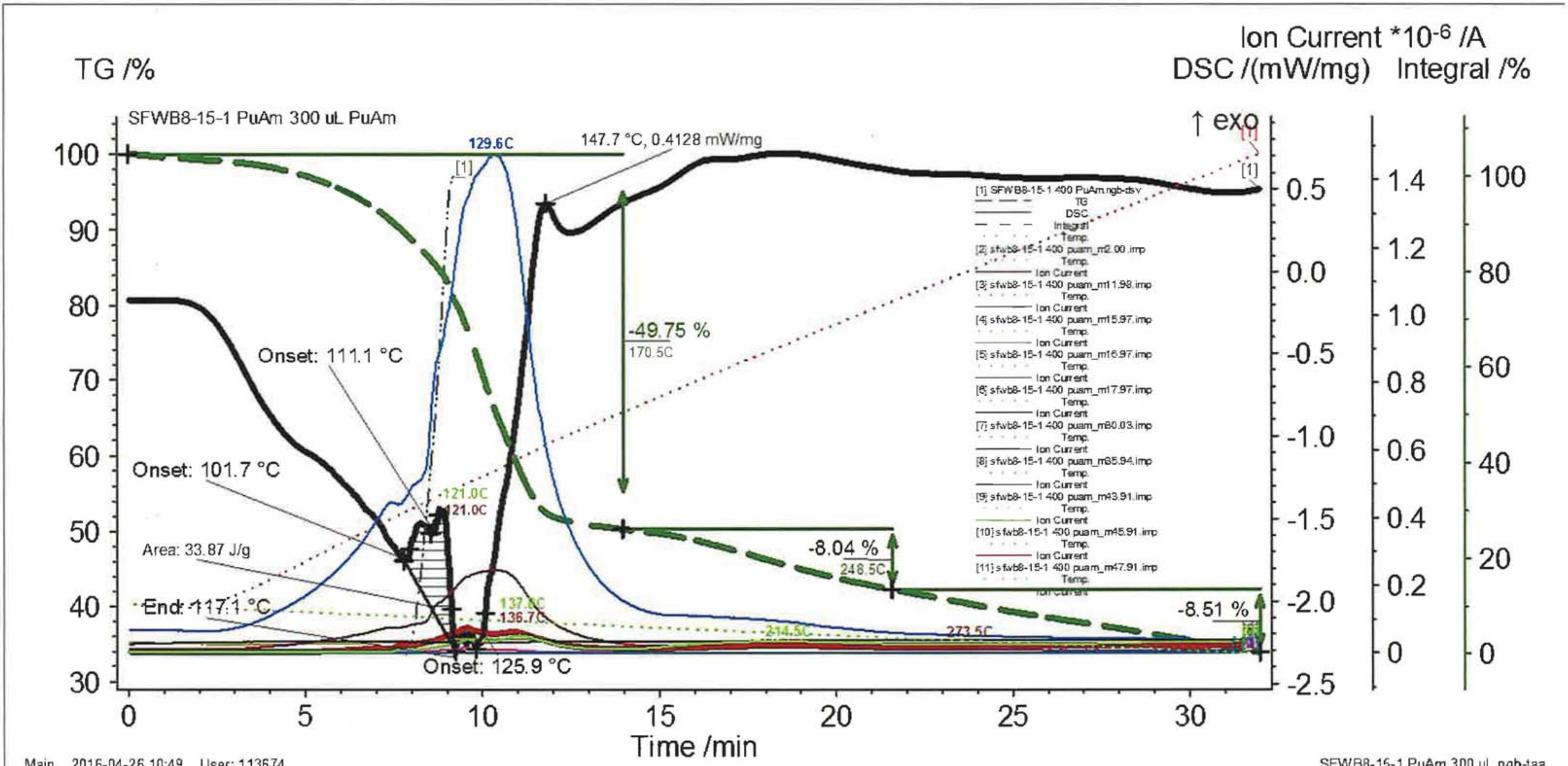

Main 2016-04-26 10:49 User: 113674

SFWB8-15-1 PuAm 300 uL.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Ma...	S...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-1 400 PuAm.ngb-dsv	2016-04-14	SFWB8-15-1 400 PuAm	SFWB8-15-1 400 PuAm	20.23	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-1 400 puam_m2.00.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 2.00		1/1	36°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-1 400 puam_m11.98.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 11.98		1/1	36°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-1 400 puam_m15.97.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 15.97		1/1	36°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-1 400 puam_m16.97.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 16.97		1/1	36°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-1 400 puam_m17.97.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 17.97		1/1	36°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-1 400 puam_m30.03.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 30.03		1/1	36°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-1 400 puam_m35.94.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 35.94		1/1	36°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-1 400 puam_m43.91.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 43.91		1/1	36°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-1 400 puam_m45.91.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 45.91		1/1	36°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-1 400 puam_m47.91.imp	2016-04-14	sfwb8-15-1 400 puam	Mass 47.91		1/1	36°C/1.1(K/min)/1°C		---

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MATL NAME: SFWB8-15-1 300ul PuAm **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-14-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WSPPTEMP022916/WSPP022916	N/A	02-28-17	DMW Initials 113674 Z No. 4-14-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08-03-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-14-16 Date	
Date & Time Sample Vial Opened	Date: 04/14/16 *(mm / dd / yy)* Time: 15:17 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/26.1°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-14-16 Date	Initials 120527 Z No. 4/14/16 Date

MATL NAME: SFWB8-15-1 300μL Pu Am **Location:** G270

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	**Date:** 04/14/16 **Time:** 15:22	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-14-16 Date	
Crucible / Pan Tare Wt.	0.16960	grams	N/A	DMW Initials 113674 Z No. 4-14-16 Date	Initials 120527 Z No. 4/14/16 Date
Net Sample Weight	0.02023		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-14-16 Date	Initials 120527 Z No. 4/14/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 14:49	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	SWJPP350C Baseline.030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-1 300mL PuAm **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-14-16 Date	Initials 120527 Z No. 4/14/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 04-14-16 DMW 113674 4-14-16 Time Started: ~~15:29~~ 15:24 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 35.6°C	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFW88-15-1 400 PuAm.ngb-dsv **ThermoStar** Filename: SFW88-15-1 400 PuAm.ngb-dsv	N/A	N/A see note DMW 113674 4-28-16	DMW Initials 113674 Z No. 4-14-16 Date	
Proteus Data	**Mass Changes:** 1) -49.75 Temp. Range ( ) 2) -8.04 Temp. Range ( ) 3) -8.51 Temp. Range ( ) 4) ______ Temp. Range ( ) **Total mass Change:** -66.30%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-14-16 Date	Initials 120527 Z No. 4/14/16 Date

Notes:

MATL NAME: SFWB8-15-1 300mL PuAm **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	<u>Volatile Species:</u> 1) H_2O/OH Peak T (129.6°C) 2) CO_2 Peak T (121.0/137.8/214.5°C 3) NO/NO_2 Peak T (121.0/136.7/273.5°C 4) _______ Peak T (______________) 5) _______ Peak T (______________)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-14-16 Date	
Total Moisture (H_2O)	N/A <u>Total Moisture =</u> ______________ <u>mg</u> ______________ <u>Wt %</u> <u>Date of most recent calibration:</u> ____/____/____ *mm dd yy* % Error (RSD, 1s): DMW ____________ = 100 x __________ ÷ __________ Std. Error Slope	113674 Wt. %	< 0.32	4-14-16 Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-14-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		QR 1492714 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

NOTE: some confusion as to whether the sample contained 400μL or 300μL of spike. Notes in lab notebook confirm that 300μL was added (see page 54 of lab notes)

DMW 113674 4-14-16

N/A Summary

1 UPC instrument - Cal. File # not required

2 Calib Thermometer, not required for chiller water

3 - Temp Profile not required to run to 1100 °C

4 - Seal % RH / Temp. meas not required

5 - Moisture Content eval. not required

DMW 113674 4-14-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2A 032316.ngb-dsv
Date/Time: 3/23/2016 11:36:57 AM (UTC-6)
End Date/Time: 3/23/2016 12:08:57 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 A
Sample name: SFWB8-15-2 A
Sample Mass: 23.27 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-2 A 03-23-16

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument:	NETZSCH STA 409PC/PG	**TG TG Range:**	30000 mg
Project:	WIPP	**Sample identity:**	SFWB8-15-2 A
Filename:	SFWB8-15-2A 032316.ngb-dsv	**Sample name:**	SFWB8-15-2 A
Date/Time:	3/23/2016 11:36:57 AM (UTC-6)	**Sample Mass:**	23.27 mg
End Date/Time:	3/23/2016 12:08:57 PM (UTC-6)	**Crucible:**	DSC/TG pan Pt-Rh
Laboratory:	55-0004-0208	**Crucible Mass:**	0 mg
Operator:	DMW	**Reference name:**	empty
Mode:	DSC-TG	**Reference Mass:**	0 mg
Measurement Type:	sample with correction	**Reference Crucible Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Material:**	WIPP surrogate
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Sample determination mode:**	Manual
Sensitivity:	WIPP 022916.ngb-esv	**Residuum measurement:**	Not possible
Crucible:	DSC/TG pan Pt-Rh	**Atmosphere:**	ARGON/50 / ARGON/30 / <no gas>/---
DSC DSC Range:	5000 µV		

Remark: SFWB8-15-2 A 03-23-16

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	39.3 °C	1.1 min	6.6 min
Onset (DSC)	93.9 °C	6.5 min	8.1 min
End (DSC)	105.0 °C	7.4 min	8.3 min
Area (DSC),o	17.64 J/g	6.4 min	8.3 min
Onset (DSC)	116.6 °C	8.7 min	11.7 min
Peak (DSC)	143.0 °C/0.5122 mW/mg	10.5 min	12.3 min
Mass Change (TG)	-38.10 %	0.0 min	13.4 min
Mass Change (TG)	-22.33 %	13.4 min	32.0 min

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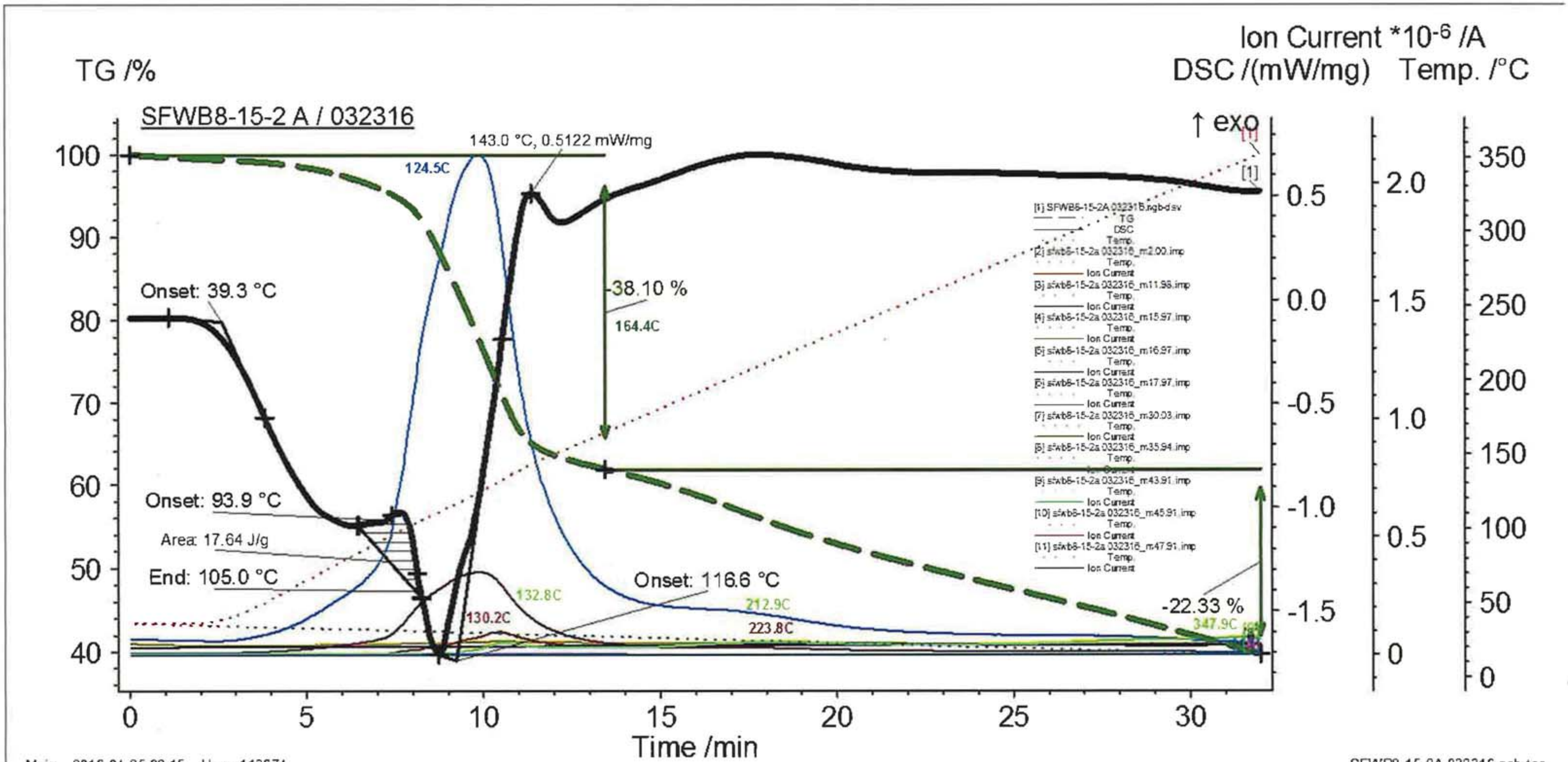

Main 2016-04-25 09:15 User: 113674

SFWB8-15-2A 032316.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2A 032316.ngb-dsv	2016-03-23	SFWB8-15-2 A	SFWB8-15-2 A	23.27	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2a 032316_m2.00.imp	2016-03-23	sfwb8-15-2a 032316	Mass 2.00		1/1	37°C/0.7(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2a 032316_m11.98.imp	2016-03-23	sfwb8-15-2a 032316	Mass 11.98		1/1	37°C/0.7(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2a 032316_m15.97.imp	2016-03-23	sfwb8-15-2a 032316	Mass 15.97		1/1	37°C/0.7(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2a 032316_m16.97.imp	2016-03-23	sfwb8-15-2a 032316	Mass 16.97		1/1	37°C/0.7(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2a 032316_m17.97.imp	2016-03-23	sfwb8-15-2a 032316	Mass 17.97		1/1	37°C/0.7(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2a 032316_m30.03.imp	2016-03-23	sfwb8-15-2a 032316	Mass 30.03		1/1	37°C/0.7(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2a 032316_m35.94.imp	2016-03-23	sfwb8-15-2a 032316	Mass 35.94		1/1	37°C/0.7(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2a 032316_m43.91.imp	2016-03-23	sfwb8-15-2a 032316	Mass 43.91		1/1	37°C/0.7(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2a 032316_m45.91.imp	2016-03-23	sfwb8-15-2a 032316	Mass 45.91		1/1	37°C/0.7(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2a 032316_m47.91.imp	2016-03-23	sfwb8-15-2a 032316	Mass 47.91		1/1	37°C/0.7(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-2A **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-23-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916/WIPP022916	N/A	02-28-17	DMW Initials
Temperature & Humidity Monitor	041888	10-25-16	113674 Z No.
Calibrated Thermometer (for Water Chiller)	N/A	N/A	3-23-16
Wall Clock	040480	8-3-16	Date

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date	
Date & Time Sample Vial Opened	Date: 03-23-16 *(mm / dd / yy)* Time: 11:26 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.4 °C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date

MATL NAME: SFWB8-15-2 A **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-23-16 Time: 11:31	Mm/dd/yy – 24 hr.	N/A	Dmw Initials 113674 Z No. 3-23-16 Date	
Crucible / Pan Tare Wt.	0.16744	grams	N/A	Dmw Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
Net Sample Weight	0.02327		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	Dmw Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
Gas Pressure	Gas Pressure (at regulator): < 10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 49	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP 350C BASELINE 030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2A **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-23-16 Time Started: 11:37 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.6	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2A 032316.ngb-dsv **ThermoStar** Filename: SFWB8-15-2A 032316.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date	
Proteus Data	**Mass Changes:** 1) -38.10 Temp. Range (RT-164.4°C) 2) -22.33 Temp. Range (164.4-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -60.43%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date

Notes:

MATL NAME: SFWB8-15-2A **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H2O Peak T (124.5°C) 2) CO2 Peak T (132.8/212.9/347.5°C) 3) NO/NO2 Peak T (130.2/223.8°F) 4) ____ Peak T ( ) 5) ____ Peak T ( )	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-23-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: 3 / 25 / 16 (mm / dd / yy) % Error (RSD, 1s): ____ = 100 x ____ ÷ ____ Std. Error Slope DMW 113674	N/A Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-23-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1 - Cal File # not required for UPC
2 - Calib. Thermometer not required for water chiller
3 - Temp profile not required to go to 1100°c
4 - SEAL %RH/Temp data not required
5 - Moisture Content data not required
DMW 113674 3-23-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-2B 032316.ngb-dsv	**Sample identity:**	SFWB8-15-2 B 032316
Date/Time:	3/23/2016 2:39:22 PM (UTC-6)	**Sample name:**	SFWB8-15-2 B 032316
End Date/Time:	3/23/2016 3:11:23 PM (UTC-6)	**Sample Mass:**	14.04 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-2 B 032316

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument:	NETZSCH STA 409PC/PG	**TG TG Range:**	30000 mg
Project:	WIPP	**Sample identity:**	SFWB8-15-2 B 032316
Filename:	SFWB8-15-2B 032316.ngb-dsv	**Sample name:**	SFWB8-15-2 B 032316
Date/Time:	3/23/2016 2:39:22 PM (UTC-6)	**Sample Mass:**	14.04 mg
End Date/Time:	3/23/2016 3:11:23 PM (UTC-6)	**Crucible:**	DSC/TG pan Pt-Rh
Laboratory:	55-0004-0208	**Crucible Mass:**	0 mg
Operator:	DMW	**Reference name:**	empty
Mode:	DSC-TG	**Reference Mass:**	0 mg
Measurement Type:	sample with correction	**Reference Crucible Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Material:**	WIPP surrogate
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Sample determination mode:**	Manual
Sensitivity:	WIPP 022916.ngb-esv	**Residuum measurement:**	Not possible
Crucible:	DSC/TG pan Pt-Rh	**Atmosphere:**	ARGON/50 / ARGON/30 / <no gas>/---
DSC DSC Range:	5000 µV		

Remark: SFWB8-15-2 B 032316

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	39.8 °C	1.5 min	5.1 min
Onset (DSC)	74.0 °C	5.5 min	6.6 min
Onset (DSC)	118.4 °C	8.8 min	10.1 min
Peak (DSC)	140.9 °C/0.8916 mW/mg	9.0 min	12.3 min
Onset (DSC)	86.6 °C	6.6 min	7.7 min
End (DSC)	99.0 °C	7.1 min	7.8 min
Area (DSC),o	9.332 J/g	5.8 min	7.8 min
Mass Change (TG)	-38.18 %	0.0 min	13.4 min
Mass Change (TG)	-20.83 %	13.4 min	32.0 min

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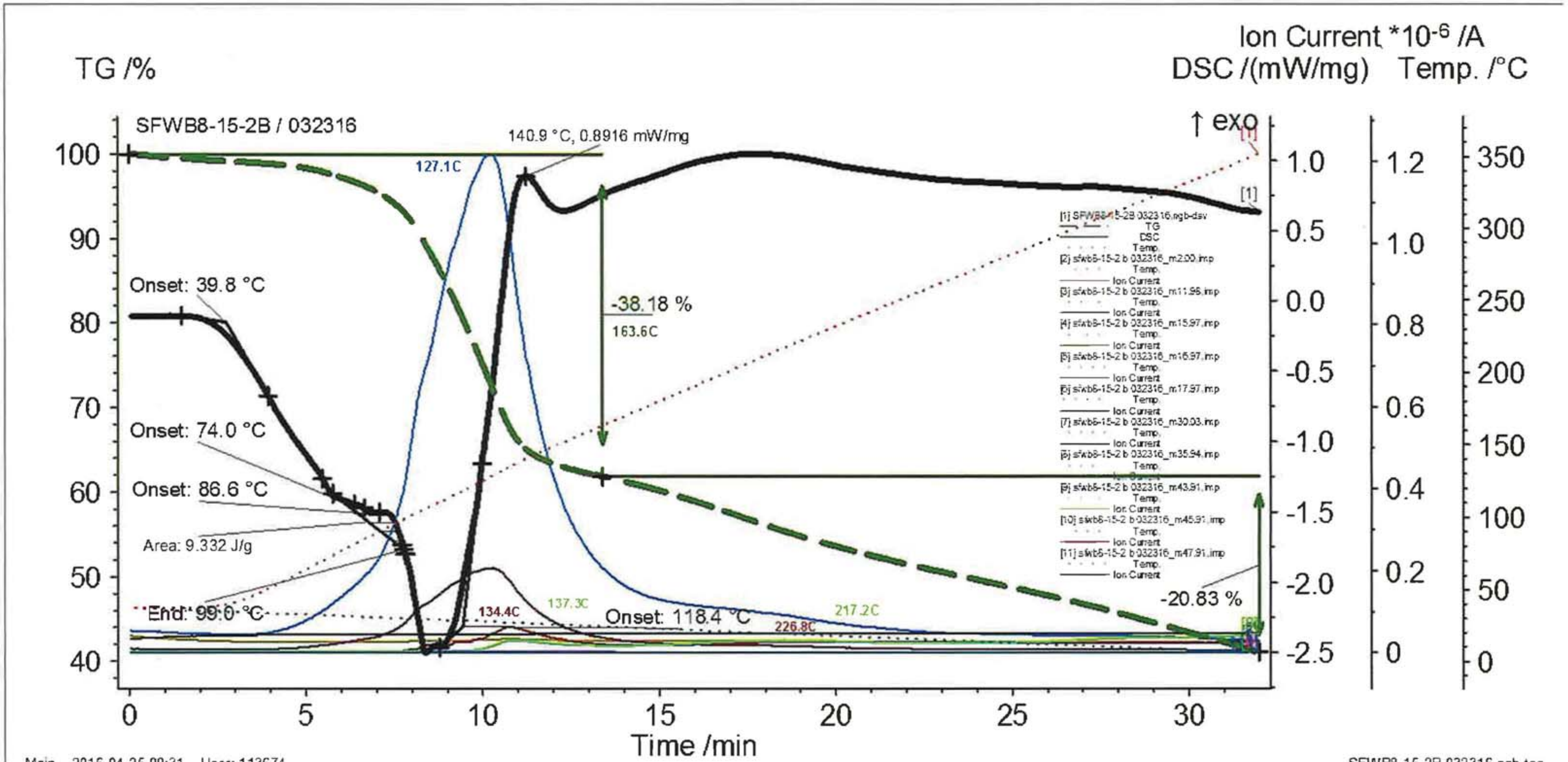

Main 2016-04-25 09:31 User: 113674

SFWB8-15-2B 032316.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mas...	Se...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2B 032316.ngb-dsv	2016-03-23	SFWB8-15-2 B 032316	SFWB8-15-2 B 032316	14.04	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 b 032316_m2.00.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 2.00		1/1	37°C/1.0(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 b 032316_m11.98.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 11.98		1/1	37°C/1.0(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 b 032316_m15.97.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 15.97		1/1	37°C/1.0(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 b 032316_m16.97.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 16.97		1/1	37°C/1.0(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 b 032316_m17.97.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 17.97		1/1	37°C/1.0(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 b 032316_m30.03.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 30.03		1/1	37°C/1.0(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 b 032316_m35.94.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 35.94		1/1	37°C/1.0(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 b 032316_m43.91.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 43.91		1/1	37°C/1.0(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 b 032316_m45.91.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 45.91		1/1	37°C/1.0(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 b 032316_m47.91.imp	2016-03-23	sfwb8-15-2 b 032316	Mass 47.91		1/1	37°C/1.0(K/min)/1°C		---

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MATL NAME: SFW38-15-2B **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-23-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916 / WIPP022916	N/A	02-28-17	DMW Initials 113674 Z No. 3-23-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date	
Date & Time Sample Vial Opened	Date: 03-23-16 *(mm / dd / yy)* Time: 14:30 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.4 °C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date

MATL NAME: SFWB8-15-2 B **Location:** G220

Sample Weights (Section 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
Date & Time Sample Weighed	**Date:** 3·23·16 **Time:** 14:32	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3·23·16 Date	
Crucible / Pan Tare Wt.	0.17942	grams	N/A	DMW Initials 113674 Z No. 3·23·16 Date	Initials 120527 Z No. 3/23/16 Date
Net Sample Weight	0.01404		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Evacuation / Backfill	Sample Chamber Evacuated? [✓ YES] / NO *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10:49	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350C BASELINE 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2 B **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-23-16 DMW 113674 3-23-16 Time Started: ~~16;06~~ 14;38 Total Analysis Time: 00;32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 37.3	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP SFW38-15-2 B.032316.ngb-dsv **Netzsch Measure** Filename: SFWB8-15-2B.032316.MDC **ThermoStar** Filename:	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date	
Proteus Data	**Mass Changes:** 1) -38.18 Temp. Range (RT-163.6°C) 2) -20.83 Temp. Range (163.6-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -59.01%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date

Notes:

MATL NAME: SFWB8-15-2B **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (127.1 °C) 2) CO_2 Peak T (137.3 / 217.2 °C) 3) NO/NO_2 Peak T (134.4 / 226.8 °C) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-23-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: ____ mm dd yy % Error (RSD, 1s): ____ = 100 x ____ ÷ ____ Std. Error Slope N/A DMW 113674 3-23-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-23-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		AWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1- no Cal. File # Required for UPC instrumentation
2- Calib. Thermometer for Water Chiller not required
3- Temp. Profile not required to go to 1100°C
4- Seal % RH / Temp. Not Required
5- Moisture Content not required
DMW 113674 3-23-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2C 032316.ngb-dsv
Date/Time: 3/23/2016 4:06:06 PM (UTC-6)
End Date/Time: 3/23/2016 4:38:07 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 C 032316
Sample name: SFWB8-15-2 C 032316
Sample Mass: 13.89 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-2 C 032316 3rd trial

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2C 032316.ngb-dsv
Date/Time: 3/23/2016 4:06:06 PM (UTC-6)
End Date/Time: 3/23/2016 4:38:07 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 C 032316
Sample name: SFWB8-15-2 C 032316
Sample Mass: 13.89 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 C 032316 3rd trial

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	41.7 °C	0.0 min	6.1 min
Onset (DSC)	81.2 °C	6.2 min	8.2 min
End (DSC)	103.0 °C	6.2 min	8.2 min
Area (DSC),o	27.18 J/g	6.2 min	8.2 min
Peak (DSC)	142.2 °C/0.9085 mW/mg	9.0 min	12.4 min
Onset (DSC)	113.8 °C	8.7 min	11.8 min
Mass Change (TG)	-39.86 %	0.0 min	13.5 min
Mass Change (TG)	-12.90 %	13.4 min	23.2 min
Mass Change (TG)	-10.08 %	23.2 min	32.0 min

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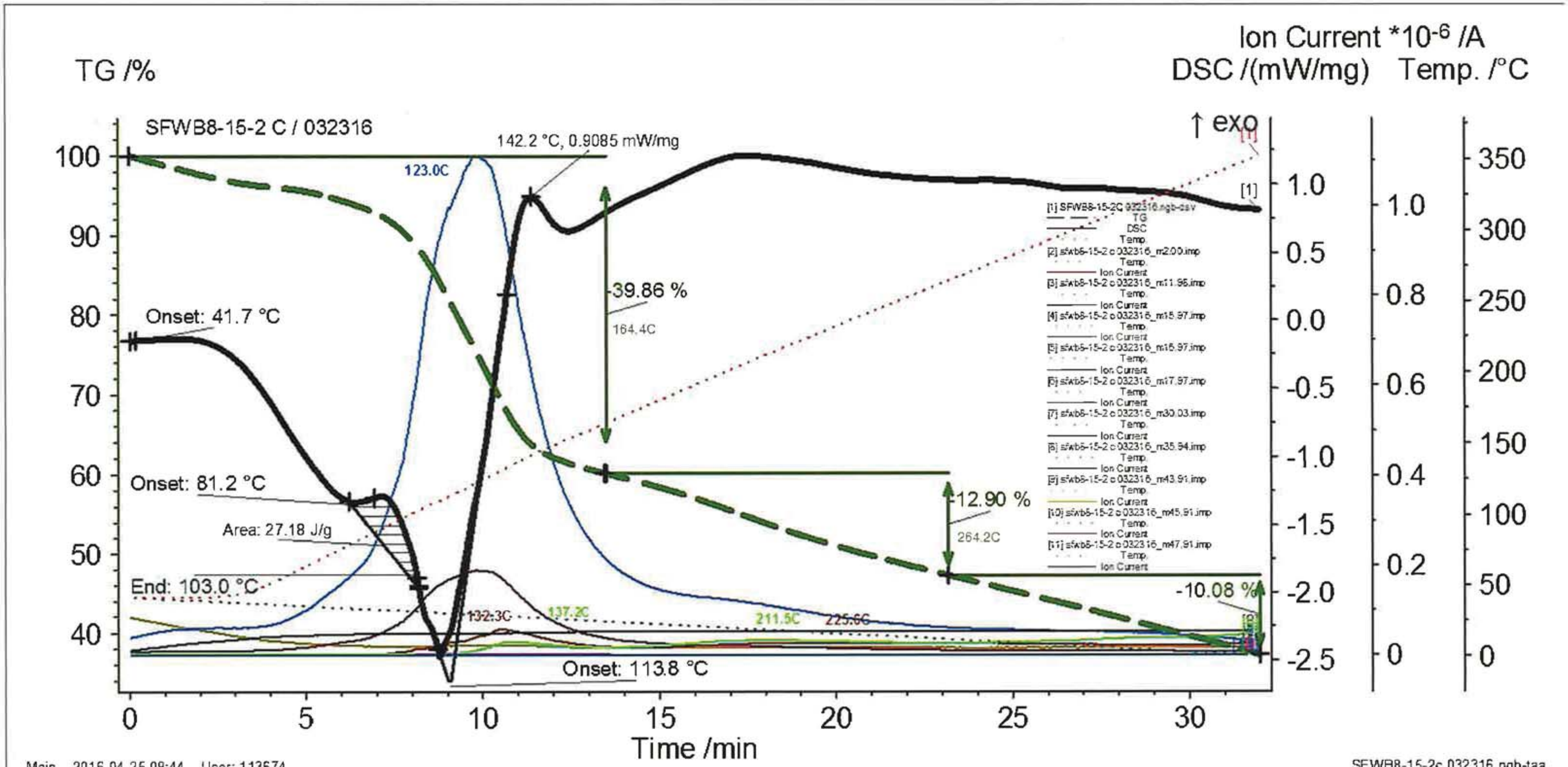


Main 2016-04-25 09:44 User: 113674

SFWB8-15-2c 032316.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mas...	Se...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2C 032316.ngb-dsv	2016-03-23	SFWB8-15-2 C 032316	SFWB8-15-2 C 032316	13.89	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 c 032316_m2.00.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 2.00		1/1	42°C/1.3(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 c 032316_m11.98.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 11.98		1/1	42°C/1.3(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 c 032316_m15.97.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 15.97		1/1	42°C/1.3(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 c 032316_m16.97.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 16.97		1/1	42°C/1.3(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 c 032316_m17.97.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 17.97		1/1	42°C/1.3(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 c 032316_m30.03.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 30.03		1/1	42°C/1.3(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 c 032316_m35.94.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 35.94		1/1	42°C/1.3(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 c 032316_m43.91.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 43.91		1/1	42°C/1.3(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 c 032316_m45.91.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 45.91		1/1	42°C/1.3(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 c 032316_m47.91.imp	2016-03-23	sfwb8-15-2 c 032316	Mass 47.91		1/1	42°C/1.3(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-2C **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-23-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916/WIPP022916	N/A	02-28-17	DMW Initials 113674 Z No. 3-23-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date
Date & Time Sample Vial Opened	Date: 03/23/16 *(mm / dd / yy)* Time: 16:01 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.4°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-23-16 Date Initials 120527 Z No. 3/23/16 Date

MATL NAME: SFWB8-15-2C **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-23-16 Time: 16:03	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-23-16 Date	
Crucible / Pan Tare Wt.	0.16641	grams	N/A	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
Net Sample Weight	0.01389		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 49	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350CBaseline030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2C **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-23-16 Time Started: 16:06 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 41.7	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2C032316.ngb-dsv **ThermoStar** Filename: SFWB8-15-2C032316.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-23-16 Date	
Proteus Data	**Mass Changes:** 1) -39.86 Temp. Range (RT-164.4°F) 2) -12.90 Temp. Range (164.4-~~364~~°C) 264.2°C 3) -10.08 Temp. Range (264.2-350°C) 4) ______ Temp. Range (______) **Total mass Change:** ~~-52.76%~~ DMW 113674/3-23-16 DMW 113174 ~~-60.84%~~ 4-28-16 -62.84%	Wt. % / °C	DMW 113674 3-23-16 Total < 0.4	DMW Initials 113674 Z No. 3-23-16 Date	Initials 120527 Z No. 3/23/16 Date

Notes:

MATL NAME: SFWB8-15-2C **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O Peak T (123.0°C) 2) CO_2 Peak T (137.2, 211.5°C) 3) NO/NO_2 Peak T (132.3, 225.6°C) 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-23-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: /4/ 1 mm 6 dd yy % Error (RSD, 1s): N/A = 100 x ______ ÷ ______ Std. Error Slope DMW 3-23-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-23-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1 - Cal. File number not required for UPC
2 - Calibrated Thermometer For water chiller not required
3 - Temp. Profile not required to run to 1100°C
4 - Seal %RH / Temp. not required
5 - Moisture Content not required

DMW 113674 3-23-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2 ACID A.ngb-dsv
Date/Time: 3/24/2016 10:59:53 AM (UTC-6)
End Date/Time: 3/24/2016 11:12:13 AM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 ACID A
Sample name: SFWB8-15-2 ACID A
Sample Mass: 24.981 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-2 ACID A 032416

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: User reset

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

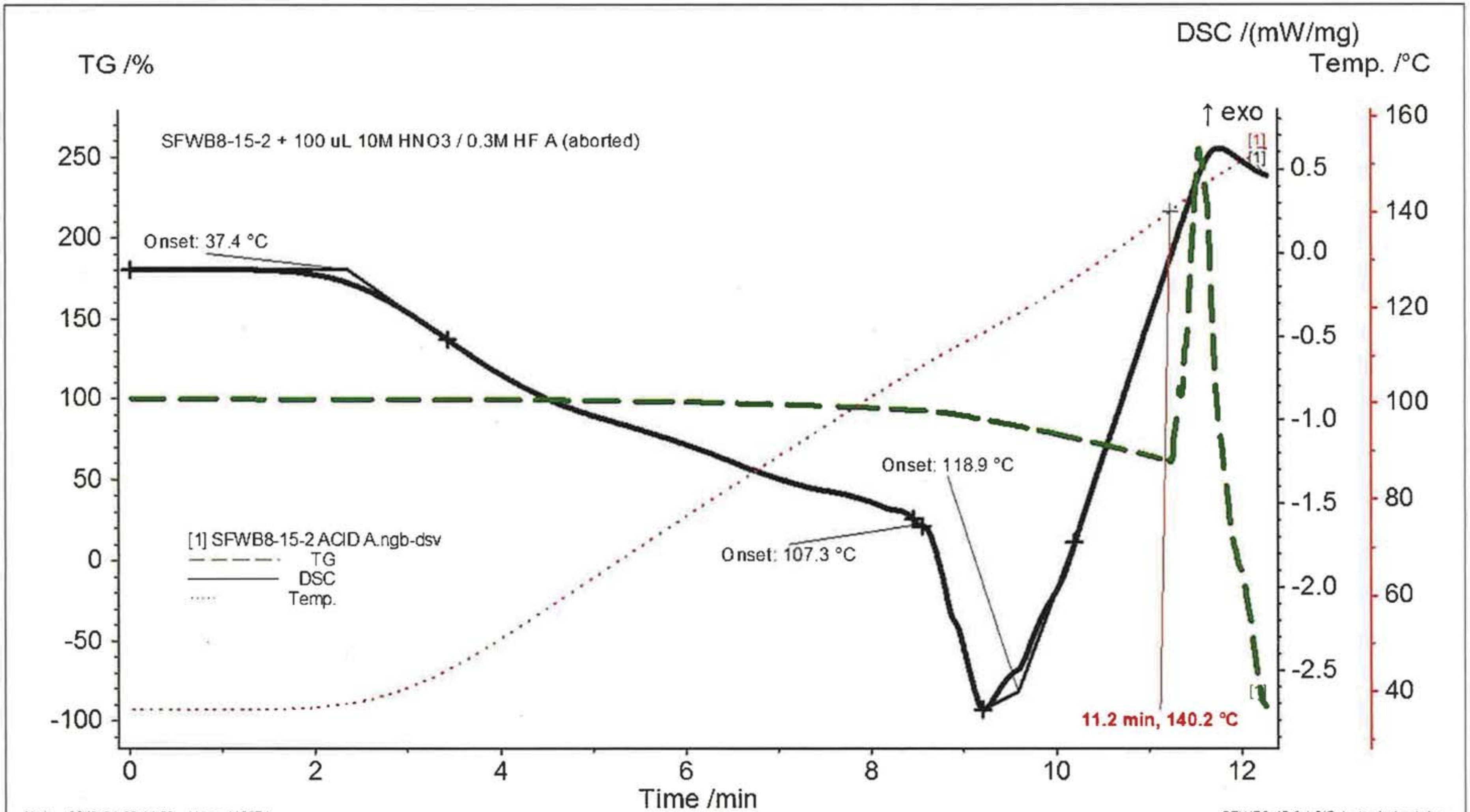
Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument:	NETZSCH STA 409PC/PG	**TG TG Range:**	30000 mg
Project:	WIPP	**Sample identity:**	SFWB8-15-2 ACID A
Filename:	SFWB8-15-2 ACID A.ngb-dsv	**Sample name:**	SFWB8-15-2 ACID A
Date/Time:	3/24/2016 10:59:53 AM (UTC-6)	**Sample Mass:**	24.981 mg
End Date/Time:	3/24/2016 11:12:13 AM (UTC-6)	**Crucible:**	DSC/TG pan Pt-Rh
Laboratory:	55-0004-0208	**Crucible Mass:**	0 mg
Operator:	DMW	**Reference name:**	empty
Mode:	DSC-TG	**Reference Mass:**	0 mg
Measurement Type:	sample with correction	**Reference Crucible Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Material:**	WIPP surrogate
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Sample determination mode:**	Manual
Sensitivity:	WIPP 022916.ngb-esv	**Residuum measurement:**	Not possible
Crucible:	DSC/TG pan Pt-Rh	**Atmosphere:**	ARGON/50 / ARGON/30 / <no gas>/---
DSC DSC Range:	5000 µV		

Remark: SFWB8-15-2 ACID A 032416

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Value (Temp.)	11.2 min	140.2 °C	
Onset (DSC)	118.9 °C	9.2 min	11.9 min
Onset (DSC)	37.4 °C	0.0 min	5.1 min
Onset (DSC)	107.3 °C	8.4 min	8.8 min



Main 2016-04-20 11:20 User: 113674

SFWB8-15-2 ACID A aborted.ngb-taa

Instrument : NETZSCH STA 409PC/PG **File :** C:\NETZSCH\Proteus61\data\SFWB8-15-2 ACID A.ngb-dsv **Remark :** SFWB8-15-2 ACID A 032416

Project :	WIPP	**Material :**	WIPP surrogate	**Segments :**	1/1
Identity :	SFWB8-15-2 ACID A	**Correction file :**	WIPP 350C Baseline 030816.ngb-bsv	**Crucible :**	DSC/TG pan Pt-Rh
Date/time :	3/24/2016 10:59:53 AM	**Temp.Cal./Sens. Files :**	WIPP temp 022916.ngb-tsv / WIPP 022916.ngb-esv	**Atmosphere :**	ARGON/50 / ARGON/30 / <no gas>/—
Laboratory :	55-0004-0208	**Range :**	30°C/10.0(K/min)/350°C	**TG corr./m. range :**	020/30000 mg
Operator :	DMW	**Sample car./TC :**	DSC(/TG) HIGH RG 2 / S	**DSC corr./m. range :**	020/5000 µV
Sample :	SFWB8-15-2 ACID A, 24.981 mg	**Mode/type of meas. :**	DSC-TG / sample with correction		

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MATL NAME: SFWB8-15-L ACID A **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-24-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022516/WIPP022916	N/A	2-28-17	DMW Initials
Temperature & Humidity Monitor	041888	10-25-16	113674 Z No.
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	3-24-16 Date

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials	
Date & Time Sample Vial Opened	Date: 03-24-16 ~~10-4~~ DMW 113674 3-24-16 *(mm / dd / yy)* Time: 10:49 *(24 hour)*	N/A	N/A	113674 Z No. 3-24-16 Date	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.7°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date

MATL NAME: SFWB8-15-2 ACID A **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-24-16 Time: 10:52	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-24-16 Date	
Crucible / Pan Tare Wt.	0.16795	grams	N/A	DMW Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date
Net Sample Weight	0.02835		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 05	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP 350C BASELINE 030816.ngb-bsv Filename: ____ *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2 ACID A **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-24-16 Time Started: 11:00 Total Analysis Time: 00:13	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36,2	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2 ACID A.ngb-dsv **ThermoStar** Filename: SFWB8-152 ACID A . MDC	N/A	N/A	DMW Initials 113674 Z No. 3-24-16 Date	
Proteus Data	**Mass Changes:** NO DATA DMW 113674 3-24-16 1) ______ Temp. Range (__________) 2) ______ Temp. Range (__________) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** ______	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date

Notes:

Run ABORTED @ 11:12AM - mass change indicated that sample swelling caused interaction w/ furnace walls. TOO MUCH SAMPLE - no results. Looks like swelling occurs ~140-150°C - USEFUL DMW 113674

3-24-16

MATL NAME: SFWB 8-15-2 ACID A **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: NO Results DMW 113674 3-24-16 1) ______ Peak T (______) 2) ______ Peak T (______) 3) ______ Peak T (______) 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-24-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: ______ mm dd yy % Error (RSD, 1s): ______ = 100 x ______ ÷ ______ Std. Error Slope N/A DMW 113674 3-24-16	Wt. %	< 0.32	DMW Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-24-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1) cal File # not issued for UPC instrument - not required
2) Calib. thermometer for - water chiller not required
3) Temp. Profile to 1100°C not required
4) Seal RH% / Temp - not required
5) moisture content NOT REQUIRED
- Run ABORTED after 13 minutes
DMW 115674 3-24-16

Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-2 ACID A2.ngb-dsv
Date/Time:	3/24/2016 2:16:17 PM (UTC-6)
End Date/Time:	3/24/2016 2:48:20 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-2 ACID A2
Sample name:	SFWB8-15-2 ACID A2
Sample Mass:	18.292 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 ACID A2 second attempt

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	38.5 °C	1.3 min	5.4 min
Onset (DSC)	120.4 °C	9.4 min	10.0 min
Onset (DSC)	86.5 °C	6.2 min	8.3 min
Peak (DSC)	142.9 °C/0.6792 mW/mg	10.4 min	12.2 min

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2 ACID A2.ngb-dsv
Date/Time: 3/24/2016 2:16:17 PM (UTC-6)
End Date/Time: 3/24/2016 2:48:20 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 ACID A2
Sample name: SFWB8-15-2 ACID A2
Sample Mass: 18.292 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-2 ACID A2 second attempt

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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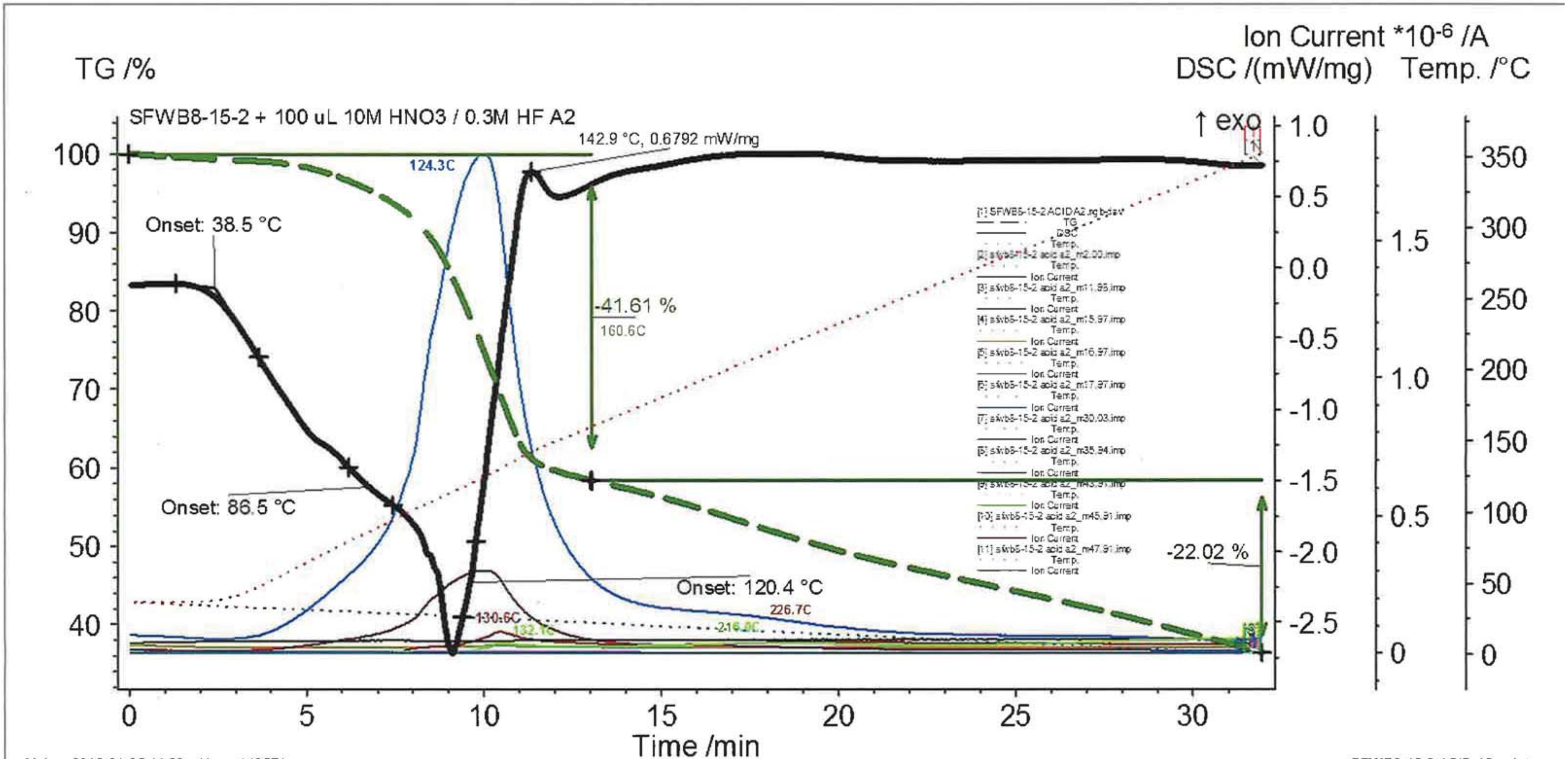

Main 2016-04-25 11:22 User: 113674

SFWB8-15-2 ACID A2.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 ACID A2.ngb-dsv	2016-03-24	SFWB8-15-2 ACID A2	SFWB8-15-2 ACID A2	18.292	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 acid a2_m2.00.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 2.00		1/1	37°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 acid a2_m11.98.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 11.98		1/1	37°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 acid a2_m15.97.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 15.97		1/1	37°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 acid a2_m16.97.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 16.97		1/1	37°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 acid a2_m17.97.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 17.97		1/1	37°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 acid a2_m30.03.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 30.03		1/1	37°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 acid a2_m35.94.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 35.94		1/1	37°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 acid a2_m43.91.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 43.91		1/1	37°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 acid a2_m45.91.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 45.91		1/1	37°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 acid a2_m47.91.imp	2016-03-24	sfwb8-15-2 acid a2	Mass 47.91		1/1	37°C/1.1(K/min)/1°C		---

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MATL NAME: SFW88-15-L ACID A2 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-24-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916/WIPP022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-24-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-24-16 Date	
Date & Time Sample Vial Opened	**Date:** 03/24/16 *(mm / dd / yy)* **Time:** 14:05 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6@31.1°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date

MATL NAME: SFWB8-15-2 ACID A2 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	**Date:** 3-24-16 **Time:** 14:10	Mm/dd/yy – 24 hr.	N/A	Dmw Initials 113674 Z No. 3-24-16 Date	
Crucible / Pan Tare Wt.	0.16484	grams	N/A	Dmw Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date
Net Sample Weight	0.01894		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	Dmw Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 05	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	wIPP350CBaseline030816.ngb~~-dsv~~ -BSV Dmw 113674 4-28-16 Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2 ACID A2 **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-24-16 Time Started: 14:17 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.9	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2 ACID A2.ngb-dsv **ThermoStar** Filename: SFWB8-15-2 ACID A2.MDC	N/A	N/A	DMW Initials 113677 Z No. 3-24-16 Date	
Proteus Data	**Mass Changes:** 1) -41.61 Temp. Range (RT-160.6°C) 2) -22.02 Temp. Range (160.6-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -63.63%	Wt. % / °C	Total < 0.4	DMW Initials 113677 Z No. 3-24-16 Date	Initials 120527 Z No. 3/24/16 Date

Notes:

MATL NAME: SFWB8-15-2 ACID A2 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (124.3°C) 2) CO_2 Peak T (132.1, 216.0°C) 3) NO/NO_2 Peak T (130.6, 226.7°C) 4) ______ Peak T (__________) 5) ______ Peak T (__________)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-24-16 Date	
Total Moisture (H_2O)	Total Moisture = ________ mg ________ Wt % Date of most recent calibration: ________ mm / dd / yy % Error (RSD, 1s): N/A = 100 x ________ ÷ ________ Std. Error Slope 113674 3-24-16 DMW	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-24-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A summary

1) Cal File No. not required for UPC instruments
2) calib. Thermometer for Water Chiller not required
3) Temp. Profile to 1100°c not required
4) Seal q. RH/Temp not required
5) Moisture content NOT REQUIRED

DMW / 113674 / 3-24-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-2 ACID B.ngb-dsv	**Sample identity:**	SFWB8-15-2 ACID B
Date/Time:	3/25/2016 10:42:35 AM (UTC-6)	**Sample name:**	SFWB8-15-2 ACID B
End Date/Time:	3/25/2016 11:14:36 AM (UTC-6)	**Sample Mass:**	15.59 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-2 ACID B - 2 white kernels

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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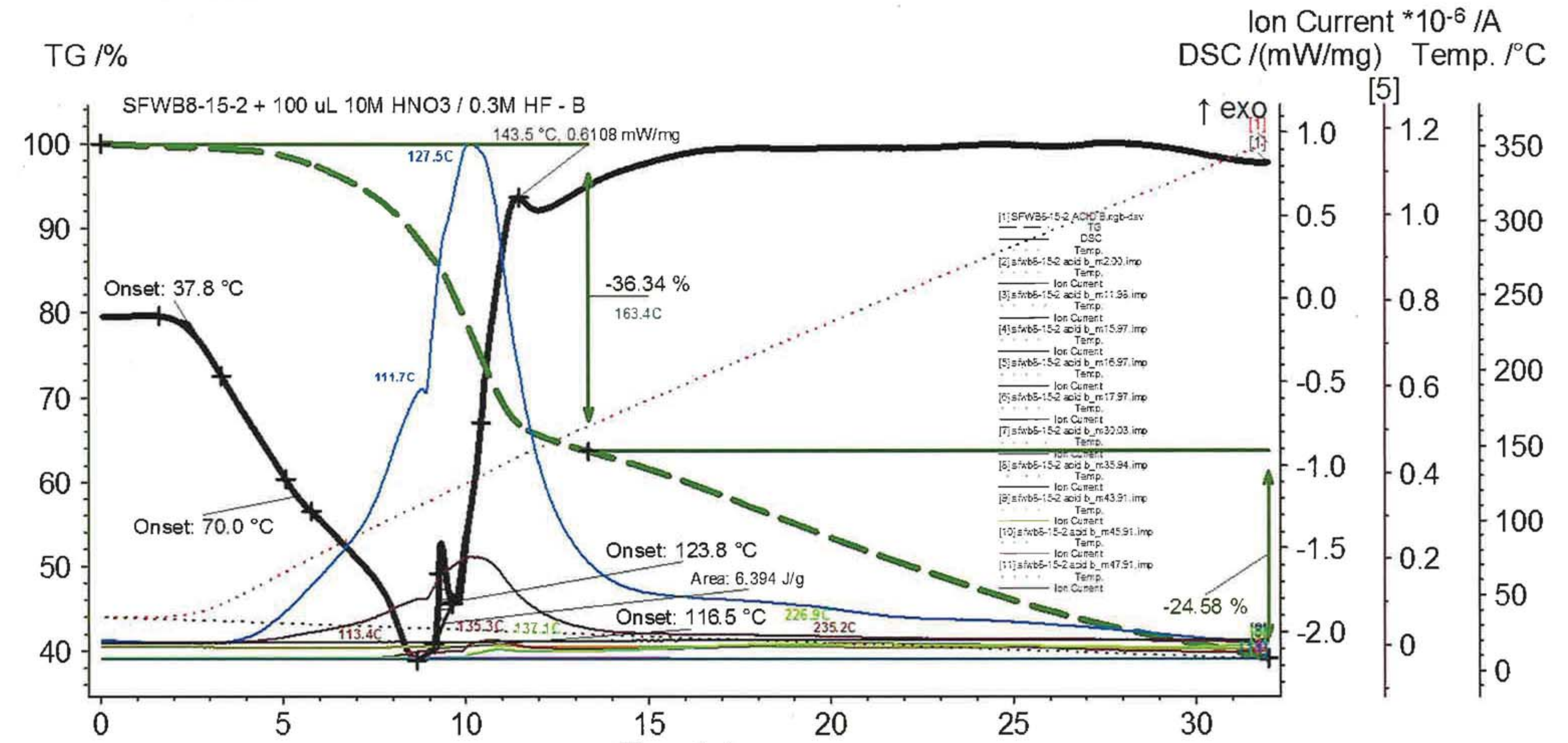
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-2 ACID B.ngb-dsv
Date/Time:	3/25/2016 10:42:35 AM (UTC-6)
End Date/Time:	3/25/2016 11:14:36 AM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-2 ACID B
Sample name:	SFWB8-15-2 ACID B
Sample Mass:	15.59 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 ACID B - 2 white kernels

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	37.8 °C	1.6 min	6.4 min
Onset (DSC)	70.0 °C	5.1 min	7.9 min
Onset (DSC)	116.5 °C	8.7 min	9.4 min
Peak (DSC)	143.5 °C/0.6108 mW/mg	9.7 min	12.1 min
Onset (DSC)	123.8 °C	9.6 min	11.6 min
Area (DSC),o	6.394 J/g	9.1 min	9.6 min



Main 2016-04-25 11:32 User: 113674

SFWB8-15-2 ACID B.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 ACID B.ngb-dsv	2016-03-25	SFWB8-15-2 ACID B	SFWB8-15-2 ACID B	15.59	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 acid b_m2.00.imp	2016-03-25	sfwb8-15-2 acid b	Mass 2.00		1/1	36°C/0.9(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 acid b_m11.98.imp	2016-03-25	sfwb8-15-2 acid b	Mass 11.98		1/1	36°C/0.9(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 acid b_m15.97.imp	2016-03-25	sfwb8-15-2 acid b	Mass 15.97		1/1	36°C/0.9(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 acid b_m16.97.imp	2016-03-25	sfwb8-15-2 acid b	Mass 16.97		1/1	36°C/0.9(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 acid b_m17.97.imp	2016-03-25	sfwb8-15-2 acid b	Mass 17.97		1/1	36°C/0.9(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 acid b_m30.03.imp	2016-03-25	sfwb8-15-2 acid b	Mass 30.03		1/1	36°C/0.9(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 acid b_m35.94.imp	2016-03-25	sfwb8-15-2 acid b	Mass 35.94		1/1	36°C/0.9(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 acid b_m43.91.imp	2016-03-25	sfwb8-15-2 acid b	Mass 43.91		1/1	36°C/0.9(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 acid b_m45.91.imp	2016-03-25	sfwb8-15-2 acid b	Mass 45.91		1/1	36°C/0.9(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 acid b_m47.91.imp	2016-03-25	sfwb8-15-2 acid b	Mass 47.91		1/1	36°C/0.9(K/min)/1°C		---

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MATL NAME: SFWB8.15-2 ACID B **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-25-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPP temp022916 /WIPP 022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-25-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-25-16 Date
Date & Time Sample Vial Opened	Date: 03/25/16 *(mm / dd / yy)* Time: 10:31 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.1°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-25-16 Date Initials 120527 Z No. 3/25/16 Date

MATL NAME: SFWB8-15-2 ACID B **Location:** G220

Sample Weights (Section 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
Date & Time Sample Weighed	**Date:** 03-25-16 **Time:** 10:34	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-25-16 Date	
Crucible / Pan Tare Wt.	0.16958	grams	N/A	DMW Initials 113674 Z No. 3-25-16 Date	Initials 120527 Z No. 3/25/16 Date
Net Sample Weight	0.01559		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Evacuation / Backfill	Sample Chamber Evacuated? YES / NO *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-25-16 Date	Initials 120527 Z No. 3/25/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 27	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP 350C Baseline 030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2 ACID B **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	dmw Initials	Initials
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3·25·16 Time Started: 10:42 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A	113674 Z No. 3-25-16 Date	120527 Z No. 3/25/16 Date
ThermoStar Reference	Sample Temp. at Start: 36.0	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2 ACID B.ngb-dsv. **ThermoStar** Filename: SFWB8-15-2 ACID B.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-25-16 Date	
Proteus Data	**Mass Changes:** 1) -36.34% Temp. Range (RT-163.4°C) 2) -24.58% Temp. Range (163.4°C-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -60.92%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-25-16 Date	Initials 120527 Z No. 3/25/16 Date

Notes:

MATL NAME: SFWB8-15-2 ACID B **Location:** G220

____

Gas Data (Section 5.6)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
ThermoStar Data	Volatile Species: 1) H2O/OH Peak T (127.5°C) 2) CO2 Peak T (137.1, 226.9°C) 3) NO/NO2 Peak T (113.4, 155.3, 255.2)°C 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3·25·16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): ______ = 100 x ______ ÷ ______ Std. Error Slope N/A DMW 113674 3-25-16	Wt. %	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-25-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 95012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/25/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A summary

1) Cal file # does not exist for UPC items
2) Calib. thermometer for water chiller not required
3) Temp profile to 1100°C not required
4) Seal Temp/%RH not required
5) Moisture content not required

DMW 113674 3-25-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-2 ACID C.ngb-dsv	**Sample identity:**	SFWB8-15-2 ACID C
Date/Time:	3/25/2016 1:35:22 PM (UTC-6)	**Sample name:**	SFWB8-15-2 ACID C
End Date/Time:	3/25/2016 2:07:22 PM (UTC-6)	**Sample Mass:**	7.668 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-2 ACID C 3rd run white blob

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

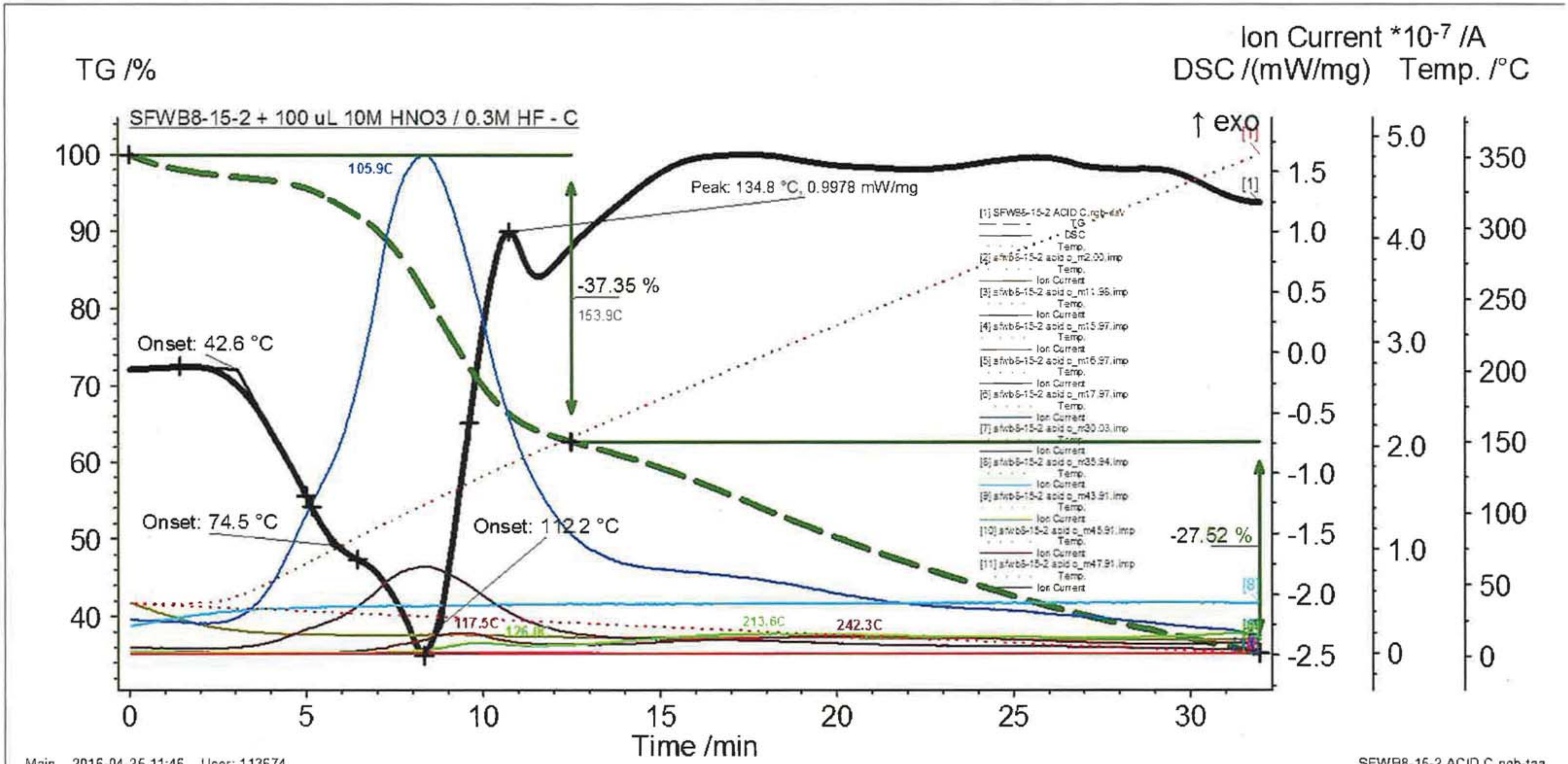
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-2 ACID C.ngb-dsv
Date/Time:	3/25/2016 1:35:22 PM (UTC-6)
End Date/Time:	3/25/2016 2:07:22 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-2 ACID C
Sample name:	SFWB8-15-2 ACID C
Sample Mass:	7.668 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 ACID C 3rd run white blob

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	42.6 °C	1.4 min	6.4 min
Onset (DSC)	74.5 °C	5.0 min	8.0 min
Onset (DSC)	112.2 °C	8.3 min	11.0 min
Peak (DSC)	134.8 °C/0.9978 mW/mg	8.8 min	11.5 min



Main 2016-04-25 11:46 User: 113674

SFWB8-15-2 ACID C.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 ACID C.ngb-dsv	2016-03-25	SFWB8-15-2 ACID C	SFWB8-15-2 ACID C	7.668	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 acid c_m2.00.imp	2016-03-25	sfwb8-15-2 acid c	Mass 2.00		1/1	38°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 acid c_m11.98.imp	2016-03-25	sfwb8-15-2 acid c	Mass 11.98		1/1	38°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 acid c_m15.97.imp	2016-03-25	sfwb8-15-2 acid c	Mass 15.97		1/1	38°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 acid c_m16.97.imp	2016-03-25	sfwb8-15-2 acid c	Mass 16.97		1/1	38°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 acid c_m17.97.imp	2016-03-25	sfwb8-15-2 acid c	Mass 17.97		1/1	38°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 acid c_m30.03.imp	2016-03-25	sfwb8-15-2 acid c	Mass 30.03		1/1	38°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 acid c_m35.94.imp	2016-03-25	sfwb8-15-2 acid c	Mass 35.94		1/1	38°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 acid c_m43.91.imp	2016-03-25	sfwb8-15-2 acid c	Mass 43.91		1/1	38°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 acid c_m45.91.imp	2016-03-25	sfwb8-15-2 acid c	Mass 45.91		1/1	38°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 acid c_m47.91.imp	2016-03-25	sfwb8-15-2 acid c	Mass 47.91		1/1	38°C/1.1(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-2 ACID C **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-25-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPtemp022916 / WIPP022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-25-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-25-16 Date
Date & Time Sample Vial Opened	Date: 03/25/16 *(mm / dd / yy)* Time: 13:28 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.12	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-25-16 Date Initials 120527 Z No. 3/25/16 Date

MATL NAME: SFWB8-15-2 ACIDC **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-25-16 Time: 13:30	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-25-16 Date	
Crucible / Pan Tare Wt.	0.16688	grams	N/A	DMW Initials 113674 Z No. 3-25-16 Date	Initials 120527 Z No. 3/25/16 Date
Net Sample Weight	0.00812		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-25-16 Date	Initials 120527 Z No. 3/25/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 :27	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350CBaseline030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFW88-15-2 ACID C **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-25-16 Date	Initials 120527 Z No. 3/25/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-25-16 Time Started: 13:36 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 37.4	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFW88-15-2 ACID C.ngb-dsv **ThermoStar** Filename: SFW88-15-2 ACID C.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-25-16 Date	
Proteus Data	**Mass Changes:** 1) -37.35 Temp. Range (RT-153.9°C) 2) -27.52 Temp. Range (153.9-350°C) 3) ____ Temp. Range (____) 4) ____ Temp. Range (____) **Total mass Change:** 64.87%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-25-16 Date	Initials 120527 Z No. 3/25/16 Date

Notes:

MATL NAME: SFWB8-15-2 ACID C **Location:** G22U

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H_2O Peak T (105.8°C) 2) CO_2 Peak T (126.0, 213.6°C) 3) NO/NO_2 Peak T (117.5, 242.3°C) 4) _____ Peak T ( ) 5) _____ Peak T ( )	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-25-16 Date	
Total Moisture (H_2O)	Total Moisture = _____ mg _____ Wt % Date of most recent calibration: / / mm dd yy % Error (RSD, 1s): _____ = 100 x _____ Std. Error / Slope N/A DMW | 113674 | 3-25-16	Wt. %	< 0.32	DM Initials _____ Z No. _____ Date	_____ Initials _____ Z No. _____ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-23-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary
1) no cal. File # for User Performed Calib. - not required
2) Calibrated Thermometer for water-chiller not required by WIPP
3) Temp Profile to 1100°C not required for this work
4) Seal RH/Temp not required for WIPP
5) Moisture Content not required for run 3017
DMW / 113674 / 3-25-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-2 200ul acid.ngb-dsv	**Sample identity:**	SFWB8-15-2 200ul acid
Date/Time:	4/12/2016 4:00:11 PM (UTC-6)	**Sample name:**	SFWB8-15-2 200ul acid
End Date/Time:	4/12/2016 4:32:12 PM (UTC-6)	**Sample Mass:**	22.848 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-2 200ul acid acid only to see if 200 ul makes a difference

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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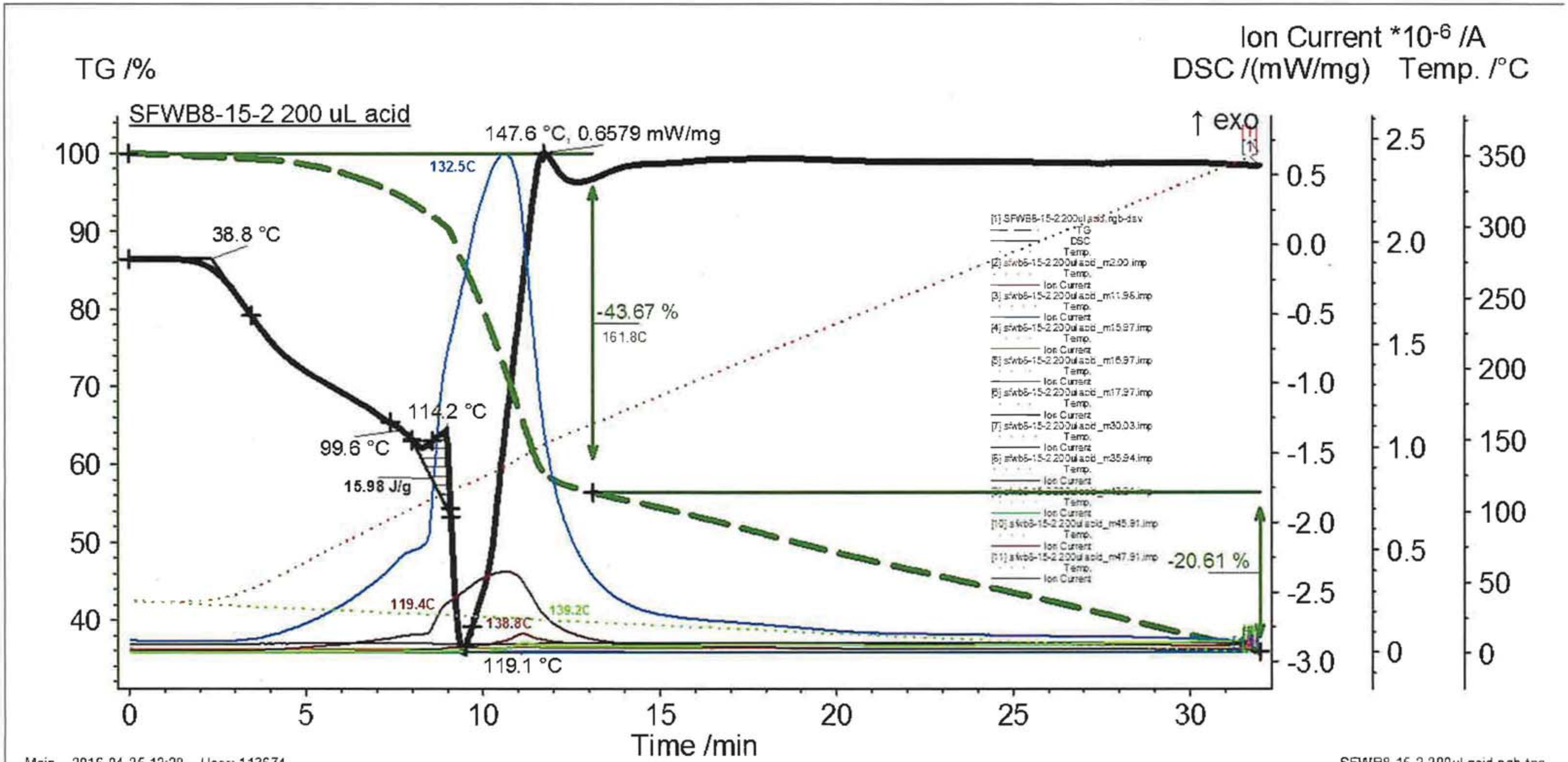
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2 200ul acid.ngb-dsv
Date/Time: 4/12/2016 4:00:11 PM (UTC-6)
End Date/Time: 4/12/2016 4:32:12 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 200ul acid
Sample name: SFWB8-15-2 200ul acid
Sample Mass: 22.848 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 200ul acid acid only to see if 200 ul makes a difference

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Mass Change (TG)	-43.67 %	0.0 min	13.1 min
Mass Change (TG)	-20.61 %	13.1 min	32.0 min
Peak (DSC)	147.6 °C/0.6579 mW/mg	9.9 min	12.9 min
Onset (DSC)	38.8 °C	0.0 min	7.3 min
Onset (DSC)	99.6 °C	7.4 min	8.6 min
Onset (DSC)	114.2 °C	8.6 min	9.6 min
Onset (DSC)	119.1 °C	9.5 min	10.4 min
Area (DSC),o	15.98 J/g	8.0 min	9.1 min



Main 2016-04-25 12:29 User: 113674

SFWB8-15-2 200ul acid.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mas...	S...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 200ul acid.ngb-dsv	2016-04-12	SFWB8-15-2 200ul acid	SFWB8-15-2 200ul acid	22.848	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 200ul acid_m2.00.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 2.00		1/1	38°C/1.2(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 200ul acid_m11.98.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 11.98		1/1	38°C/1.2(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 200ul acid_m15.97.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 15.97		1/1	38°C/1.2(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 200ul acid_m16.97.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 16.97		1/1	38°C/1.2(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 200ul acid_m17.97.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 17.97		1/1	38°C/1.2(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 200ul acid_m30.03.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 30.03		1/1	38°C/1.2(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 200ul acid_m35.94.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 35.94		1/1	38°C/1.2(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 200ul acid_m43 91 imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 43 91		1/1	38°C/1 2(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 200ul acid_m45.91.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 45.91		1/1	38°C/1.2(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 200ul acid_m47.91.imp	2016-04-12	sfwb8-15-2 200ul acid	Mass 47.91		1/1	38°C/1.2(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-2 200uL AcI) **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-12-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTemp 022916 / WIPP 022916	N/A	02/28/17	DMW Initials 113674 Z No. 4-12-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-12-16 Date	
Date & Time Sample Vial Opened	Date: 04/12/16 *(mm / dd / yy)* Time: 15:50 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6%/32.6°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date

MATL NAME: SFWB8-15-2 200mL ACID **Location:** G-220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 04-12-16 Time: 15:52	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-12-16 Date	
Crucible / Pan Tare Wt.	0.16886	grams	N/A	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date
Net Sample Weight	0.02425		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10:41	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP 350C Baseline 030816.ngb-tsv ✓ *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2 200uL Acid **Location:** G270

Run Parameters (Sections 5.4 & 5.6)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 4-12-16 Time Started: 16:04 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 37.8	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP SFWB8-15-2 200uLacid.ngb-dsv **Netzsch Measure** Filename: SFWB8-15-2 200uLacid.MDC **ThermoStar** Filename:	N/A	N/A	DMW Initials 113674 Z No. 4-12-16 Date	
Proteus Data	**Mass Changes:** 1) -43.67% Temp. Range (RT-161.8°C) 2) -20.61% Temp. Range (161.8-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -64.28%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-12-16 Date	Initials 120527 Z No. 4/12/16 Date

Notes:

MATL NAME: SFWB8-15-2 200 mL ACID **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H2O Peak T (132.5°C) 2) CO2 Peak T (139.2°C) 3) NO/NO2 Peak T (119.4, 138.8°C) 4) _______ Peak T (__________) 5) _______ Peak T (__________)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-12-16 Date	
Total Moisture (H_2O)	Total Moisture = __________ mg __________ Wt % Date of most recent calibration: __/__/__ 113674 mm dd yy % Error (RSD, 1s): DMW N/A __________ = 100 x ________ ÷ ________ Std. Error Slope	4-12-16 Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-12-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A summary

1) NO CAL FILE # for user calibrated equipment
2) NO requirement to go to 1100°c for non-3013, which this is.
3) NON-3013 has no seal requirement
4) NO requirement for calib. thermometer for water chiller on non-3013 runs
5) no requirement for Moisture analysis or quantitation for WIPP.

DMW 113674 4-12-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-2 PUAM1.ngb-dsv	**Sample identity:**	SFWB8-15-2 PUAM1
Date/Time:	3/30/2016 3:03:47 PM (UTC-6)	**Sample name:**	SFWB8-15-2 PUAM1
End Date/Time:	3/30/2016 3:35:46 PM (UTC-6)	**Sample Mass:**	12.48 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-2 PUAM1 - first spiked run

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

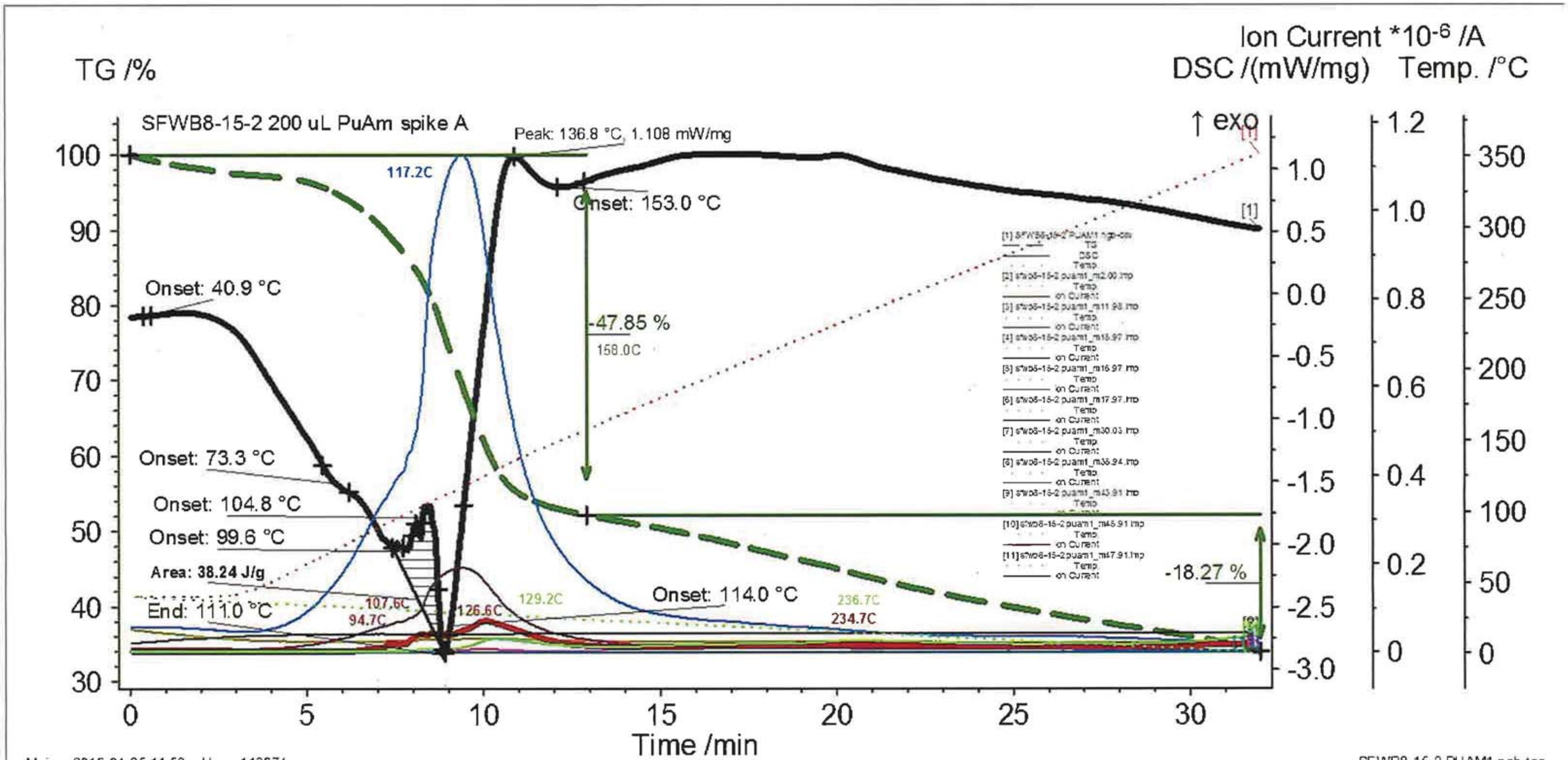
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2 PUAM1.ngb-dsv
Date/Time: 3/30/2016 3:03:47 PM (UTC-6)
End Date/Time: 3/30/2016 3:35:46 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 PUAM1
Sample name: SFWB8-15-2 PUAM1
Sample Mass: 12.48 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 PUAM1 - first spiked run

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	40.9 °C	0.4 min	5.4 min
Onset (DSC)	73.3 °C	5.5 min	7.0 min
Onset (DSC)	99.6 °C	7.7 min	8.2 min
Onset (DSC)	104.8 °C	8.1 min	8.7 min
Onset (DSC)	114.0 °C	8.9 min	9.7 min
Onset (DSC)	153.0 °C	12.1 min	20.1 min
Peak (DSC)	136.8 °C/1.108 mW/mg	9.1 min	12.3 min
End (DSC)	111.0 °C	8.3 min	8.9 min
Area (DSC),o	38.24 J/g	7.4 min	8.8 min



Main 2016-04-25 11:58 User: 113674

SFWB8-15-2 PUAM1.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segme...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 PUAM1.ngb-dsv	2016-03-30	SFWB8-15-2 PUAM1	SFWB8-15-2 PUAM1	12.48	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 puam1_m2.00.imp	2016-03-30	sfwb8-15-2 puam1	Mass 2.00		1/1	41°C/1.3(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 puam1_m11.98.imp	2016-03-30	sfwb8-15-2 puam1	Mass 11.98		1/1	41°C/1.3(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 puam1_m15.97.imp	2016-03-30	sfwb8-15-2 puam1	Mass 15.97		1/1	41°C/1.3(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 puam1_m16.97.imp	2016-03-30	sfwb8-15-2 puam1	Mass 16.97		1/1	41°C/1.3(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 puam1_m17.97.imp	2016-03-30	sfwb8-15-2 puam1	Mass 17.97		1/1	41°C/1.3(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 puam1_m30.03.imp	2016-03-30	sfwb8-15-2 puam1	Mass 30.03		1/1	41°C/1.3(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 puam1_m35.94.imp	2016-03-30	sfwb8-15-2 puam1	Mass 35.94		1/1	41°C/1.3(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 puam1_m43.91.imp	2016-03-30	sfwb8-15-2 puam1	Mass 43.91		1/1	41°C/1.3(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 puam1_m45.91.imp	2016-03-30	sfwb8-15-2 puam1	Mass 45.91		1/1	41°C/1.3(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 puam1_m47.91.imp	2016-03-30	sfwb8-15-2 puam1	Mass 47.91		1/1	41°C/1.3(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-7 PuAm1 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Current DOP effective revision	R11	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-30-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTemp022916/WIPP 022916	N/A	02/28/17	DMW Initials
Temperature & Humidity Monitor	041888	10-25-16	113674 Z No.
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	3-30-16 Date

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

<table>
<tr><th>Description</th><th>Cal. File No.</th><th>Cal. Exp. Date</th><th>Performed By</th><th colspan="2">Description</th></tr>
<tr><td>LANMAS / LAMCAS Balance ID</td><td>B366</td><td>N/A</td><td>N/A</td><td colspan="2" rowspan="2">DMW
Initials
113674
Z No.
3-30-16
Date</td></tr>
<tr><td>Date & Time Sample Vial Opened</td><td>Date: 03/30/16
(mm / dd / yy)
Time: 14:58
(24 hour)</td><td>N/A</td><td>N/A</td></tr>
<tr><td>Glovebox Conditions</td><td>Seal %RH/Temp.: N/A
GB235%RH/Temp.: 0.6/36.8°C</td><td>%RH/C°</td><td>%RH < 15,
Seal %RH not >3% more than GB235 %RH</td><td>DMW
Initials
113674
Z No.
3-30-16
Date</td><td>Initials
120527
Z No.
3/30/16
Date</td></tr>
</table>

MATL NAME: SFWB8-15-2 PUAM1 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	**Date:** 03-30-16 DMW 113674 3-30-16 **Time:** ~~13:13~~ 15:00	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-30-16 Date	
Crucible / Pan Tare Wt.	0.16796	grams	N/A	DMW Initials 113674 Z No. 3-30-16 Date	Initials 120527 Z No. 3/30/16 Date
Net Sample Weight	0.01248		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-30-16 Date	Initials 120527 Z No. 3/30/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 13 : 17	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP 350C Baseline 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

DMW 113674 3-30-16

MATL NAME: SFWB8-15-2 PuAm1 **Location:** ~~DA~~ G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-30-16 Date	Initials 120527 Z No. 3/30/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 03-30-16 Time Started: 15:04 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 40.9	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2 PuAm1.ngb-dsv **ThermoStar** Filename: SFWB8-15-2 PuAm1.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-30-16 Date	
Proteus Data	**Mass Changes:** 1) -47.85 Temp. Range (RT-158.0°C) 2) -18.27 Temp. Range (158.0-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -66.12%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-30-16 Date	Initials 120527 Z No. 3/30/16 Date

Notes:

PMT2-MPR-DOP-015,R3	***Thermal Analysis with Off-Gas Analysis by Quadrupole Mass Spectrometry***	Page 57 of 63

MATL NAME: SFWB 8-15-2-PUAM1 **Location:** 6220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (117.2°C) 2) CO_2 Peak T (120.2, 236.7°C) 3) NO/NO_2 Peak T (94.7/107.6/126.6)/234.7°C 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-30-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: ___/___/___ mm dd yy % Error (RSD, 1s): ______ = 100 x ______ ÷ ______ Std. Error Slope N/A DMW 113674 3-30-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-30-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A summary

1) Cal File # not required for user calibrated equipment
2) Water temp. not required for WIPP analyses (water chiller T°)
3) Temp. Profile not required to go to 1100°C for WIPP
4) No Seal %RH/Temp required for WIPP samples
5) Moisture content measurement not required for WIPP

DMW 113674 - 3/30/16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-2 PuAm 2.ngb-dsv
Date/Time: 4/8/2016 9:40:42 AM (UTC-6)
End Date/Time: 4/8/2016 10:12:44 AM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-2 PuAm 2
Sample name: SFWB8-15-2 PuAm 2
Sample Mass: 22.504 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-2 PuAm 2 second attempt spiked run

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-2 PuAm 2.ngb-dsv
Date/Time:	4/8/2016 9:40:42 AM (UTC-6)
End Date/Time:	4/8/2016 10:12:44 AM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-2 PuAm 2
Sample name:	SFWB8-15-2 PuAm 2
Sample Mass:	22.504 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 PuAm 2 second attempt spiked run

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	37.0 °C	0.0 min	4.4 min
Onset (DSC)	102.0 °C	7.9 min	8.3 min
Onset (DSC)	94.8 °C	7.2 min	7.7 min
End (DSC)	110.2 °C	8.3 min	8.8 min
Area (DSC),o	6.185 J/g	7.9 min	8.8 min
Peak (DSC)	141.1 °C/0.5028 mW/mg	10.3 min	12.0 min
Onset (DSC)	115.2 °C	9.0 min	9.4 min

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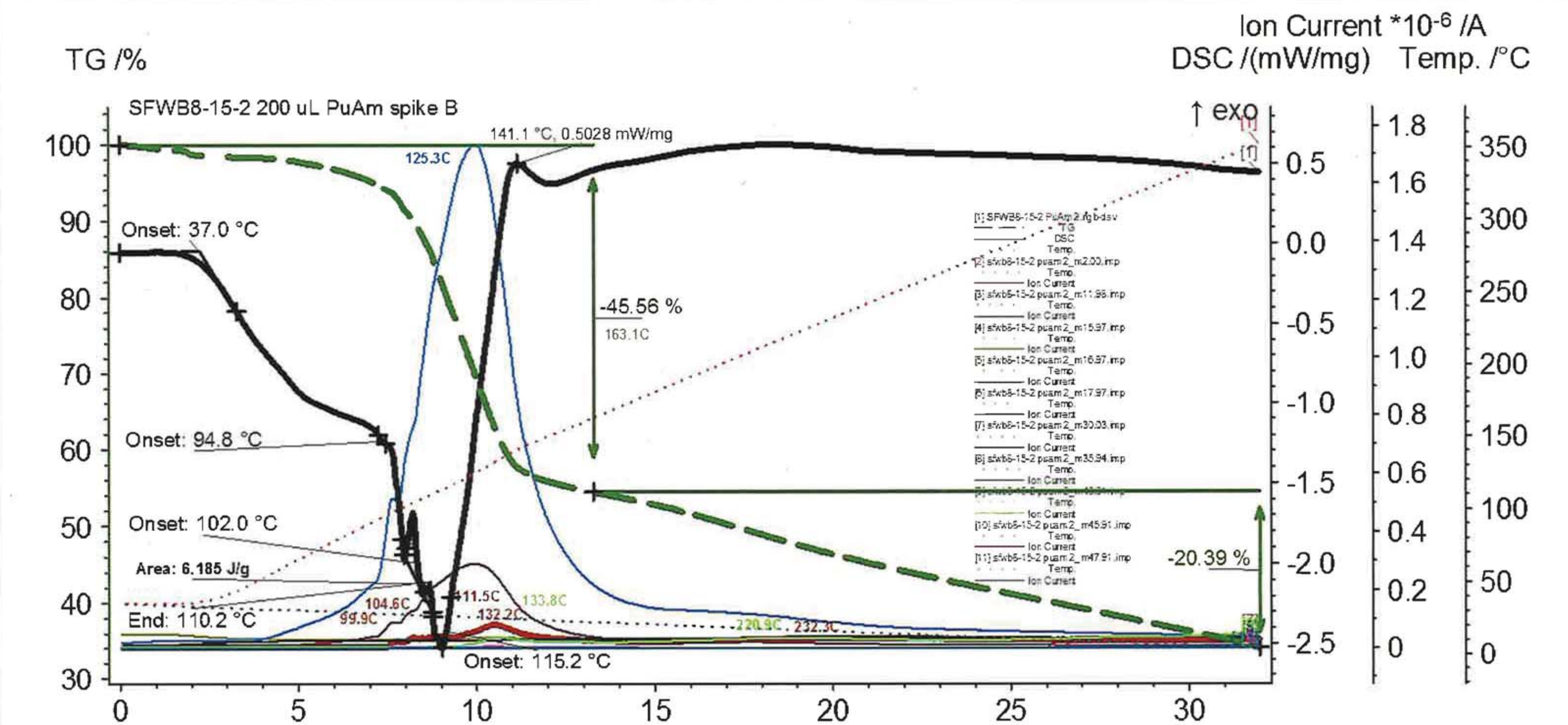

Main 2016-04-25 12:16 User: 113674

SFWB8-15-2 PuAm 2.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 PuAm 2.ngb-dsv	2016-04-08	SFWB8-15-2 PuAm 2	SFWB8-15-2 PuAm 2	22.504	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 puam 2_m2.00.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 2.00		1/1	35°C/1.0(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 puam 2_m11.98.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 11.98		1/1	35°C/1.0(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 puam 2_m15.97.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 15.97		1/1	35°C/1.0(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 puam 2_m16.97.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 16.97		1/1	35°C/1.0(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 puam 2_m17.97.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 17.97		1/1	35°C/1.0(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 puam 2_m30.03.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 30.03		1/1	35°C/1.0(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 puam 2_m35.94.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 35.94		1/1	35°C/1.0(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 puam 2_m43.91.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 43.91		1/1	35°C/1.0(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 puam 2_m45.91.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 45.91		1/1	35°C/1.0(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 puam 2_m47.91.imp	2016-04-08	sfwb8-15-2 puam 2	Mass 47.91		1/1	35°C/1.0(K/min)/1°C		---

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MATL NAME: SFWB8-15-2 PUAM2 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-8-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTemp022916 / WIPP 022916	N/A	02/28/17	DMW Initials 113674 Z No. 4-8-16 Date
Temperature & Humidity Monitor	049888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-8-16 Date	
Date & Time Sample Vial Opened	Date: 04/08/16 *(mm / dd / yy)* Time: 9:33 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.67/30.0°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date

PMT2-MPR-DOP-015,R3	***Thermal Analysis with Off-Gas Analysis by Quadrupole Mass Spectrometry***	Page 54 of 63

MATL NAME: SFWB8-15-2 PuAm2 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 4/8/16 Time: 9:36	Mm/dd/yy – 24 hr.	N/A	Dmw Initials 113674 Z No. 4-8-16 Date	
Crucible / Pan Tare Wt.	0.16957	grams	N/A	Dmw Initials 113674 Z No. 4-8-16 Date	Initials 12052 Z No. 4/8/16 Date
Net Sample Weight	0.02260		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / NO *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	Dmw Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 9 : 20	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP 350C Baseline 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2 PuAm2 **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	Dmw Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 04/08/16 Time Started: 9:41 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 35.3	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2 PuAm2.ngb-dsv **ThermoStar** Filename: SFWB8-15-2 PuAm2.MOC	N/A	N/A	Dmw Initials 113674 Z No. 4-8-16 Date	
Proteus Data	**Mass Changes:** 1) -45.56 Temp. Range (RT-163.1°C) 2) -20.39 Temp. Range (163.1-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -65.95%	Wt. % / °C	Total < 0.4	Dmw Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date

Notes:

MATL NAME: SFW B8-15-2 PuAm2 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (125.3) 2) CO_2 Peak T (133.8 / 220.9°C) 3) NO/NO_2 Peak T (99.9 / 104.6 / 111.5 / 132.2 / 232.3 °C) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-8-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __ / __ / __ (mm dd yy) % Error (RSD, 1s): N-A = 100 x ____ ÷ ____ Std. Error Slope DMW 113874 4-8-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-8-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A* SUMMARY

1) User calibrated equipment has no cal. File # - not required
2) Water Chiller temp. not required - calibrated thermometer not required
3) For non 3013 - samples can be heated to any temperature - not required to be 1100°C
4) For non 3013, seal temp/%RH data not required
5) For WIPP - H_2O quantitation not required.

DMW 113674 4-8-16

Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-2 PuAm 3.ngb-dsv
Date/Time:	4/8/2016 3:33:26 PM (UTC-6)
End Date/Time:	4/8/2016 4:05:26 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-2 PuAm 3
Sample name:	SFWB8-15-2 PuAm 3
Sample Mass:	10.652 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-2 PuAm 3 - 3rd spiked run

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	38.8 °C	0.0 min	5.5 min
Onset (DSC)	95.0 °C	7.2 min	8.2 min
End (DSC)	103.9 °C	7.2 min	8.2 min
Area (DSC),o	7.831 J/g	7.4 min	8.2 min
Peak (DSC)	137.2 °C/0.8207 mW/mg	8.7 min	11.9 min
Onset (DSC)	111.2 °C	8.6 min	9.8 min

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-2 PuAm 3.ngb-dsv	**Sample identity:**	SFWB8-15-2 PuAm 3
Date/Time:	4/8/2016 3:33:26 PM (UTC-6)	**Sample name:**	SFWB8-15-2 PuAm 3
End Date/Time:	4/8/2016 4:05:26 PM (UTC-6)	**Sample Mass:**	10.652 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-2 PuAm 3 - 3rd spiked run

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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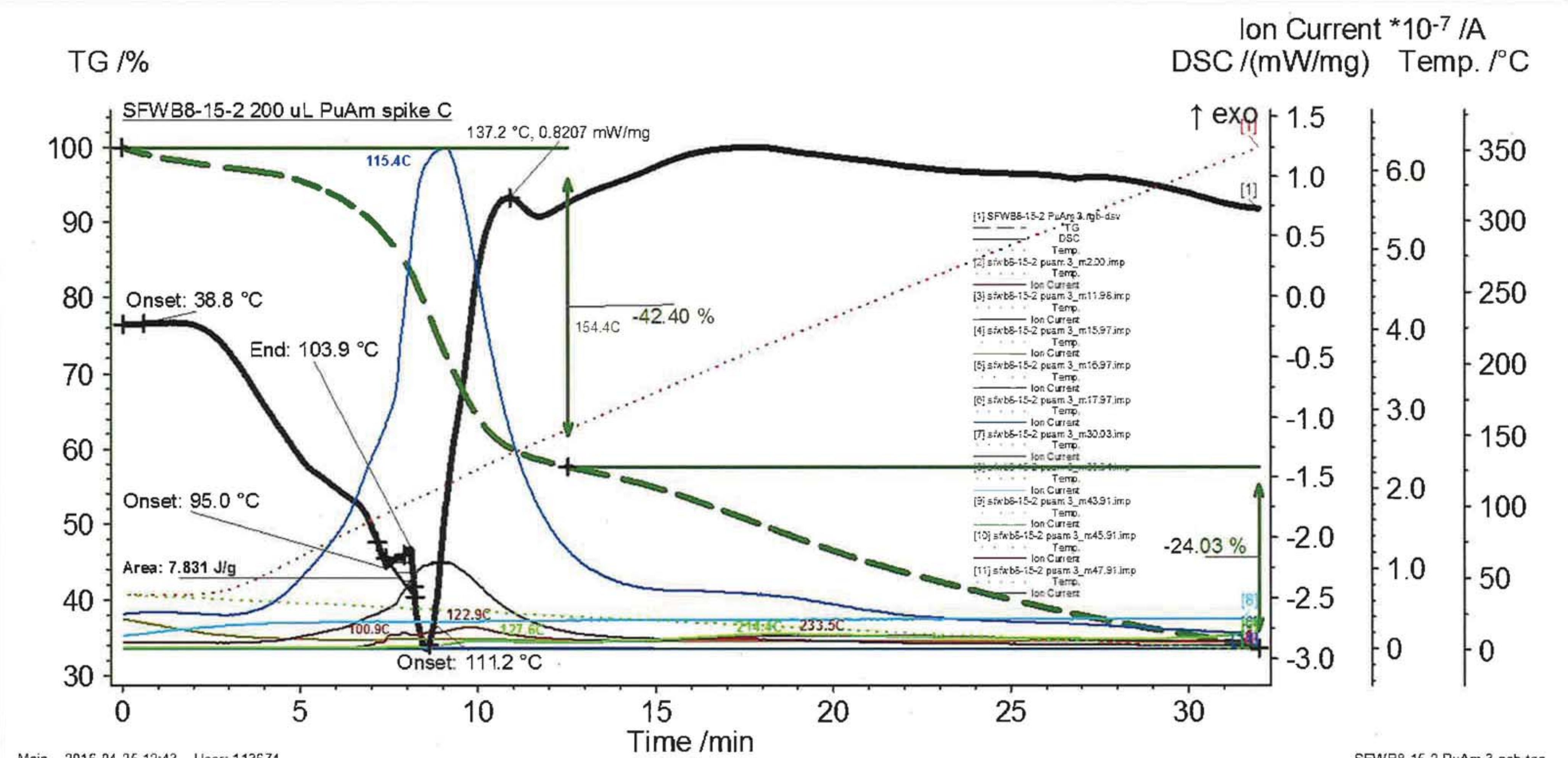

Main 2016-04-25 12:43 User: 113674

SFWB8-15-2 PuAm 3.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-2 PuAm 3.ngb-dsv	2016-04-08	SFWB8-15-2 PuAm 3	SFWB8-15-2 PuAm 3	10.652	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-2 puam 3_m2.00.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 2.00		1/1	39°C/1.2(K/min)/1°C		---
[3] QMS 403	sfwb8-15-2 puam 3_m11.98.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 11.98		1/1	39°C/1.2(K/min)/1°C		---
[4] QMS 403	sfwb8-15-2 puam 3_m15.97.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 15.97		1/1	39°C/1.2(K/min)/1°C		---
[5] QMS 403	sfwb8-15-2 puam 3_m16.97.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 16.97		1/1	39°C/1.2(K/min)/1°C		---
[6] QMS 403	sfwb8-15-2 puam 3_m17.97.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 17.97		1/1	39°C/1.2(K/min)/1°C		---
[7] QMS 403	sfwb8-15-2 puam 3_m30.03.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 30.03		1/1	39°C/1.2(K/min)/1°C		---
[8] QMS 403	sfwb8-15-2 puam 3_m35.94.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 35.94		1/1	39°C/1.2(K/min)/1°C		---
[9] QMS 403	sfwb8-15-2 puam 3_m43.91.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 43.91		1/1	39°C/1.2(K/min)/1°C		---
[10] QMS 403	sfwb8-15-2 puam 3_m45.91.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 45.91		1/1	39°C/1.2(K/min)/1°C		---
[11] QMS 403	sfwb8-15-2 puam 3_m47.91.imp	2016-04-08	sfwb8-15-2 puam 3	Mass 47.91		1/1	39°C/1.2(K/min)/1°C		---

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MATL NAME: SFWB8-15-2-PUAM3 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-8-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)*: TG / [✓ DSC] Cal. File Name: WTPPTemp 022916.ngb-tsv/WTPP022916.ngb.eiv	N/A	02/28/17	DMW Initials
Temperature & Humidity Monitor	041888	10-25-16	113674 Z No.
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	4-8-16 Date

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-8-16 Date	
Date & Time Sample Vial Opened	Date: 04/08/16 *(mm / dd / yy)* Time: 13:52 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.4 / 30.9°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date

MATL NAME: SFWB8-15-2 PuAm3 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 04/08/16 Time: 13:54	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-8-16 Date	
Crucible / Pan Tare Wt.	0.16787	grams	N/A	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
Net Sample Weight	0.01815		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 9:20	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350C Baseline 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-2 PuAm3 **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	Dmw Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 04/08/16 Time Started: 14:01 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.5	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-2 PuAm3.ngb-dsv **ThermoStar** Filename: SFWB8-15-2 PuAm3.MDC	N/A	N/A	Dmw Initials 113674 Z No. 4-8-16 Date	
Proteus Data	**Mass Changes:** 1) -42.40 Temp. Range (RT-154.4°C) 2)- 24.03 Temp. Range (154.4-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -66.43%	Wt. % / °C	Total < 0.4	Dmw Initials 113674 Z No. 4-8-16 Date	Initials 120527 Z No. 4/8/16 Date

Notes:

MATL NAME: SFW 88-15-2 Pu Am 3 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (115.4°C) 2) CO_2 Peak T (127.6, 214.4°C) 3) NO/NO_2 Peak T (100.9, 122.9, 233.5°C) 4) ______ Peak T ( ) 5) ______ Peak T ( )	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-8-16 Date	
Total Moisture (H_2O) N/A	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, ~~1s~~): 113674 4-8-16 DMW = 100 x ______ ÷ ______ Std. Error Slope	Wt. %	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-8-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 119274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A summary
1) user calibrated instrument has no cal. File #
2) Calib. Thermometer not required - Water temp. controlled by circulator.
3) Seal RH/Temp - not required for non-3013
4) Temp profile to 1100°C not req'd for non 3013
5) Total Moisture measurement not required for non-3013

DMW 113674, 4/8/16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4A 031516.ngb-dsv
Date/Time: 3/15/2016 3:56:56 PM (UTC-6)
End Date/Time: 3/15/2016 4:28:57 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-4A
Sample name: SFWB8-15-4A
Sample Mass: 22.87 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-4A FIRST RUN OF SURROGATE

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

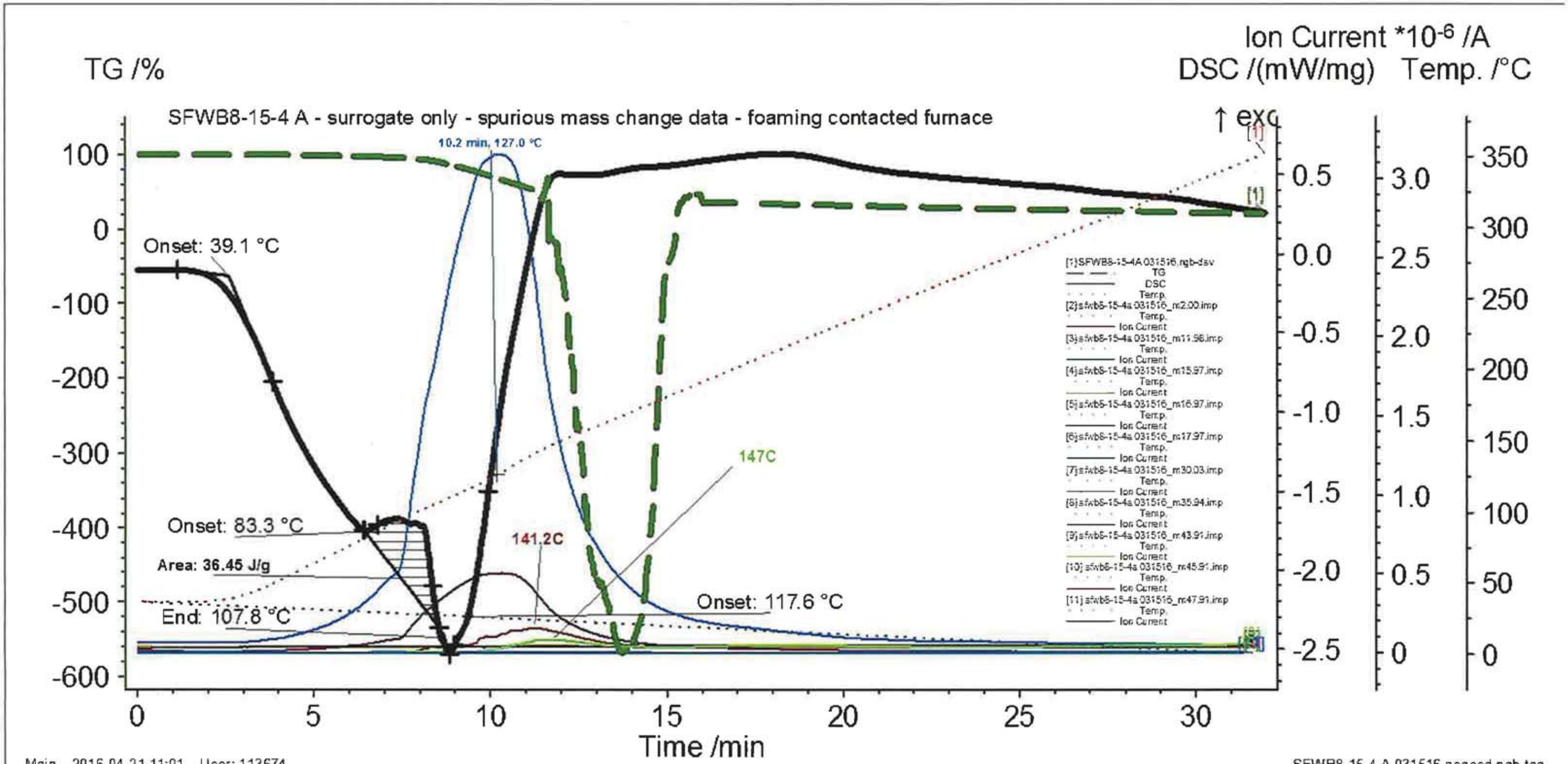
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-4A 031516.ngb-dsv
Date/Time:	3/15/2016 3:56:56 PM (UTC-6)
End Date/Time:	3/15/2016 4:28:57 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-4A
Sample name:	SFWB8-15-4A
Sample Mass:	22.87 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4A FIRST RUN OF SURROGATE

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	117.6 °C	9.0 min	12.1 min
Onset (DSC)	39.1 °C	1.1 min	5.6 min
Onset (DSC)	83.3 °C	6.4 min	7.6 min
End (DSC)	107.8 °C	7.8 min	8.8 min
Area (DSC),o	36.45 J/g	6.4 min	8.5 min



SFWB8-15-4 A - surrogate only - spurious mass change data - foaming contacted furnace

Main 2016-04-21 11:01 User: 113674

SFWB8-15-4 A 031516 nogood.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/mg	Segment	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-4A 031516.ngb-dsv	2016-03-15	SFWB8-15-4A	SFWB8-15-4A	22.87	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-4a 031516_m2.00.imp	2016-03-15	sfwb8-15-4a 031516	Mass 2.00		1/1	37°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4a 031516_m11.98.imp	2016-03-15	sfwb8-15-4a 031516	Mass 11.98		1/1	37°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4a 031516_m15.97.imp	2016-03-15	sfwb8-15-4a 031516	Mass 15.97		1/1	37°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4a 031516_m16.97.imp	2016-03-15	sfwb8-15-4a 031516	Mass 16.97		1/1	37°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4a 031516_m17.97.imp	2016-03-15	sfwb8-15-4a 031516	Mass 17.97		1/1	37°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4a 031516_m30.03.imp	2016-03-15	sfwb8-15-4a 031516	Mass 30.03		1/1	37°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4a 031516_m35.94.imp	2016-03-15	sfwb8-15-4a 031516	Mass 35.94		1/1	37°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-4a 031516_m43.91.imp	2016-03-15	sfwb8-15-4a 031516	Mass 43.91		1/1	37°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4a 031516_m45.91.imp	2016-03-15	sfwb8-15-4a 031516	Mass 45.91		1/1	37°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4a 031516_m47.91.imp	2016-03-15	sfwb8-15-4a 031516	Mass 47.91		1/1	37°C/1.1(K/min)/1°C		---

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3-15-16
DMW 113674

MATL NAME: ~~8~~ SFWB8-15-4A **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-15-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date DMW 113674 4-28-16	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916 / WIPP022916	N/A DMW 113674 3-15-16	02/~~29~~ 28/17	DMW Initials 113674 Z No. 3-15-16 Date
Temperature & Humidity Monitor	~~At~~ 041888	10·25·2016	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08-03-2016	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-15-16 Date	
Date & Time Sample Vial Opened	Date: 03/15/16 *(mm / dd / yy)* Time: 15:45 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/31.6°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-15-16 Date	DG Initials 107962 Z No. 3/15/16 Date

MATL NAME: SFWB8-15·4A **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-15-16 Time: 15:47	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-15-16 Date	
Crucible / Pan Tare Wt.	0.16693	grams	N/A	DMW Initials 113674 Z No. 3-15-16 Date	DG. Initials 107962 Z No. 3/15/16 Date
Net Sample Weight	0.02287		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-15-16 Date	DG. Initials 107962 Z No. 3/15/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 15 : 35	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP 350C BASELINE 030816 Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4A **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-15-16 Date	DG. Initials 107962 Z No. 3/15/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-15-16 Time Started: 15:57 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 37.0	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-4A 031516.ngb-dsv **ThermoStar** Filename: SFWB8-15-4A 031516.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-15-16 Date	
Proteus Data	**Mass Changes:** N/A 1) _______ Temp. Range (__________) 2) _______ Temp. Range (__________) 3) _______ Temp. Range (__________) 4) _______ Temp. Range (__________) **Total mass Change:** N/A	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-15-16 Date	DG. Initials 107962 Z No. 3/15/16 Date

Notes: Run no good - Foaming sample contacted furnace & ruined weight measrement DMW 113674 3-15-16

MATL NAME: SFWB8-15-4 A **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (127.0°C) 2) CO_2 Peak T (147 °C) 3) NO/NO_2 Peak T (141.2°C) 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-15-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: ___ / ___ / ___ (mm dd yy) % Error (RSD) = ______ = 100 x ______ ÷ ______ Std. Error Slope N/A DMW 113674 3-15-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-15-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

1) cal Files are User Calibrated
2) seal Temp/RH is a 3013-only requirement
3) mass changes not readable for this sample - made contact w/ furnace during run.
4) Remaining data spurious - as far as mass changes are concerned

DSC onsets & Gas data are good.

DMW 113674 3-15-16

5) Total moisture not required by customer

6) Calibrated thermometer for chiller water not required.

DMW 113674 4-28-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4B 031516.ngb-dsv
Date/Time: 3/16/2016 3:10:16 PM (UTC-6)
End Date/Time: 3/16/2016 3:42:17 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-4B 031616
Sample name: SFWB8-15-4B 031616
Sample Mass: 12.37 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-4B o31616

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

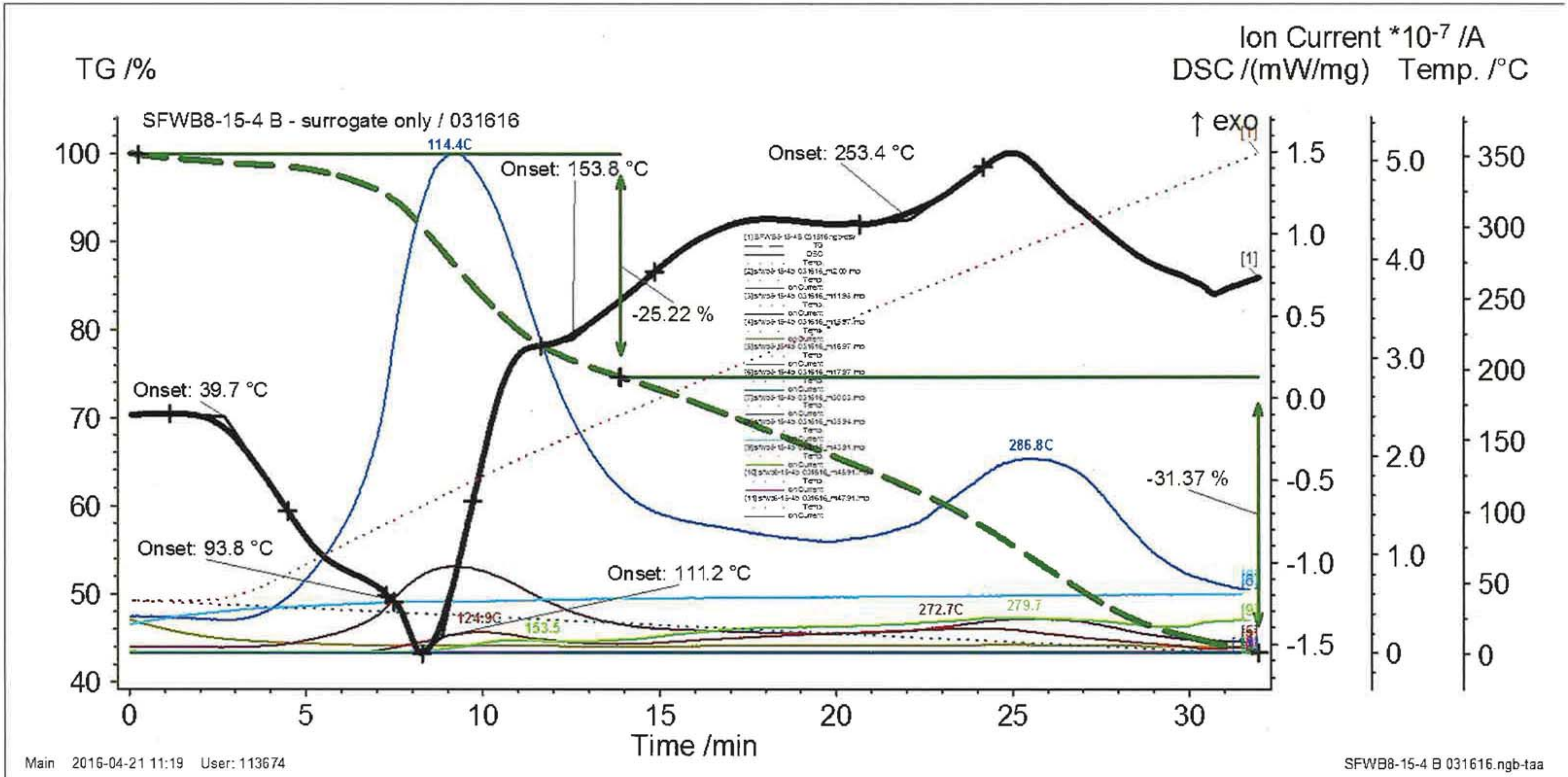
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4B 031516.ngb-dsv
Date/Time: 3/16/2016 3:10:16 PM (UTC-6)
End Date/Time: 3/16/2016 3:42:17 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-4B 031616
Sample name: SFWB8-15-4B 031616
Sample Mass: 12.37 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4B o31616

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	39.7 °C	1.1 min	6.1 min
Onset (DSC)	93.8 °C	7.3 min	7.9 min
Onset (DSC)	111.2 °C	8.3 min	11.6 min
Onset (DSC)	153.8 °C	11.6 min	18.1 min
Onset (DSC)	253.4 °C	20.7 min	25.9 min



[#] Instrument	File	Date	Identity	Sample	Mass...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-4B 031516.ngb-dsv	2016-03-16	SFWB8-15-4B 031616	SFWB8-15-4B 031616	12.37	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-4b 031616_m2.00.imp	2016-03-16	sfwb8-15-4b 031616	Mass 2.00		1/1	38°C/1.2(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4b 031616_m11.98.imp	2016-03-16	sfwb8-15-4b 031616	Mass 11.98		1/1	38°C/1.2(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4b 031616_m15.97.imp	2016-03-16	sfwb8-15-4b 031616	Mass 15.97		1/1	38°C/1.2(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4b 031616_m16.97.imp	2016-03-16	sfwb8-15-4b 031616	Mass 16.97		1/1	38°C/1.2(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4b 031616_m17.97.imp	2016-03-16	sfwb8-15-4b 031616	Mass 17.97		1/1	38°C/1.2(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4b 031616_m30.03.imp	2016-03-16	sfwb8-15-4b 031616	Mass 30.03		1/1	38°C/1.2(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4b 031616_m35.94.imp	2016-03-16	sfwb8-15-4b 031616	Mass 35.94		1/1	38°C/1.2(K/min)/1°C		---
[9] QMS 403	sfwb8-15-4b 031616_m43.91.imp	2016-03-16	sfwb8-15-4b 031616	Mass 43.91		1/1	38°C/1.2(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4b 031616_m45.91.imp	2016-03-16	sfwb8-15-4b 031616	Mass 45.91		1/1	38°C/1.2(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4b 031616_m47.91.imp	2016-03-16	sfwb8-15-4b 031616	Mass 47.91		1/1	38°C/1.2(K/min)/1°C		---

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MATL NAME: SFWB8-15-4B **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113174 Z No. 3-16-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WJPP TEMP 022916/WIPP 022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-16-16 Date
Temperature & Humidity Monitor	041888	10/25/2016	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8/3/2016	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-16-16 Date	
Date & Time Sample Vial Opened	Date: 3-16-16 *(mm / dd / yy)* Time: 15:01 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.5 C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-16-16 Date	DG. Initials 107962 Z No. 3/16/16 Date

MATL NAME: SFWB8-15-4 B **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-16-16 Time: 15:03	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-16-16 Date	
Crucible / Pan Tare Wt.	0.16795	grams	N/A	DMW Initials	DG. Initials
Net Sample Weight	DMW 113674 3-16-16 ~~0.00~~ 0.01237		~~>3 g, < 18 g~~ N/A	113674 Z No. 3-16-16 Date	107962 Z No. 3/16/16 Date

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-16-16 Date	DG. Initials 107962 Z No. 3/16/16 Date
Gas Pressure	Gas Pressure (at regulator): < 10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 14:49	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP 350C Baseline 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4 B **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-16-16 Date	DG. Initials 107962 Z No. 3/16/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-16-16 Time Started: 15:10 Total Analysis Time: 00:28	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 38.1	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-4B 031516.ngb-dsv **ThermoStar** Filename: SFWB8-15-4B 031516.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-16-16 Date	
Proteus Data	**Mass Changes:** 1) -25.22% Temp. Range (RT - 150°C) 2) -31.37% Temp. Range (150°C - 350°C) 3) ______ Temp. Range (______) 4) ______ Temp. Range (______) **Total mass Change:** -56.59%	Wt. % / °C	N/A ~~Total < 0.4~~ DMW 113674 3-16-16	DMW Initials 113674 Z No. 3-16-16 Date	DG. Initials 107962 Z No. 3/16/16 Date

Notes:

MATL NAME: SFWB8-15-4B **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (114.4, 286.8) 2) CO_2 Peak T (133.5 / 279.7) 3) NO/NO_2 Peak T (124.9 / 272.7) 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113174 Z No. 3-16-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % N/A DMW 113174 3-16-16 Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): ______ = 100 x ______ ÷ ______ Std. Error Slope	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113174 Z No. 3-16-16 Date
Supervisor – DS Review	N/A	N/A		J.A. Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A	HWM 149274 4/28/16	~~HHa~~ HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

1) Cal File is a user-calibration, no File No. issued
2) NOT a 3013 Run, various weight and moisture limits DO NOT APPLY
3) Total moisture measurement not required

DMW 117674 3-16-16

regarding #2 above!
- calibrated thermometer for chilled water - not required
- run not required to go to 1100°C
- Seal %RH and temp. measurement not required

DMW 117674 4-28-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-4C 031616.ngb-dsv	**Sample identity:**	SFWB8-15-4C
Date/Time:	3/16/2016 4:24:03 PM (UTC-6)	**Sample name:**	SFWB8-15-4C 031616
End Date/Time:	3/16/2016 4:56:05 PM (UTC-6)	**Sample Mass:**	13.79 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-4C

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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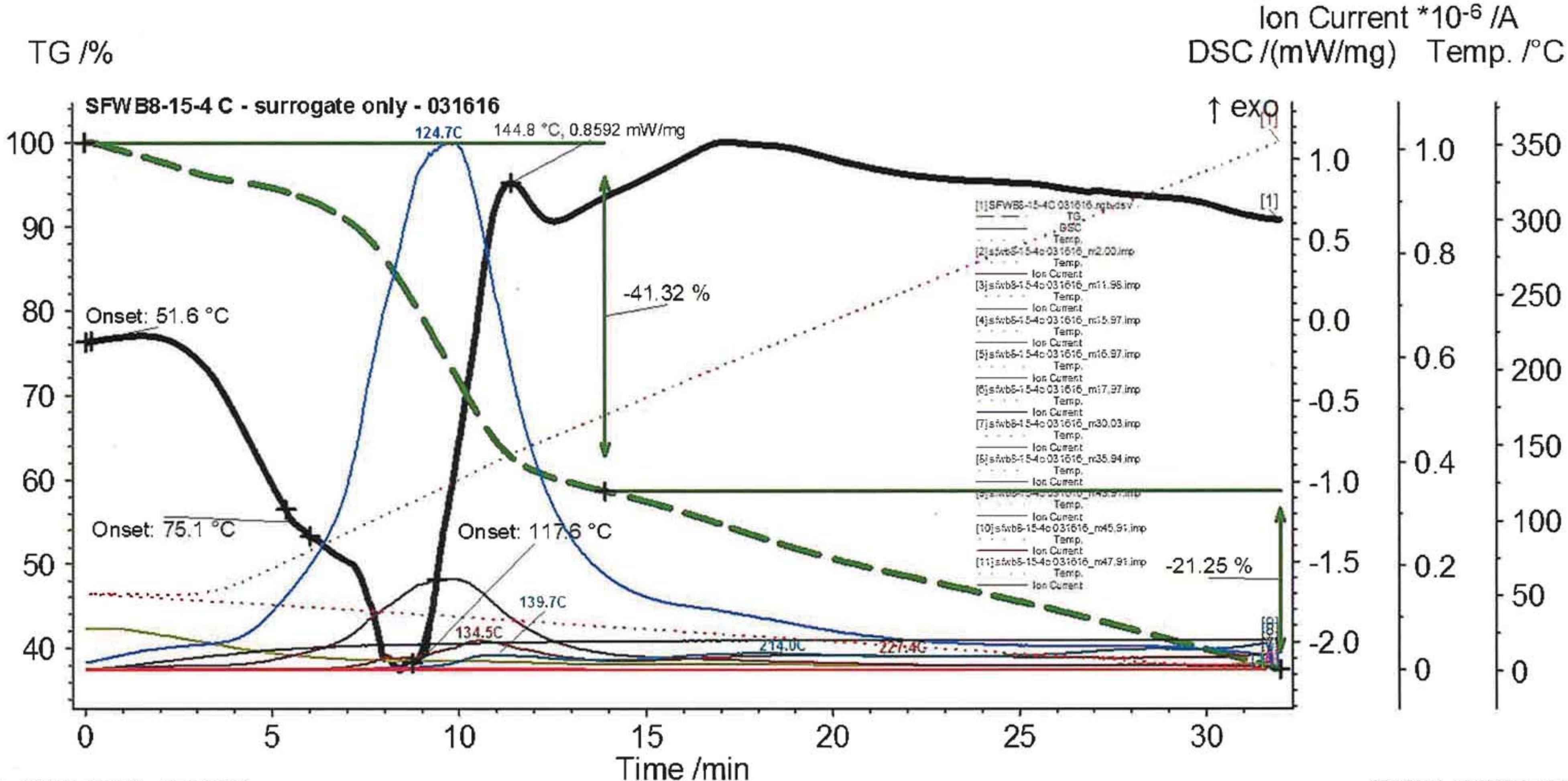
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4C 031616.ngb-dsv
Date/Time: 3/16/2016 4:24:03 PM (UTC-6)
End Date/Time: 3/16/2016 4:56:05 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-4C
Sample name: SFWB8-15-4C 031616
Sample Mass: 13.79 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4C

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	51.6 °C	0.0 min	5.8 min
Onset (DSC)	75.1 °C	5.4 min	7.5 min
Onset (DSC)	117.6 °C	8.8 min	9.6 min
Peak (DSC)	144.8 °C/0.8592 mW/mg	10.7 min	12.7 min



Main 2016-04-21 11:59 User: 113674

SFWB8-15-4 C 031616.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-4C 031616.ngb-dsv	2016-03-16	SFWB8-15-4C	SFWB8-15-4C 031616	13.79	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-4c 031616_m2.00.imp	2016-03-16	sfwb8-15-4c 031616	Mass 2.00		1/1	52°C/1.6(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4c 031616_m11.98.imp	2016-03-16	sfwb8-15-4c 031616	Mass 11.98		1/1	52°C/1.6(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4c 031616_m15.97.imp	2016-03-16	sfwb8-15-4c 031616	Mass 15.97		1/1	52°C/1.6(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4c 031616_m16.97.imp	2016-03-16	sfwb8-15-4c 031616	Mass 16.97		1/1	52°C/1.6(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4c 031616_m17.97.imp	2016-03-16	sfwb8-15-4c 031616	Mass 17.97		1/1	52°C/1.6(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4c 031616_m30.03.imp	2016-03-16	sfwb8-15-4c 031616	Mass 30.03		1/1	52°C/1.6(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4c 031616_m35.94.imp	2016-03-16	sfwb8-15-4c 031616	Mass 35.94		1/1	52°C/1.6(K/min)/1°C		---
[9] QMS 403	sfwb8-15-4c 031616_m43.91.imp	2016-03-16	sfwb8-15-4c 031616	Mass 43.91		1/1	52°C/1.6(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4c 031616_m45.91.imp	2016-03-16	sfwb8-15-4c 031616	Mass 45.91		1/1	52°C/1.6(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4c 031616_m47.91.imp	2016-03-16	sfwb8-15-4c 031616	Mass 47.91		1/1	52°C/1.6(K/min)/1°C		---

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MATL NAME: SFWB 8-15-4 C **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-16-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No (*circle one*)	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPP temp 022916 / WIPP 022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-16-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-16-16 Date
Date & Time Sample Vial Opened	Date: 03/16/16 (*mm / dd / yy*) Time: 16:01 (*24 hour*)	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.5 C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-16-16 Date DG. Initials 107962 Z No. 3/16/16 Date

MATL NAME: SFWB8-15-4C **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	**Date:** 3-16-16 **Time:** 16:06	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-16-16 Date	
Crucible / Pan Tare Wt.	0.16641	grams	N/A	DMW Initials 113674 Z No. 3-16-16 Date	DG Initials 107962 Z No. 3/16/16 Date
Net Sample Weight	0.01379		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-16-16 Date	DG Initials 107962 Z No. 3/16/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 14 : 49	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP 350C Baseline 030816 *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4-C **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Temperature Profile	Heating Rate: 10°c	C°/min	≥ 5, ≤ 20	DMM Initials 113674 Z No. 3-16-16 Date	DG. Initials 107962 Z No. 3/16/16 Date
	Maximum Temp.: 350°c (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-16-16 Time Started: 16:24 Total Analysis Time: 00:28	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 51.7	C°	N/A	N/A	
Run Data Files	**<u>All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"</u>** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-4C.031616.ngb-dsv **ThermoStar** Filename: SFWB8-15-4C.031616.MDC	N/A	N/A	DMM Initials 113674 Z No. 3-16-16 Date	
Proteus Data	**<u>Mass Changes:</u>** 1) -41.32% Temp. Range (RT - 155°C) 2) -21.25% Temp. Range (155°C - 350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -62.57%	Wt. % / °C	Total < 0.4	DMM Initials 113674 Z No. 3-16-16 Date	DG. Initials 107962 Z No. 3/16/16 Date

Notes:

UCNI

DMW 113674
3-16-16

MATL NAME: SFWB8-~~14~~ 15 -4 C **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (124.7°C) 2) CO_2 Peak T (139.7, 218.0°C) 3) NO/NO_2 Peak T (134.5, 227.4°C) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-16-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __ / __ / __ (mm dd yy) % Error (RSD, 1s): N/A = 100 x ____ ÷ ____ Std. Error Slope DMW 113674 3-16-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-16-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		Hwm QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

1) Cal File # for TGA-DSC calibration does not apply
- user calibrated equipment
2) N/A for thermometer - NOT REQUIRED for non-3013
3) Generic 3013 req. to go to 1100°c does not apply
4) Seal %RH / Temp - 3013 ONLY, does not apply
5) moisture determination does not apply for these measurements

DMW 113674 3-16-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4D 031716.ngb-dsv
Date/Time: 3/17/2016 10:50:32 AM (UTC-6)
End Date/Time: 3/17/2016 11:22:32 AM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-4D 031716
Sample name: SFWB8-15-4D 031716
Sample Mass: 16.47 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-4D 4TH RUN

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4D 031716.ngb-dsv
Date/Time: 3/17/2016 10:50:32 AM (UTC-6)
End Date/Time: 3/17/2016 11:22:32 AM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-4D 031716
Sample name: SFWB8-15-4D 031716
Sample Mass: 16.47 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4D 4TH RUN

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	117.7 °C	9.0 min	9.8 min
Onset (DSC)	38.4 °C	0.6 min	6.0 min
Onset (DSC)	94.3 °C	6.1 min	7.9 min
Onset (DSC)	104.2 °C	8.1 min	8.7 min
Onset (DSC)	69.2 °C	3.8 min	6.4 min
End (DSC)	110.9 °C	8.3 min	8.8 min
Area (DSC),o	2.45 J/g	8.1 min	8.8 min
Peak (DSC)	141.7 °C/0.7788 mW/mg	9.4 min	12.3 min

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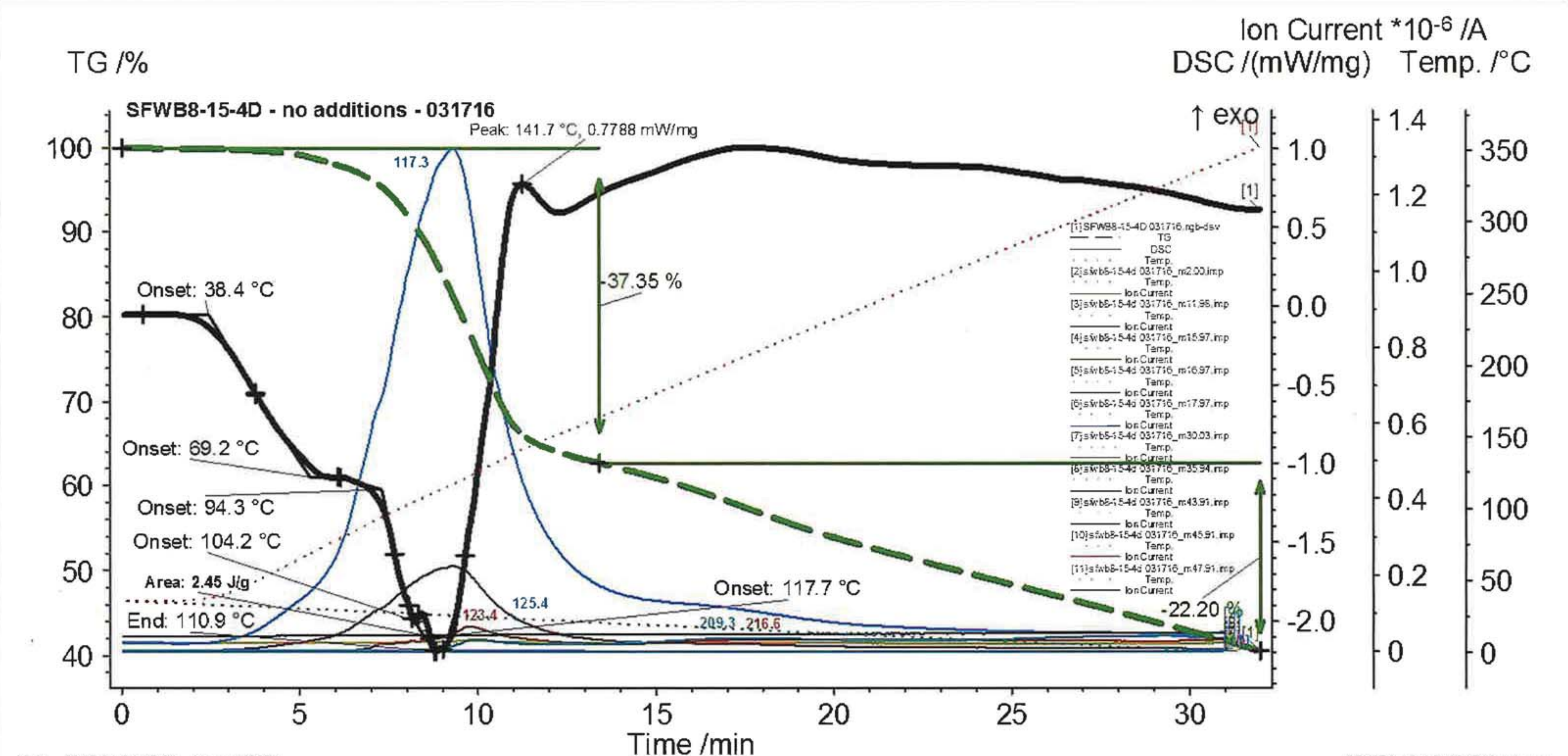

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SFWB8-15-4 D 031716.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass...	Seg...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-4D 031716.ngb-dsv	2016-03-17	SFWB8-15-4D 031716	SFWB8-15-4D 031716	16.47	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/---	DSC:020, TG:020
[2] QMS 403	sfwb8-15-4d 031716_m2.00.imp	2016-03-17	sfwb8-15-4d 031716	Mass 2.00		1/1	37°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4d 031716_m11.98.imp	2016-03-17	sfwb8-15-4d 031716	Mass 11.98		1/1	37°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4d 031716_m15.97.imp	2016-03-17	sfwb8-15-4d 031716	Mass 15.97		1/1	37°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4d 031716_m16.97.imp	2016-03-17	sfwb8-15-4d 031716	Mass 16.97		1/1	37°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4d 031716_m17.97.imp	2016-03-17	sfwb8-15-4d 031716	Mass 17.97		1/1	37°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4d 031716_m30.03.imp	2016-03-17	sfwb8-15-4d 031716	Mass 30.03		1/1	37°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4d 031716_m35.94.imp	2016-03-17	sfwb8-15-4d 031716	Mass 35.94		1/1	37°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-4d 031716_m43.91.imp	2016-03-17	sfwb8-15-4d 031716	Mass 43.91		1/1	37°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4d 031716_m45.91.imp	2016-03-17	sfwb8-15-4d 031716	Mass 45.91		1/1	37°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4d 031716_m47.91.imp	2016-03-17	sfwb8-15-4d 031716	Mass 47.91		1/1	37°C/1.1(K/min)/1°C		---

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MATL NAME: SFWB8-15-4 D **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113174 Z No. 3-17-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916 / WIPP022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-17-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113174 Z No. 3-17-16 Date	
Date & Time Sample Vial Opened	Date: 03/17/16 *(mm / dd / yy)* Time: 10:34 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.8°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113174 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date

MATL NAME: SFW138 -15-4D **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	03-17-16 Date: ~~10~~ DMW 113674 3/17/16 Time: 10:35 AM	Mm/dd/yy – 24 hr.	N/A	Dmw Initials 113674 Z No. 3-17-16 Date	
Crucible / Pan Tare Wt.	0.16486	grams	N/A	DMW Initials	DG. Initials
Net Sample Weight	0.01647		>3 g, < 18 g	113674 Z No. 3-17-16 Date	107962 Z No. 3/17/16 Date

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 02	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350CBaseline.030816 *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4 D **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-17-16 Time Started: 10:53 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.4	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-4D.031716.ngb-dsv **ThermoStar** Filename: SFWB8-15-4D.031716.MDC	N/A	N/A	DMW Initials 113674 Z No. 3-17-16 Date	
Proteus Data	**Mass Changes:** 1) -37.35% Temp. Range (RT-150°C) 2) -22.20% Temp. Range (150°-350°C) 3) ____ Temp. Range (____) 4) ____ Temp. Range (____) **Total mass Change:** -59.55%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date

Notes:

MATL NAME: SFWB8-15-4 D **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (117.3°C) 2) CO_2 Peak T (125.4°, 209.3°C) 3) NO/NO_2 Peak T (123.4°, 216.6°C) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-17-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __ / __ / __ mm dd yy %-Error (RSD, 1s): N/A DMW 113674 3-17-16 ____ = 100 x ____ ÷ ____ Std. Error Slope	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-17-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 09501? Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A summary

1) user calibrated DSC-TGA - no calib. file
2) calib. thermometer unnecessary for non 3013 work
3) NON 3013 analysis does not have to go to 1100°C
not required!
4) Seal %RH/T - not required for non-3013 work
5) moisture determination not required for WIPP study

DMW 113674 3-17-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-ACID A 031716.ngb-dsv	**Sample identity:**	SFWB8-15-ACID A 031716
Date/Time:	3/17/2016 2:45:50 PM (UTC-6)	**Sample name:**	SFWB8-15-ACID A 031716
End Date/Time:	3/17/2016 3:17:50 PM (UTC-6)	**Sample Mass:**	17.204 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-4 ACID A 031716

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-ACID A 031716.ngb-dsv
Date/Time:	3/17/2016 2:45:50 PM (UTC-6)
End Date/Time:	3/17/2016 3:17:50 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-ACID A 031716
Sample name:	SFWB8-15-ACID A 031716
Sample Mass:	17.204 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4 ACID A 031716

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	39.0 °C	1.1 min	5.5 min
Onset (DSC)	120.0 °C	9.3 min	10.1 min
Onset (DSC)	151.1 °C	11.8 min	26.9 min
Onset (DSC)	65.6 °C	3.8 min	5.6 min
Onset (DSC)	108.6 °C	8.5 min	9.2 min
Area (DSC),o	2.717 J/g	8.6 min	9.3 min
Peak (DSC)	143.6 °C/0.6526 mW/mg	9.3 min	12.1 min

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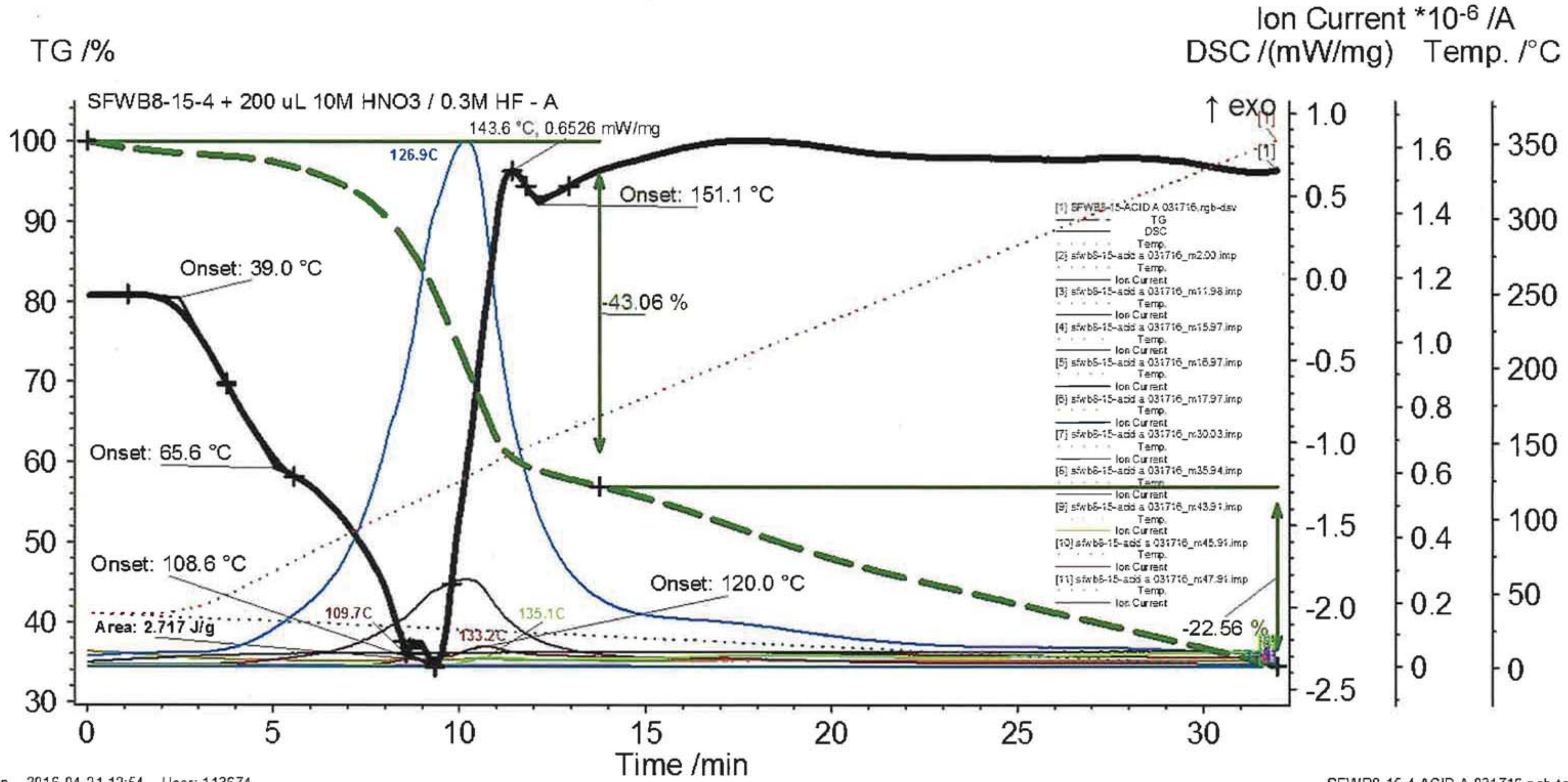

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SFWB8-15-4 ACID A 031716.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mas...	S..	Range	Atmosphere	Corr.
[1] STA 409PC/...	SFWB8-15-ACID A 031716.ngb-...	2016-03-...	SFWB8-15-ACID A 031...	SFWB8-15-ACID A 031...	17.2...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[2] QMS 403	sfwb8-15-acid a 031716_m2.00.i...	2016-03-...	sfwb8-15-acid a 031716	Mass 2.00		1...	37°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-acid a 031716_m11.98....	2016-03-...	sfwb8-15-acid a 031716	Mass 11.98		1...	37°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-acid a 031716_m15.97....	2016-03-...	sfwb8-15-acid a 031716	Mass 15.97		1...	37°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-acid a 031716_m16.97....	2016-03-...	sfwb8-15-acid a 031716	Mass 16.97		1...	37°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-acid a 031716_m17.97....	2016-03-...	sfwb8-15-acid a 031716	Mass 17.97		1...	37°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-acid a 031716_m30.03....	2016-03-...	sfwb8-15-acid a 031716	Mass 30.03		1...	37°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-acid a 031716_m35.94....	2016-03-...	sfwb8-15-acid a 031716	Mass 35.94		1...	37°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-acid a 031716_m43.91	2016-03-	sfwb8-15-acid a 031716	Mass 43.91		1...	37°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-acid a 031716_m45.91....	2016-03-...	sfwb8-15-acid a 031716	Mass 45.91		1...	37°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-acid a 031716_m47.91....	2016-03-...	sfwb8-15-acid a 031716	Mass 47.91		1...	37°C/1.1(K/min)/1°C		---

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MATL NAME: SFWB8-15 ACID A 031716 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-17-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTEMP022916 / WIPP022916	N/A	02/28/17	DMW Initials 113674 Z No. 3-17-16 Date
Temperature & Humidity Monitor	041888	10/25/16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08/03/2016	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-17-16 Date	
Date & Time Sample Vial Opened	Date: 03/17/16 *(mm / dd / yy)* Time: 14:36 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.8	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-17-16 Date	DG Initials 107962 Z No. 3/17/16 Date

MATL NAME: 8FWB8-15-4 ACID A **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-17-16 Time: 14:40	Mm/dd/yy – 24 hr.	N/A	DMM Initials 113674 Z No. 3-17-16 Date	
Crucible / Pan Tare Wt.	0.16956	grams	N/A	DMM Initials 113674 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date
Net Sample Weight	0.01918		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMM Initials 113674 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10:02	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WTPP 350C Baseline 030816 *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4 ACID A **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-17-16 Time Started: 14:46 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 37.3	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-ACID A 031716.ngb-dsv **ThermoStar** Filename: SFWB8-15-ACIDA 031716.Mdc	N/A	N/A	DMW Initials 113674 Z No. 3-17-16 Date	
Proteus Data	**Mass Changes:** 1) -13.06 Temp. Range (RT-150°C) 2) -22.56 Temp. Range (150°C-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -65.62%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-17-16 Date	DG. Initials 107962 Z No. 3/17/16 Date

Notes:

MATL NAME: SFWB8-15-4 ACID A **Location:** G220

Gas Data (Section 5.6)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (126.9°C) 2) CO_2 Peak T (135.1°C) 3) NO/NO_2 Peak T (109.7°/133.29°C) 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-17-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: __/__/__ (mm dd yy) % Error (RSD, 1s): N/A = 100 x ______ ÷ ______ Std. Error Slope	DMW 113674 Wt. % 3-17-16	< 0.32	______ Initials ______ Z No. ______ Date	______ Initials ______ Z No. ______ Date

Signatures

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-17-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A review:

1) cal file name for TGA+DSC Calib is N/A because it is a user calibrated system
2) calibrated thermometer is not required for non-3013 work
3) Temp. Profile to 350°C for this work
4) Seal %RH/Temp not required for non-3013
5) moisture analysis not required for WIPP surrogates

DMW 113674 3-17-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-ACID B 031816.ngb-dsv
Date/Time: 3/18/2016 10:05:43 AM (UTC-6)
End Date/Time: 3/18/2016 10:37:44 AM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-ACID B 031816
Sample name: SFWB8-15-ACID B 031816
Sample Mass: 16.397 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: surrogate + acid 2nd run - gray bead component

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

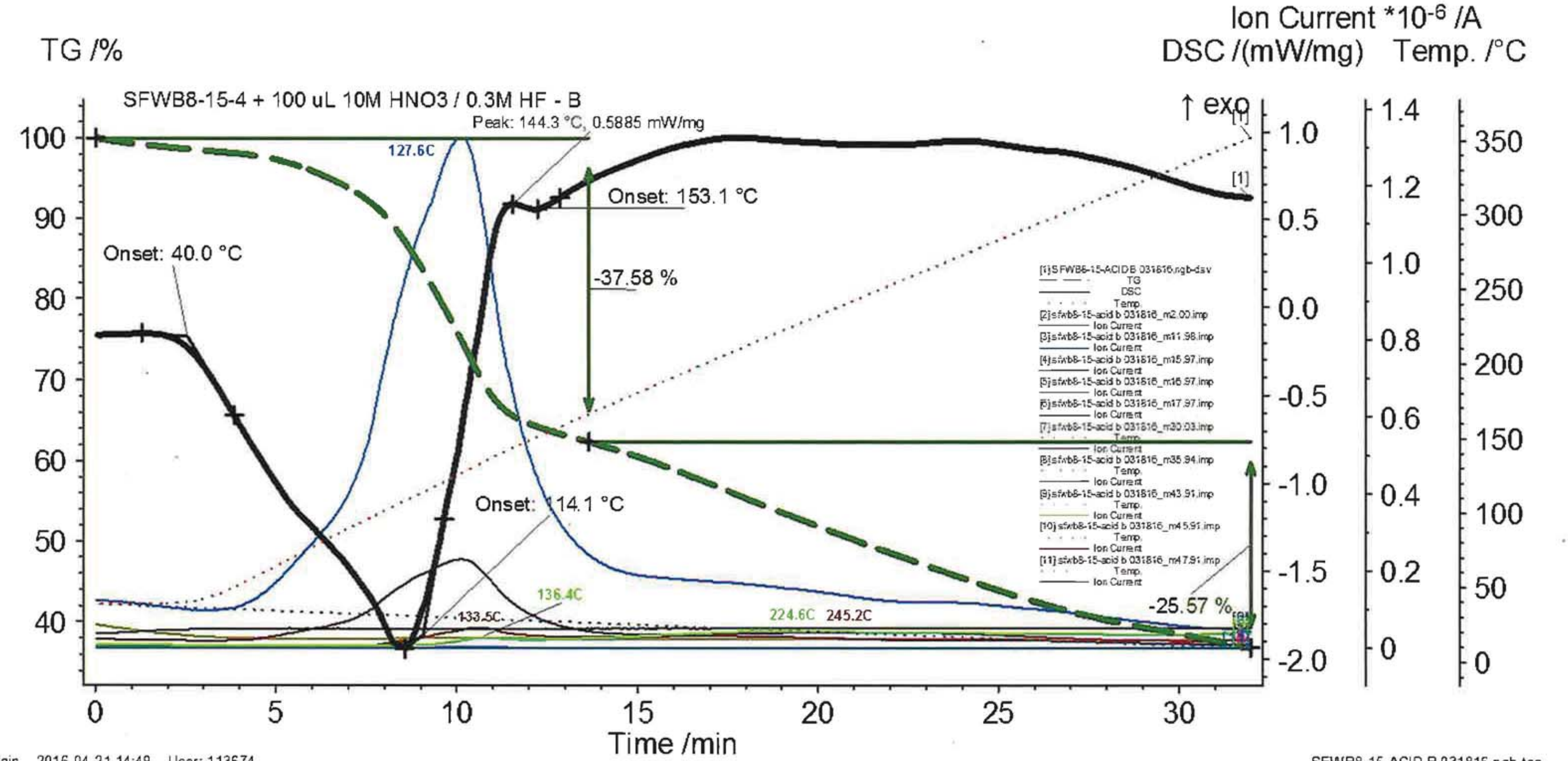
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-ACID B 031816.ngb-dsv
Date/Time: 3/18/2016 10:05:43 AM (UTC-6)
End Date/Time: 3/18/2016 10:37:44 AM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-ACID B 031816
Sample name: SFWB8-15-ACID B 031816
Sample Mass: 16.397 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: surrogate + acid 2nd run - gray bead component

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	40.0 °C	1.3 min	5.8 min
Onset (DSC)	114.1 °C	8.6 min	9.9 min
Onset (DSC)	153.1 °C	12.2 min	18.5 min
Peak (DSC)	144.3 °C/0.5885 mW/mg	10.7 min	12.2 min



Main 2016-04-21 14:49 User: 113674

SFWB8-15-ACID B 031816.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mas...	S..	Range	Atmosphere	Corr.
[1] STA 409PC/...	SFWB8-15-ACID B 031816.ngb-...	2016-03-...	SFWB8-15-ACID B 031...	SFWB8-15-ACID B 031...	16.3...	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[2] QMS 403	sfwb8-15-acid b 031816_m2.00.i...	2016-03-...	sfwb8-15-acid b 031816	Mass 2.00		1...	39°C/0.9(K/min)/1°C		---
[3] QMS 403	sfwb8-15-acid b 031816_m11.98....	2016-03-...	sfwb8-15-acid b 031816	Mass 11.98		1...	39°C/0.9(K/min)/1°C		---
[4] QMS 403	sfwb8-15-acid b 031816_m15.97....	2016-03-...	sfwb8-15-acid b 031816	Mass 15.97		1...	39°C/0.9(K/min)/1°C		---
[5] QMS 403	sfwb8-15-acid b 031816_m16.97....	2016-03-...	sfwb8-15-acid b 031816	Mass 16.97		1...	39°C/0.9(K/min)/1°C		---
[6] QMS 403	sfwb8-15-acid b 031816_m17.97....	2016-03-...	sfwb8-15-acid b 031816	Mass 17.97		1...	39°C/0.9(K/min)/1°C		---
[7] QMS 403	sfwb8-15-acid b 031816_m30.03....	2016-03-...	sfwb8-15-acid b 031816	Mass 30.03		1...	39°C/0.9(K/min)/1°C		---
[8] QMS 403	sfwb8-15-acid b 031816_m35.94....	2016-03-...	sfwb8-15-acid b 031816	Mass 35.94		1...	39°C/0.9(K/min)/1°C		---
[9] QMS 403	sfwb8-15-acid b 031816_m43.91....	2016-03-...	sfwb8-15-acid b 031816	Mass 43.91		1...	39°C/0.9(K/min)/1°C		---
[10] QMS 403	sfwb8-15-acid b 031816_m45.91....	2016-03-...	sfwb8-15-acid b 031816	Mass 45.91		1...	39°C/0.9(K/min)/1°C		---
[11] QMS 403	sfwb8-15-acid b 031816_m47.91....	2016-03-...	sfwb8-15-acid b 031816	Mass 47.91		1...	39°C/0.9(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-4 ACID B **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-18-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPP Temp 022916/ WIPP 022916	N/A	02/28/17	DMW Initials
Temperature & Humidity Monitor	041888	10-25-16	113674 Z No.
Calibrated Thermometer (for Water Chiller)	N/A	N/A	3-18-16 Date
Wall Clock	040480	08-03-16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 03.18.16 Date
Date & Time Sample Vial Opened	Date: 03/18/16 *(mm / dd / yy)* Time: 9:58 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/35.0%	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-18-16 Date DG. Initials 107962 Z No. 3/18/16 Date

MATL NAME: SFWB8-18-4 ACID B **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 3-18-16 Time: 10:00	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3-18-16 Date	
Crucible / Pan Tare Wt.	0.16886	grams	N/A	DMW Initials 113674 Z No. 3-18-16 Date	DG. Initials 107962 Z No. 3/18/16 Date
Net Sample Weight	0.01726		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-18-16 Date	DG. Initials 107962 Z No. 3/18/16 Date
Gas Pressure	Gas Pressure (at regulator): < 10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 9:47	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350C Baseline 030816 *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4 ACID B **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**	**Verified By**
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	Dmw Initials	DG. Initials
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 03-18-16 Time Started: 10:06 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A	113674 Z No. 3-18-16 Date	107962 Z No. 3/18/16 Date
ThermoStar Reference	Sample Temp. at Start: 38.8	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-ACIDB031816.ngb-dsv **ThermoStar** Filename: SFWB8-15-ACIDB031816.moc	N/A	N/A	Dmw Initials 113674 Z No. 3-18-16 Date	
Proteus Data	**Mass Changes:** 1) -37.58% Temp. Range (RT-166°C) 2) -25.57% Temp. Range (166°C-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -63.15%	Wt. % / °C	Total < 0.4	Dmw Initials 113674 Z No. 3-18-16 Date	DG. Initials 107962 Z No. 3/18/16 Date

Notes:

MATL NAME: SFWB8-15-4 ACID B **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (127.6°C) 2) CO_2 Peak T (136.4° & 224.6°C) 3) NO/NO_2 Peak T (133.5° & 245.2°C) 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-18-16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: N/A __/__/__ (mm dd yy) % Error (RSD, 1s): ____ = 100 x ____ ÷ ____ Std. Error Slope DMW 113674 3-18-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-18-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary

1) Cal File # for user performed Calibration not required
2) Calib. Thermom. not required for non-3013 work
3) Temp. Profile not required to go to 1100°C for non-3013
4) Seal %RH & Temp. not required for non-3013 work
5) moisture analysis not required for WIPP

DMM 113674 3-18-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-ACID C 031816.ngb-dsv
Date/Time: 3/18/2016 11:42:07 AM (UTC-6)
End Date/Time: 3/18/2016 12:14:09 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-ACID C 031816
Sample name: SFWB8-15-ACID C 031816
Sample Mass: 19.33 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-ACID C 031816

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

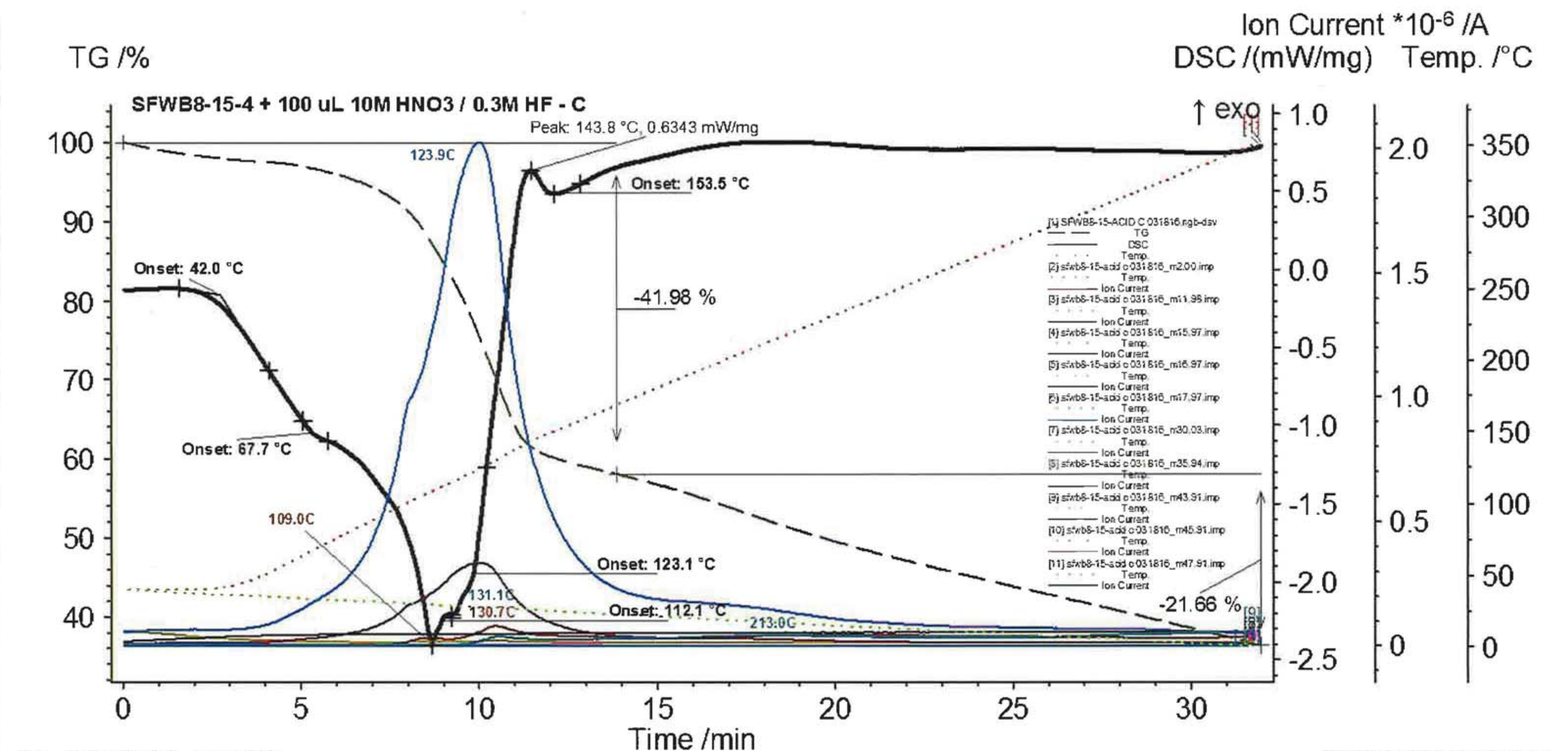
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-ACID C 031816.ngb-dsv
Date/Time: 3/18/2016 11:42:07 AM (UTC-6)
End Date/Time: 3/18/2016 12:14:09 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-ACID C 031816
Sample name: SFWB8-15-ACID C 031816
Sample Mass: 19.33 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-ACID C 031816

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	42.0 °C	1.6 min	5.7 min
Onset (DSC)	67.7 °C	5.1 min	8.5 min
Onset (DSC)	112.1 °C	8.7 min	10.2 min
Onset (DSC)	123.1 °C	9.2 min	10.5 min
Onset (DSC)	153.5 °C	12.1 min	14.6 min
Peak (DSC)	143.8 °C/0.6343 mW/mg	10.3 min	12.1 min



Main 2016-04-21 15:13 User: 113674

SFWB8-15-ACID C 031816.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Ma...	S...	Range	Atmosphere	Corr.
[1] STA 409PC/...	SFWB8-15-ACID C 031816.ngb-...	2016-03-...	SFWB8-15-ACID C 031...	SFWB8-15-ACID C 031...	19....	1...	30°C/10.0(K/min)/35...	ARGON/50 / ARGON/30 / <no gas...	DSC:020, TG:...
[2] QMS 403	sfwb8-15-acid c 031816_m2.00.imp	2016-03-...	sfwb8-15-acid c 031816	Mass 2.00		1...	41°C/1.3(K/min)/1°C		---
[3] QMS 403	sfwb8-15-acid c 031816_m11.98.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 11.98		1...	41°C/1.3(K/min)/1°C		---
[4] QMS 403	sfwb8-15-acid c 031816_m15.97.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 15.97		1...	41°C/1.3(K/min)/1°C		---
[5] QMS 403	sfwb8-15-acid c 031816_m16.97.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 16.97		1...	41°C/1.3(K/min)/1°C		---
[6] QMS 403	sfwb8-15-acid c 031816_m17.97.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 17.97		1...	41°C/1.3(K/min)/1°C		---
[7] QMS 403	sfwb8-15-acid c 031816_m30.03.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 30.03		1...	41°C/1.3(K/min)/1°C		---
[8] QMS 403	sfwb8-15-acid c 031816_m35.94.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 35.94		1...	41°C/1.3(K/min)/1°C		---
[9] QMS 403	sfwb8-15-acid c 031816_m43.91.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 43.91		1...	41°C/1.3(K/min)/1°C		---
[10] QMS 403	sfwb8-15-acid c 031816_m45.91.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 45.91		1...	41°C/1.3(K/min)/1°C		---
[11] QMS 403	sfwb8-15-acid c 031816_m47.91.i...	2016-03-...	sfwb8-15-acid c 031816	Mass 47.91		1...	41°C/1.3(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15 ACID C **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	**Data**	**Units**	**Acceptance Criteria**	**Performed By**
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3·18·16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**
Carrier Type ***(circle one)*** **: TG / [✓ DSC]** **Cal. File Name:** WIPP temp 022916 / WIPP 022916	N/A	02/28/16	DMW Initials 113674 Z No. 3-18-16 Date
Temperature & Humidity Monitor	041888	10/25/16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08/03/16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	**Cal. File No.**	**Cal. Exp. Date**	**Performed By**	**Description**	
LANMAS / LAMCAS Balance ID	B3CC	N/A	N/A	DMW Initials 113674 Z No. 3-18-16 Date	
Date & Time Sample Vial Opened	Date: 03/18/16 *(mm / dd / yy)* Time: 11:36 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/32.2	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-18-16 Date	DG. Initials 107962 Z No. 3/18/16 Date

MATL NAME: SFWB8-4-15 ACID C **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 03-18-16 Time: 11:38	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 3/18/16 Date	
Crucible / Pan Tare Wt.	0.16747	grams	N/A	DMW Initials 113674 Z No. 3-18-16 Date	DG Initials 107962 Z No. 3/18/16 Date
Net Sample Weight	0.01933		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 3-18-16 Date	DG Initials 107962 Z No. 3/18/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 9 : 47	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	WIPP350C BASELINE030816.ngb-bsv Filename: *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4 ACIDC **Location:** G270

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-18-16 Date	DG. Initials 107962 Z No. 3/18/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-18-16 Time Started: 11:42 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 40.8	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP DMW 113674 3-18-16 SFWB8-15-4 ACIDC 030816.ngb-dsv **Netzsch Measure** Filename: SFWB8-15-4 ACIDC 031816, MDC **ThermoStar** Filename:	N/A	N/A	DMW Initials 113674 Z No. 3-18-16 Date	
Proteus Data	**Mass Changes:** 1) -41.98 Temp. Range (RT - 168.7°C) 2) -21.66 Temp. Range (168.7-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -63.64%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-18-16 Date	DG. Initials 107962 Z No. 3/18/16 Date

Notes:

DMW 113674 3-18-16

UCNI

MATL NAME: ~~SFWB8-15-4~~ SFWB8-15-4 ACID C **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (123.9°C) 2) CO_2 Peak T (131.1° / 213.0°C) 3) NO/NO_2 Peak T (109.0°C/130.7°C) 4) _____ Peak T (_____) 5) _____ Peak T (_____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3·18·16 Date	
Total Moisture (H_2O) N/A	Total Moisture = _____ mg _____ Wt % Date of most recent calibration: __/__/__ mm dd yy % Error (RSD, 1s): _____ = 100 x _____ ÷ _____ Std. Error Slope DMW 113674 3-18-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3·18·16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 094012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary

1) no cal file # for user calibrated TGA/DSC
2) Thermometer for water chiller not required for WIPP
3) Temp Profile for WIPP does not have to go to 1100°C
4) Seal %RH/Temp not required for WIPP
5) moisture analysis not required for WIPP

DMW 113674 3-18-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4 200ul FeNO.ngb-dsv
Date/Time: 4/15/2016 9:52:29 AM (UTC-6)
End Date/Time: 4/15/2016 10:24:29 AM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-4 200ul FeNO
Sample name: SFWB8-15-4 200ul FeNO
Sample Mass: 16.768 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-4 200ul FeNO - 200 uL of ~3M Fe(NO3)-9H2O solution in 10M HNO3 / 0.3M HF

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

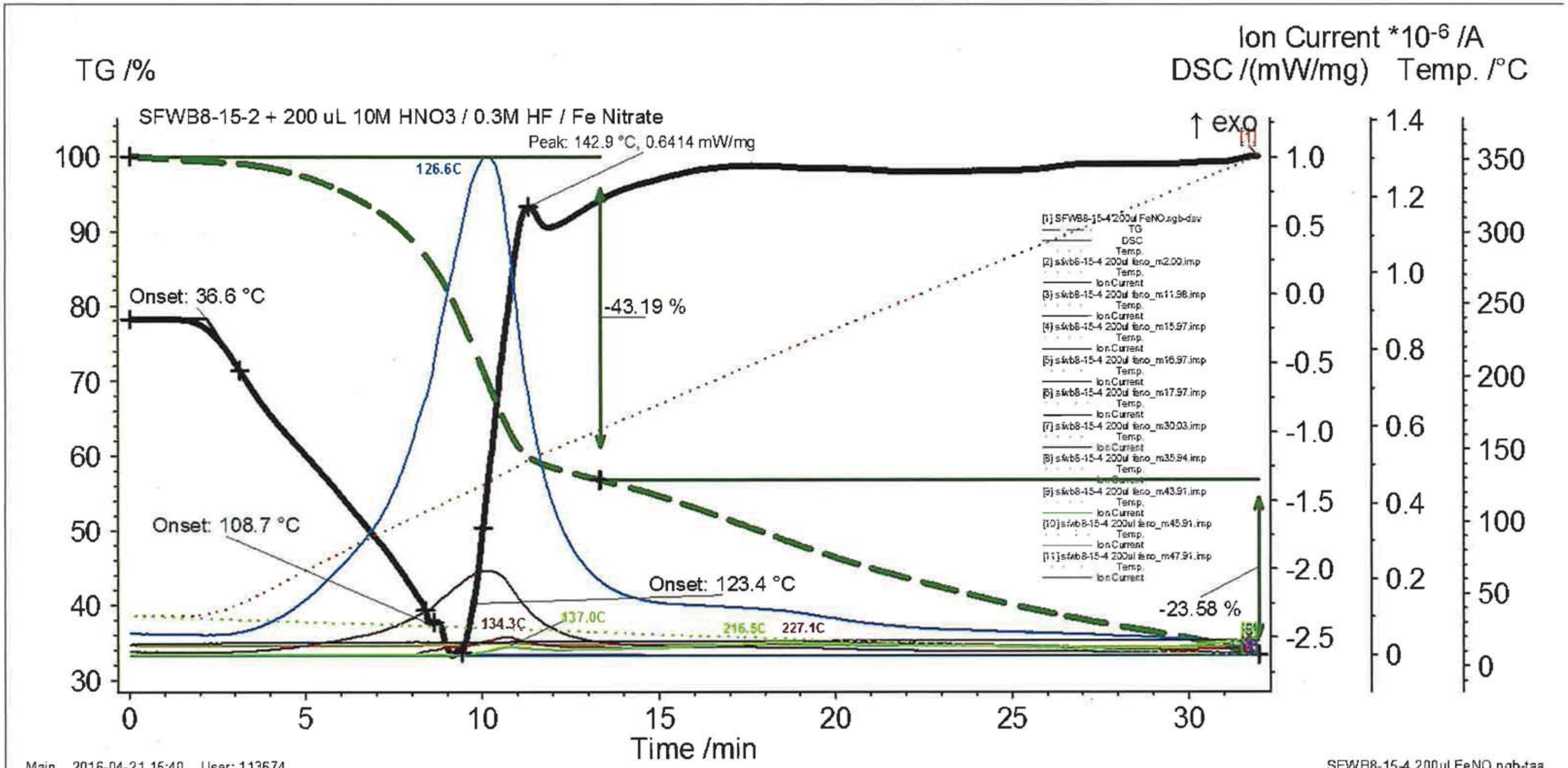
Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

Instrument:	NETZSCH STA 409PC/PG	**TG TG Range:**	30000 mg
Project:	WIPP	**Sample identity:**	SFWB8-15-4 200ul FeNO
Filename:	SFWB8-15-4 200ul FeNO.ngb-dsv	**Sample name:**	SFWB8-15-4 200ul FeNO
Date/Time:	4/15/2016 9:52:29 AM (UTC-6)	**Sample Mass:**	16.768 mg
End Date/Time:	4/15/2016 10:24:29 AM (UTC-6)	**Crucible:**	DSC/TG pan Pt-Rh
Laboratory:	55-0004-0208	**Crucible Mass:**	0 mg
Operator:	DMW	**Reference name:**	empty
Mode:	DSC-TG	**Reference Mass:**	0 mg
Measurement Type:	sample with correction	**Reference Crucible Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Material:**	WIPP surrogate
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Sample determination mode:**	Manual
Sensitivity:	WIPP 022916.ngb-esv	**Residuum measurement:**	Not possible
Crucible:	DSC/TG pan Pt-Rh	**Atmosphere:**	ARGON/50 / ARGON/30 / <no gas>/---
DSC DSC Range:	5000 µV		

Remark: SFWB8-15-4 200ul FeNO - 200 uL of ~3M Fe(NO3)-9H2O solution in 10M HNO3 / 0.3M HF

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	108.7 °C	8.4 min	8.9 min
Onset (DSC)	36.6 °C	0.0 min	6.9 min
Onset (DSC)	123.4 °C	9.4 min	10.7 min
Peak (DSC)	142.9 °C/0.6414 mW/mg	9.3 min	11.9 min



Main 2016-04-21 15:40 User: 113674

SFWB8-15-4 200ul FeNO.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mas...	S...	Range	Atmosphere	Corr.
[1] STA 409PC/...	SFWB8-15-4 200ul FeNO.ngb-dsv	2016-04-...	SFWB8-15-4 200ul Fe...	SFWB8-15-4 200ul Fe...	16.7...	1/1	30°C/10.0(K/min)/350...	ARGON/50 / ARGON/30 / <no gas>...	DSC:020, TG:...
[2] QMS 403	sfwb8-15-4 200ul feno_m2.00.imp	2016-04-...	sfwb8-15-4 200ul feno	Mass 2.00		1/1	35°C/0.8(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4 200ul feno_m11.98.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 11.98		1/1	35°C/0.8(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4 200ul feno_m15.97.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 15.97		1/1	35°C/0.8(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4 200ul feno_m16.97.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 16.97		1/1	35°C/0.8(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4 200ul feno_m17.97.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 17.97		1/1	35°C/0.8(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4 200ul feno_m30.03.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 30.03		1/1	35°C/0.8(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4 200ul feno_m35.94.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 35.94		1/1	35°C/0.8(K/min)/1°C		---
[9] QMS 403	sfwb8-15-4 200ul feno_m43.91.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 43.91		1/1	35°C/0.8(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4 200ul feno_m45.91.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 45.91		1/1	35°C/0.8(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4 200ul feno_m47.91.i...	2016-04-...	sfwb8-15-4 200ul feno	Mass 47.91		1/1	35°C/0.8(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-4+700uL F+NO **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-15-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTemp 022916/WIPP 022916	N/A	02/28/16	DMW Initials
Temperature & Humidity Monitor	041888	10/25/16	113674 Z No.
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08/03/16	4-15-16 Date

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-15-16 Date	
Date & Time Sample Vial Opened	Date: 04/15/16 *(mm / dd / yy)* Time: 9:43 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/29.0	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-15-16 Date	DG. Initials 107962 Z No. 4/15/16 Date

MATL NAME: SFWB8-15-4 ~~FeNO~~ 200uL FeNO DMW 113674 4-15-16 **Location:** ~~G220~~ G220 DMW 113674 4-15-16

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 4-15-16 Time: 9:45	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-15-16 Date	
Crucible / Pan Tare Wt.	0.16482	grams	N/A	DMW Initials 113674 Z No. 4-15-16 Date	DG. Initials 107962 Z No. 4/15/16 Date
Net Sample Weight	0.01799		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-15-16 Date	DG. Initials 107962 Z No. 4/15/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 9:40	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP 350C Baseline 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4+200uL FeNO **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMM Initials 113674 Z No. 4-15-16 Date	DG. Initials 107962 Z No. 4/15/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 4-15-16 Time Started: 9:54 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 34.9	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-4 200ul FeNO.ngb-dsv **ThermoStar** Filename: SFWB8-15-4 200ul FeNO.MDC	N/A	N/A	DMM Initials 113674 Z No. 4-15-16 Date	
Proteus Data	**Mass Changes:** 1) -43.19 Temp. Range (RT-163.9°C) 2) -23.58 Temp. Range (163.9-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -66.77%	Wt. % / °C	Total < 0.4	DMM Initials 113674 Z No. 4-15-16 Date	DG. Initials 107962 Z No. 4/15/16 Date

Notes:

MATL NAME: SFWB8-15-4 200MG FENO **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (126.6°C) 2) CO_2 Peak T (137.0/216.5°C) 3) NO/NO_2 Peak T (134.3/227.1°C) 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-15-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: ______ mm dd yy % Error (RSD, 1s): ______ = 100 x ______ ÷ ______ Std. Error Slope N/A DMW 113674 4-15-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-15-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		HWM QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A Summary

1) Cal File number not required for User Calibrated Systems. This is a User calibrated instrument.
2) Thermometer for water chiller not required for non-3013
3) Profile to 1100°C only req. for 3013
4) Seal %RH / Temp meas. not req. for WIPP
5) moisture quantification not required for WIPP

113674 4-15-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-4 PUAM1.ngb-dsv	**Sample identity:**	SFWB8-15-4 PUAM1
Date/Time:	3/30/2016 1:35:17 PM (UTC-6)	**Sample name:**	SFWB8-15-4 PUAM1
End Date/Time:	3/30/2016 2:07:18 PM (UTC-6)	**Sample Mass:**	20.30 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-4 PUAM1 surrogate + spike 1

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

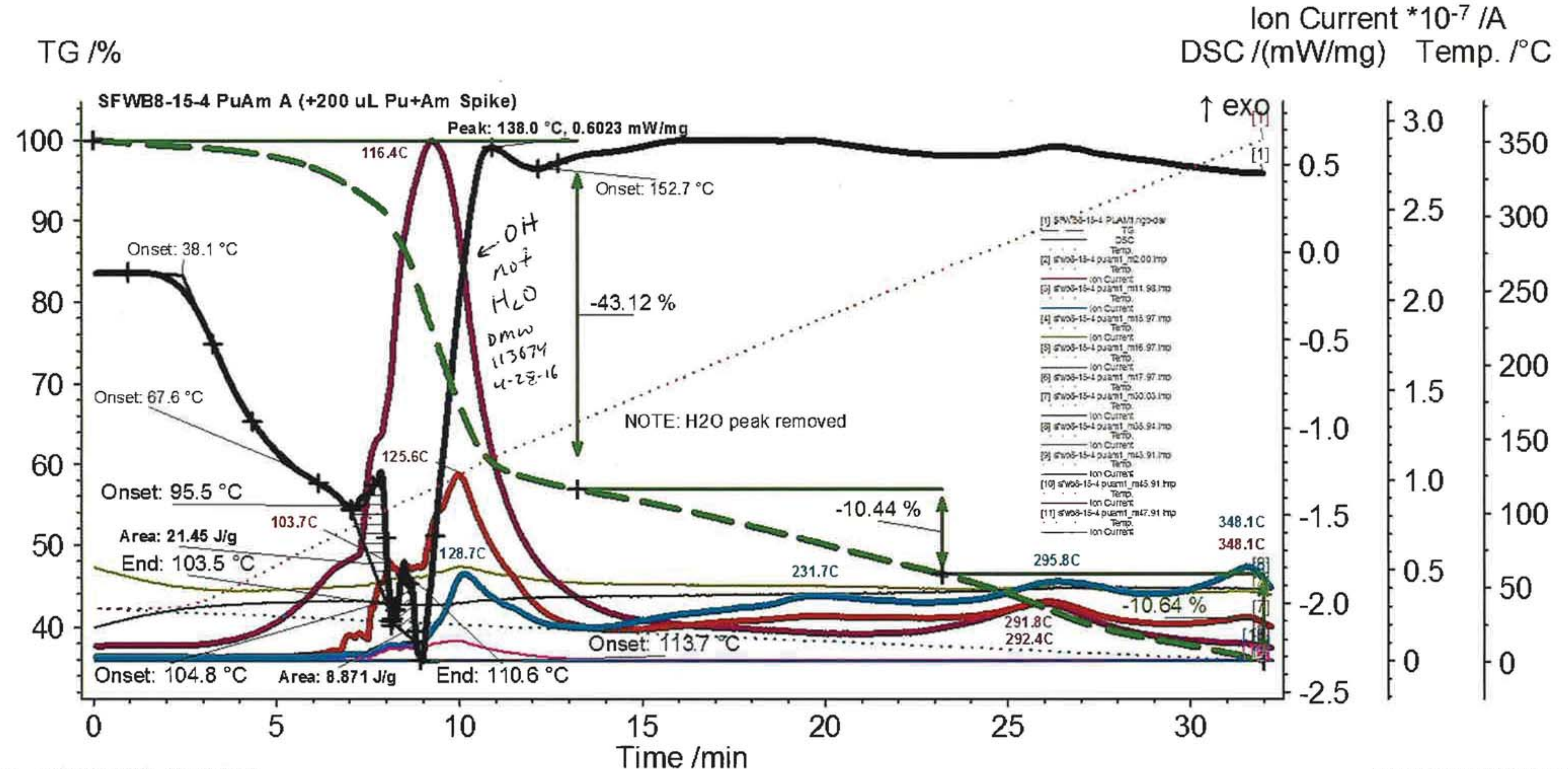
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4 PUAM1.ngb-dsv
Date/Time: 3/30/2016 1:35:17 PM (UTC-6)
End Date/Time: 3/30/2016 2:07:18 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-4 PUAM1
Sample name: SFWB8-15-4 PUAM1
Sample Mass: 20.30 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4 PUAM1 surrogate + spike 1

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	38.1 °C	1.0 min	5.3 min
Onset (DSC)	67.6 °C	4.3 min	6.7 min
Onset (DSC)	104.8 °C	8.2 min	8.5 min
Onset (DSC)	113.7 °C	8.9 min	11.2 min
Onset (DSC)	152.7 °C	12.2 min	13.9 min
Onset (DSC)	95.5 °C	7.1 min	7.7 min
End (DSC)	103.5 °C	7.1 min	8.1 min
Area (DSC),o	21.45 J/g	7.0 min	8.2 min
End (DSC)	110.6 °C	8.1 min	8.8 min
Area (DSC),o	8.871 J/g	8.1 min	8.8 min
Peak (DSC)	138.0 °C/0.6023 mW/mg	10.0 min	12.3 min



← OH
not
H_2O
DMW
113674
4-28-16

Main 2016-04-21 16:05 User: 113674

SFWB8-15-4 PUAM1.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segme...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-4 PUAM1.ngb-dsv	2016-03-30	SFWB8-15-4 PUAM1	SFWB8-15-4 PUAM1	20.30	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-4 puam1_m2.00.imp	2016-03-30	sfwb8-15-4 puam1	Mass 2.00		1/1	36°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4 puam1_m11.98.imp	2016-03-30	sfwb8-15-4 puam1	Mass 11.98		1/1	36°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4 puam1_m15.97.imp	2016-03-30	sfwb8-15-4 puam1	Mass 15.97		1/1	36°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4 puam1_m16.97.imp	2016-03-30	sfwb8-15-4 puam1	Mass 16.97		1/1	36°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4 puam1_m17.97.imp	2016-03-30	sfwb8-15-4 puam1	Mass 17.97		1/1	36°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4 puam1_m30.03.imp	2016-03-30	sfwb8-15-4 puam1	Mass 30.03		1/1	36°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4 puam1_m35.94.imp	2016-03-30	sfwb8-15-4 puam1	Mass 35.94		1/1	36°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-4 puam1_m43.91.imp	2016-03-30	sfwb8-15-4 puam1	Mass 43.91		1/1	36°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4 puam1_m45.91.imp	2016-03-30	sfwb8-15-4 puam1	Mass 45.91		1/1	36°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4 puam1_m47.91.imp	2016-03-30	sfwb8-15-4 puam1	Mass 47.91		1/1	36°C/1.1(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-4-PUAM1 **Location:** G220

Thermal Analysis Data Sheet

☒**Use Every Time**

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 3-30-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)*: TG / DSC Cal. File Name: WIPP TEMP 022916 / WIPP 022916	N/A	02/28/17	DMW Initials
Temperature & Humidity Monitor	041888	10-25-16	113674 Z No.
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08-03-16	3-30-16 Date

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 3-30-16 Date
Date & Time Sample Vial Opened	Date: 03/30/16 *(mm / dd / yy)* Time: 13:29 *(24 hour)*	N/A	N/A	
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/36.8°C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 3-30-16 Date DG. Initials 107962 Z No. 3/30/16 Date

MATL NAME: SFWB8-15-4-PuAM1 **Location:** G270

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 03/30/16 Time: 13:30	Mm/dd/yy – 24 hr.	N/A	DMMW Initials 113674 Z No. 3-30-16 Date	
Crucible / Pan Tare Wt.	0.16639	grams	N/A	DMMW Initials 113674 Z No. 3-30-16 Date	D.G. Initials 107962 Z No. 3/30/16 Date
Net Sample Weight	0.02030		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMMW Initials 113674 Z No. 3-30-16 Date	D.G. Initials 107962 Z No. 3/30/16 Date
Gas Pressure	Gas Pressure (at regulator): < 10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 11:57	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350C BASELINE 030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: ______________________ **Location:** ______________

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 3-30-16 Date	DG. Initials 107962 Z No. 3/30/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 3-30-16 Time Started: 13:35 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.3	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP SFWB8-15-4 PUAM1.ngb-dsv **Netzsch Measure** Filename: SFWB8-15-4 PUAM1. MDC **ThermoStar** Filename:	N/A	N/A	DMW Initials 113674 Z No. 3-30-16 Date	
Proteus Data	**Mass Changes:** 1) -43.12 Temp. Range (RT-162.5°C) 2) -10.44 Temp. Range (162.5-264.4°C) 3) - 10.64 Temp. Range (264.4-350°C) 4) ______ Temp. Range (__________) **Total mass Change:** -64.20 %	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 3-30-16 Date	DG. Initials 107962 Z No. 3/30/16 Date

Notes:

MATL NAME: SFWB8-15-4-PUAM1 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H_2O Peak T (116.4/291.8°C) 2) CO_2 Peak T (128.7/231.7/295.8)/348.1°C 3) NO/NO_2 Peak T (125.6/292.4/348.1°C 4) ______ Peak T (______) 5) ______ Peak T (______)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 3-30-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: ___/___/___ mm dd yy % Error (RSD, 1s): N/A DMW 113674 3-30-16 ______ = 100 x ______ ÷ ______ Std. Error Slope	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 3-30-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A		QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet do not need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A explanations

1) Cal File # does not exist for USER CALIBRATED ITEMS
2) Calib. Thermometer for chiller not required for WIPP
3) Temp. Profile not required to go to 1100°C for WIPP
4) seal RH% & Temp meas. not required for WIPP
5) moisture measurement not required for WIPP

DMW 113674 3-30-16

Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4 PUAM2.ngb-dsv
Date/Time: 4/7/2016 4:05:31 PM (UTC-6)
End Date/Time: 4/7/2016 4:37:32 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh

DSC DSC Range: 5000 µV
TG TG Range: 30000 mg
Sample identity: SFWB8-15-4 PuAm 2
Sample name: SFWB8-15-4 PuAm 2
Sample Mass: 10.254 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible

Remark: SFWB8-15-4 PuAm 2 - 2nd spiked run 4-7-16

Furnace: STD SiC(PC)
Sample carrier: DSC(/TG) HIGH RG 2
Measurement End: Normal end

Furnace TC: S
Sample TC: S

Gas1: ARGON **Flow:** 50 ml/min **predefined**
Gas2: ARGON **Flow:** 30 ml/min **predefined**
Gas3: <no gas> **Flow:** **predefined**

Start criteria
Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

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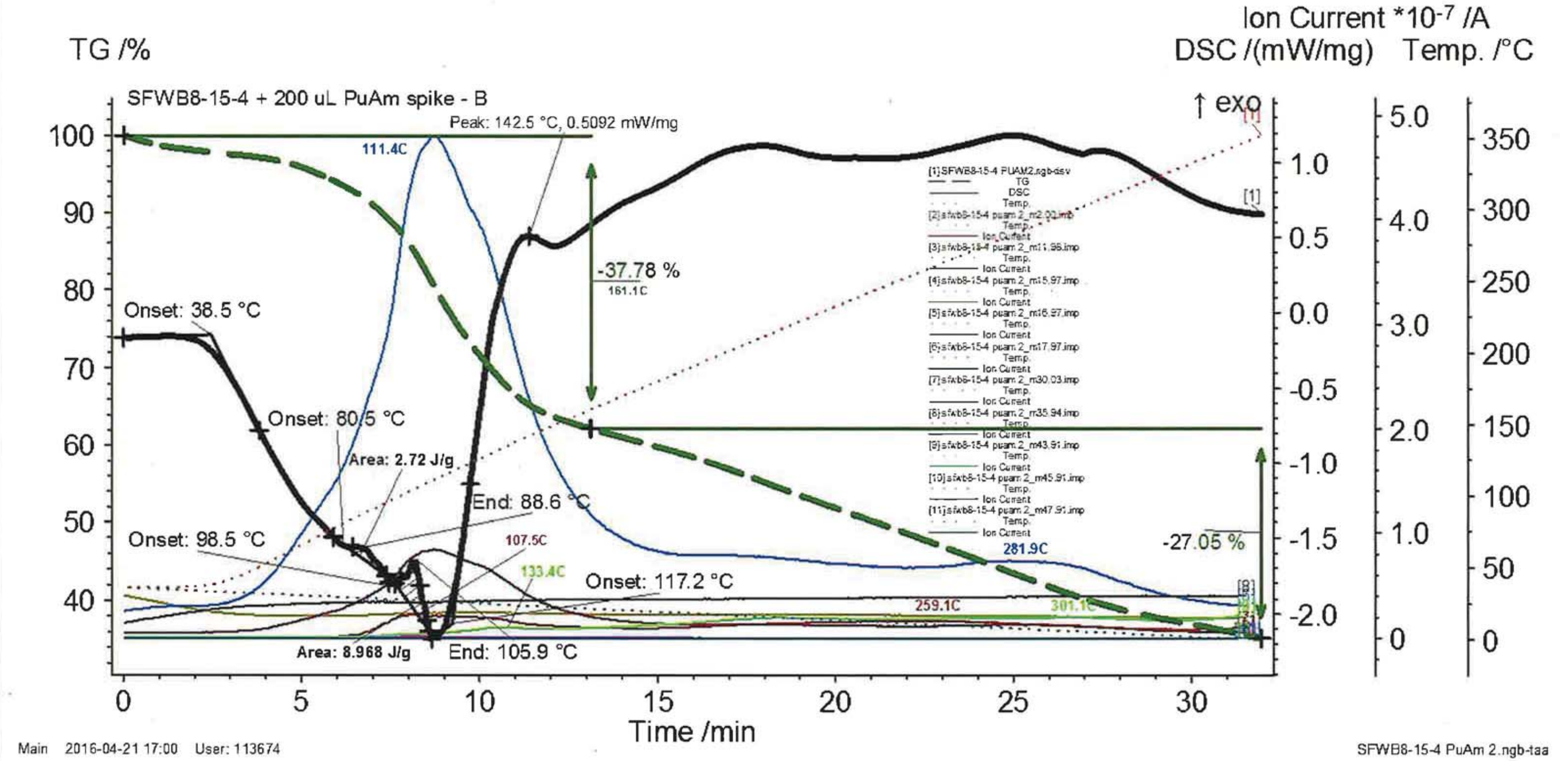
Instrument: NETZSCH STA 409PC/PG
Project: WIPP
Filename: SFWB8-15-4 PUAM2.ngb-dsv
Date/Time: 4/7/2016 4:05:31 PM (UTC-6)
End Date/Time: 4/7/2016 4:37:32 PM (UTC-6)
Laboratory: 55-0004-0208
Operator: DMW
Mode: DSC-TG
Measurement Type: sample with correction
Correction: WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.: WIPP temp 022916.ngb-tsv
Sensitivity: WIPP 022916.ngb-esv
Crucible: DSC/TG pan Pt-Rh
DSC DSC Range: 5000 µV

TG TG Range: 30000 mg
Sample identity: SFWB8-15-4 PuAm 2
Sample name: SFWB8-15-4 PuAm 2
Sample Mass: 10.254 mg
Crucible: DSC/TG pan Pt-Rh
Crucible Mass: 0 mg
Reference name: empty
Reference Mass: 0 mg
Reference Crucible Mass: 0 mg
Material: WIPP surrogate
Sample determination mode: Manual
Residuum measurement: Not possible
Atmosphere: ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4 PuAm 2 - 2nd spiked run 4-7-16

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	38.5 °C	0.0 min	5.6 min
Onset (DSC)	80.5 °C	5.9 min	6.9 min
Onset (DSC)	98.5 °C	7.5 min	7.9 min
End (DSC)	88.6 °C	5.9 min	7.4 min
Area (DSC),o	2.72 J/g	5.9 min	7.4 min
End (DSC)	105.9 °C	7.9 min	8.5 min
Area (DSC),o	8.968 J/g	7.6 min	8.6 min
Onset (DSC)	117.2 °C	8.7 min	11.6 min
Peak (DSC)	142.5 °C/0.5092 mW/mg	10.4 min	12.2 min



Main 2016-04-21 17:00 User: 113674

SFWB8-15-4 PuAm 2.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-4 PUAM2.ngb-dsv	2016-04-07	SFWB8-15-4 PuAm 2	SFWB8-15-4 PuAm 2	10.254	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-4 puam 2_m2.00.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 2.00		1/1	37°C/1.1(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4 puam 2_m11.98.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 11.98		1/1	37°C/1.1(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4 puam 2_m15.97.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 15.97		1/1	37°C/1.1(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4 puam 2_m16.97.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 16.97		1/1	37°C/1.1(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4 puam 2_m17.97.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 17.97		1/1	37°C/1.1(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4 puam 2_m30.03.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 30.03		1/1	37°C/1.1(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4 puam 2_m35.94.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 35.94		1/1	37°C/1.1(K/min)/1°C		---
[9] QMS 403	sfwb8-15-4 puam 2_m43.91.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 43.91		1/1	37°C/1.1(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4 puam 2_m45.91.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 45.91		1/1	37°C/1.1(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4 puam 2_m47.91.imp	2016-04-07	sfwb8-15-4 puam 2	Mass 47.91		1/1	37°C/1.1(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFWB8-15-4 PUAMQ **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	DMW Initials 113674 Z No. 4-7-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPPTemp022916 / WIPP022916	N/A	02/28/17	DMW Initials 113674 Z No. 4-7-16 Date
Temperature & Humidity Monitor	041888	10-25-16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	8-3-16	

Analysis Type: TG / [✓ DSC] other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	DMW Initials 113674 Z No. 4-7-16 Date	
Date & Time Sample Vial Opened	**Date:** 04/07/16 *(mm / dd / yy)* **Time:** 16:00 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.6/30.4	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	DMW Initials 113674 Z No. 4-7-16 Date	DG. Initials 107962 Z No. 4/7/16 Date

MATL NAME: SFWB8-15-4 PUAM2 **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	Date: 4-7-16 Time: 16:02	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4-7-16 Date	
Crucible / Pan Tare Wt.	0.16481	grams	N/A	DMW Initials 113674 Z No. 4-7-16 Date	DG Initials 107962 Z No. 4/7/16 Date
Net Sample Weight	0.01071		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES / [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-7-16 Date	DG. Initials 107962 Z No. 4/7/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 15	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350CBASELINE030816.ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: ____________________ **Location:** ____________

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-7-16 Date	DG. Initials 107962 Z No. 4/7/16 Date
	Maximum Temp.: 350°C (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 4-7-16 Time Started: 16:09 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.4	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-4 PUAM2.ngb-dsv **ThermoStar** Filename: SFWB8-15-4 PUAM2.MDC	N/A	N/A	DMW Initials 113674 Z No. 4-7-16 Date	
Proteus Data	**Mass Changes:** 1) -37.78 Temp. Range (RT - 161.1°C) 2) -27.05 Temp. Range (161.1°C -350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -64.83 %	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-7-16 Date	DG. Initials 107962 Z No. 4/7/16 Date

Notes:

MATL NAME: SFWB8-15-4 PuAM2 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) H_2O/OH Peak T (111.4, 281.9°C) 2) CO_2 Peak T (133.4, 301.1°C) 3) NO/NO_2 Peak T (107.5, 259.1°C) 4) ______ Peak T (__________) 5) ______ Peak T (__________)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 4-7-16 Date	
Total Moisture (H_2O)	Total Moisture = ______ mg ______ Wt % Date of most recent calibration: ______ mm dd yy % Error (RSD, 1s): N/A ______ = 100 x ______ ÷ ______ Std. Error Slope 113674 DMW 4-7-16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4-7-16 Date
Supervisor – DS Review	N/A	N/A		Supervisor 095012 Z No. 4/26/16 Date
Quality Representative – DS Review	N/A	N/A	HWM 149274 4/28/16	HW ~~1449274~~ QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

DMW 113674 4-7-16

N/A Caveats

1) user calibrated TGA-DSC is user calibrated - no file # exists.
2) Calib. thermometer not required for WIPP work
3) non-3013 analyses do not have to go to 1100°C
4) seal % RH/Temp not required for non-3013 work
5) moisture analysis not required by WIPP

DMW 113674 4-7-16

Instrument:	NETZSCH STA 409PC/PG	**DSC DSC Range:**	5000 µV
Project:	WIPP	**TG TG Range:**	30000 mg
Filename:	SFWB8-15-4 PuAm 3.ngb-dsv	**Sample identity:**	SFWB8-15-4 PuAm 3
Date/Time:	4/8/2016 1:57:31 PM (UTC-6)	**Sample name:**	SFWB8-15-4 PuAm 3
End Date/Time:	4/8/2016 2:29:32 PM (UTC-6)	**Sample Mass:**	17.830 mg
Laboratory:	55-0004-0208	**Crucible:**	DSC/TG pan Pt-Rh
Operator:	DMW	**Crucible Mass:**	0 mg
Mode:	DSC-TG	**Reference name:**	empty
Measurement Type:	sample with correction	**Reference Mass:**	0 mg
Correction:	WIPP 350C Baseline 030816.ngb-bsv	**Reference Crucible Mass:**	0 mg
Temp.Calib.:	WIPP temp 022916.ngb-tsv	**Material:**	WIPP surrogate
Sensitivity:	WIPP 022916.ngb-esv	**Sample determination mode:**	Manual
Crucible:	DSC/TG pan Pt-Rh	**Residuum measurement:**	Not possible

Remark: SFWB8-15-4 PuAm 3 - 3rd run spiked

Furnace:	STD SiC(PC)	**Furnace TC:**	S
Sample carrier:	DSC(/TG) HIGH RG 2	**Sample TC:**	S
Measurement End:	Normal end		

Gas1:	ARGON	**Flow:**	50 ml/min	**predefined**
Gas2:	ARGON	**Flow:**	30 ml/min	**predefined**
Gas3:	<no gas>	**Flow:**		**predefined**

Start criteria

Reset after maximum standby time: No

List of temperature steps:

Num	Mode	Temp. °C	HR K/min	Acq.Rate pts/min	Duration hh:mm	STC	G1	G2	G3
---	Stand-by heating	30.0	20.0			1	1	1	0
---	Stand-by isothermal	30.0			00:05	1	1	1	0
1	Dynamic	350.0	10.0	100.00	00:32	1	1	1	0
---	Emergency	370.0					1	1	0
---	Final stand-by heating	30.0	40.0		00:08	1	0	0	0
---	Final stand-by isothermal	30.0			02:00	1	0	0	0

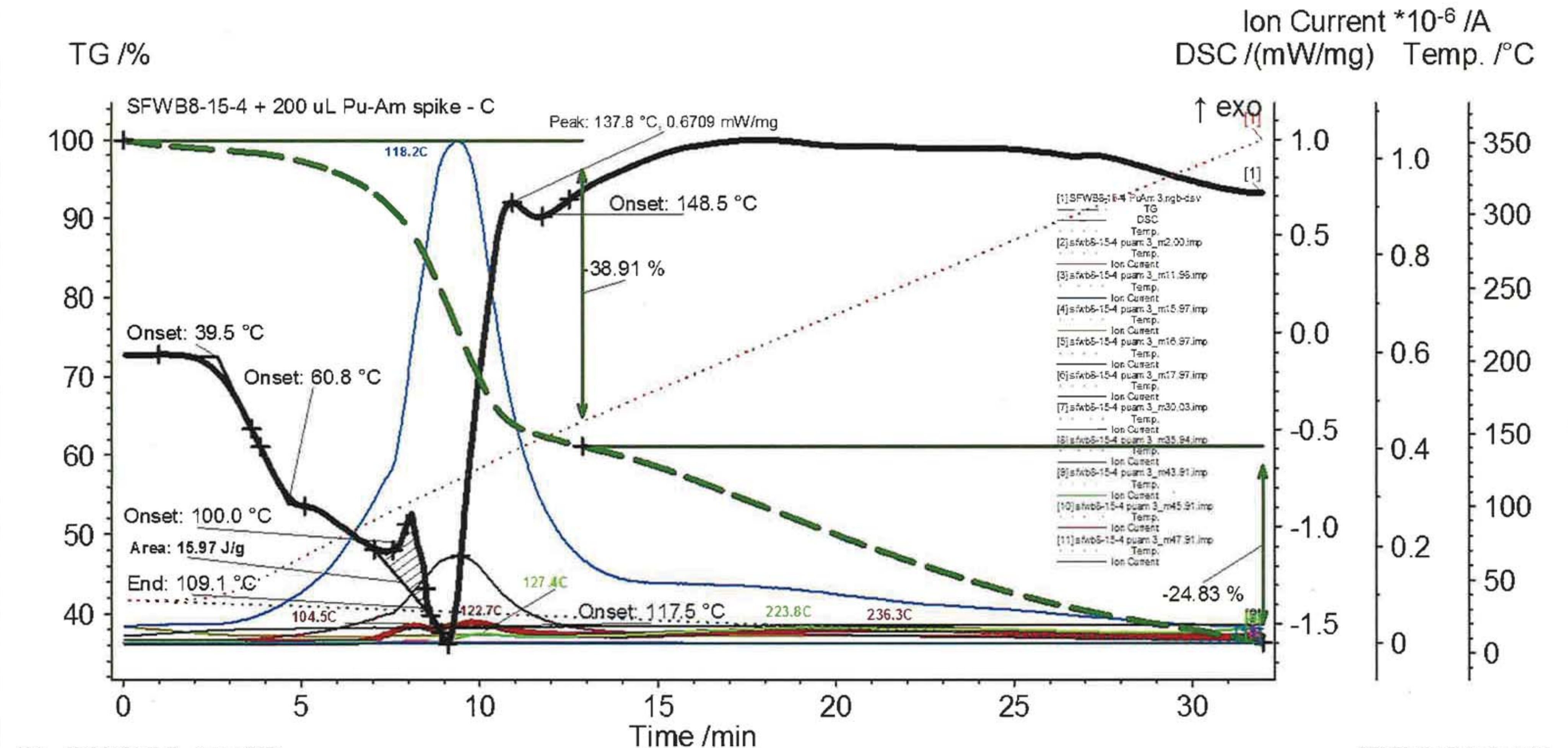
Instrument:	NETZSCH STA 409PC/PG
Project:	WIPP
Filename:	SFWB8-15-4 PuAm 3.ngb-dsv
Date/Time:	4/8/2016 1:57:31 PM (UTC-6)
End Date/Time:	4/8/2016 2:29:32 PM (UTC-6)
Laboratory:	55-0004-0208
Operator:	DMW
Mode:	DSC-TG
Measurement Type:	sample with correction
Correction:	WIPP 350C Baseline 030816.ngb-bsv
Temp.Calib.:	WIPP temp 022916.ngb-tsv
Sensitivity:	WIPP 022916.ngb-esv
Crucible:	DSC/TG pan Pt-Rh
DSC DSC Range:	5000 µV

TG TG Range:	30000 mg
Sample identity:	SFWB8-15-4 PuAm 3
Sample name:	SFWB8-15-4 PuAm 3
Sample Mass:	17.830 mg
Crucible:	DSC/TG pan Pt-Rh
Crucible Mass:	0 mg
Reference name:	empty
Reference Mass:	0 mg
Reference Crucible Mass:	0 mg
Material:	WIPP surrogate
Sample determination mode:	Manual
Residuum measurement:	Not possible
Atmosphere:	ARGON/50 / ARGON/30 / <no gas>/---

Remark: SFWB8-15-4 PuAm 3 - 3rd run spiked

Segments: 1/1 : 30°C/10.0(K/min)/350°C

Parameters	Result	Range (min)	Range (max)
Onset (DSC)	39.5 °C	1.0 min	5.1 min
Onset (DSC)	100.0 °C	7.6 min	8.1 min
Onset (DSC)	117.5 °C	9.1 min	10.9 min
Onset (DSC)	148.5 °C	11.8 min	17.7 min
Onset (DSC)	60.8 °C	3.9 min	5.8 min
End (DSC)	109.1 °C	7.9 min	8.9 min
Area (DSC),o	15.97 J/g	7.0 min	8.6 min
Peak (DSC)	137.8 °C/0.6709 mW/mg	9.2 min	11.6 min



Main 2016-04-21 16:40 User: 113674

SFWB8-15-4 PuAm 3.ngb-taa

[#] Instrument	File	Date	Identity	Sample	Mass/...	Segm...	Range	Atmosphere	Corr.
[1] STA 409PC/PG	SFWB8-15-4 PuAm 3.ngb-dsv	2016-04-08	SFWB8-15-4 PuAm 3	SFWB8-15-4 PuAm 3	17.830	1/1	30°C/10.0(K/min)/350°C	ARGON/50 / ARGON/30 / <no gas>/—	DSC:020, TG:020
[2] QMS 403	sfwb8-15-4 puam 3_m2.00.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 2.00		1/1	37°C/0.9(K/min)/1°C		---
[3] QMS 403	sfwb8-15-4 puam 3_m11.98.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 11.98		1/1	37°C/0.9(K/min)/1°C		---
[4] QMS 403	sfwb8-15-4 puam 3_m15.97.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 15.97		1/1	37°C/0.9(K/min)/1°C		---
[5] QMS 403	sfwb8-15-4 puam 3_m16.97.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 16.97		1/1	37°C/0.9(K/min)/1°C		---
[6] QMS 403	sfwb8-15-4 puam 3_m17.97.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 17.97		1/1	37°C/0.9(K/min)/1°C		---
[7] QMS 403	sfwb8-15-4 puam 3_m30.03.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 30.03		1/1	37°C/0.9(K/min)/1°C		---
[8] QMS 403	sfwb8-15-4 puam 3_m35.94.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 35.94		1/1	37°C/0.9(K/min)/1°C		—
[9] QMS 403	sfwb8-15-4 puam 3_m43.91.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 43.91		1/1	37°C/0.9(K/min)/1°C		---
[10] QMS 403	sfwb8-15-4 puam 3_m45.91.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 45.91		1/1	37°C/0.9(K/min)/1°C		---
[11] QMS 403	sfwb8-15-4 puam 3_m47.91.imp	2016-04-08	sfwb8-15-4 puam 3	Mass 47.91		1/1	37°C/0.9(K/min)/1°C		---

Created with NETZSCH Proteus software

MATL NAME: SFW88-15-4 PuAm2 **Location:** G220

Thermal Analysis Data Sheet

☒Use Every Time

DOP and DS Verification (Section 5.3)

Parameter	Data	Units	Acceptance Criteria	Performed By
Current DOP effective revision	R4	Revision number	DOP is latest effective revision, as verified on Documentum	OMW Initials 113674 Z No. 4-8-16 Date
Verify that DS is the current effective version.	[✓ Yes] / No *(circle one)*	N/A	Yes	

M&TE (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By
Carrier Type *(circle one)* **: TG / [✓ DSC]** **Cal. File Name:** WIPP temp 022916 / WIPP 022916	N/A	02/28/17	OMW Initials 113674 Z No. 4-8-16 Date
Temperature & Humidity Monitor	041888	10/25/16	
Calibrated Thermometer (for Water Chiller)	N/A	N/A	
Wall Clock	040480	08/03/16	

Analysis Type: TG / [✓ DSC] / other

Crucible Type: alumina TG beaker / alumina DSC pans / [✓ Pt DSC pans] / other

3013 ONLY: Verify TGA/DSC Temperature Profile (to 1100°C at ≤20°C/minute): Yes / No N/A

Glovebox Conditions (Sections 5.3 & 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
LANMAS / LAMCAS Balance ID	B366	N/A	N/A	OMW Initials 113674 Z No. 4/8/16 Date	
Date & Time Sample Vial Opened	Date: 04/08/16 *(mm / dd / yy)* Time: 13:52 *(24 hour)*	N/A	N/A		
Glovebox Conditions	Seal %RH/Temp.: N/A GB235%RH/Temp.: 0.4/30.9 °C	%RH/C°	%RH < 15, Seal %RH not >3% more than GB235 %RH	OMW Initials 113674 Z No. 4-8-16 Date	D.G. Initials 107962 Z No. 4/8/16 Date

MATL NAME: SFWB8-15-4 PUAMZ **Location:** G220

Sample Weights (Section 5.4)

Description	Cal. File No.	Cal. Exp. Date	Performed By	Description	
Date & Time Sample Weighed	**Date:** 04/08/16 **Time:** 13:54	Mm/dd/yy – 24 hr.	N/A	DMW Initials 113674 Z No. 4/8/16 Date	
Crucible / Pan Tare Wt.	0.16787	grams	N/A	DMW Initials 113674 Z No. 4/8/16 Date	D.G. Initials 107962 Z No. 4/8/16 Date
Net Sample Weight	0.01815		>3 g, < 18 g		

Instrument Setup (Section 5.4)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Evacuation / Backfill	Sample Chamber Evacuated? YES [✓ NO] *(circle one)* Backfill / Carrier Gas Type: UHP Ar	N/A	N/A	DMW Initials 113674 Z No. 4-8-16 Date	D.G. Initials 107962 Z No. 4/8/16 Date
Gas Pressure	Gas Pressure (at regulator): <10	psig	< 10		
Gas & Flowmeter Readings	Gas Flow 1: 50 Gas Flow 2: 30 Time gas flows turned on: 10 : 15	nh:mm	N/A		
Baseline (used for thermal buoyancy correction)	Filename: WIPP350CBaseline.030816ngb-bsv *('N/A' if no buoyancy curve is used)*	N/A	N/A		

MATL NAME: SFWB8-15-4 PuAM3 **Location:** G220

Run Parameters (Sections 5.4 & 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
Temperature Profile	Heating Rate: 10	C°/min	≥ 5, ≤ 20	DMW Initials 113674 Z No. 4-8-16 Date	D.G. Initials 107962 Z No. 4/8/16 Date
	Maximum Temp.: 350 (≥1000°C for 3013 Packaging)	C°	< 1500°C		
	Date Started: 04/07/16 Time Started: 13:58 Total Analysis Time: 00:32	24 hour - mm/dd/yy	N/A		
ThermoStar Reference	Sample Temp. at Start: 36.5	C°	N/A	N/A	
Run Data Files	**All Data stored on: "TA55 NMT-15 Powder Characterization on Winnmt2"** Folder: WIPP **Netzsch Measure** Filename: SFWB8-15-4 PuAM3.ngb-dsv **ThermoStar** Filename: SFWB8-15-4 PuAM3.MDC	N/A	N/A	DMW Initials 113674 Z No. 4-8-16 Date	
Proteus Data	**Mass Changes:** 1) -38.91 Temp. Range (RT-158.3°C) 2) -24.83 Temp. Range (158.3-350°C) 3) ______ Temp. Range (__________) 4) ______ Temp. Range (__________) **Total mass Change:** -63.74%	Wt. % / °C	Total < 0.4	DMW Initials 113674 Z No. 4-8-16 Date	DG. Initials 107962 Z No. 4/8/16 Date

Notes:

MATL NAME: SFW88-15-4 PuAM3 **Location:** G220

Gas Data (Section 5.6)

Parameter	Data	Units	Acceptance Criteria	Performed By	Verified By
ThermoStar Data	Volatile Species: 1) OH/H_2O Peak T (118.2°C) 2) CO_2 Peak T (127.4, 223.8)°C 3) NO/NO_2 Peak T (104.5, 122.7, 236.3°C 4) ____ Peak T (____) 5) ____ Peak T (____)	Volatile Species / °C	N/A	DMW Operator 113674 Z No. 04/08/16 Date	
Total Moisture (H_2O)	Total Moisture = ____ mg ____ Wt % Date of most recent calibration: __ / __ / __ (mm dd yy) % Error (RSD, 1s): N/A = 100 x ____ ÷ ____ Std. Error Slope DMW 113674 04/08/16	Wt. %	< 0.32	Initials Z No. Date	Initials Z No. Date

Signatures

Parameter	Data	Units	Acceptance Criteria	Performed By
Operator – DS review	N/A	N/A		DMW Operator 113674 Z No. 4/8/16 Date
Supervisor – DS Review	N/A	N/A		Z.A. Supervisor 095012 Z No. 4/28/16 Date
Quality Representative – DS Review	N/A	N/A		Hwm QR 149274 Z No. 4/28/16 Date

Note: Hand-written N/A entries must have SME explanation. However, computer-generated (typed) N/A entries on this data sheet <u>do not</u> need SME explanation. QR review will ensure all entries are complete before approving.

Comments:

N/A caveats

1 OPC instrumentation does not req. Cal File #. DMW 113674 4-28-16

2 - Calib. Thermometer not required to monitor ~~wtter~~ chiller water

3 - Profile not required to go to 1100°c

4 - Seal %RH & Temp. measurement not required

5 - moisture content not required

113674 - DMW 4-8-16

1-21-16 $RbNO_3$ test Cont'd

	T_{cert}	ΔHcert	T_{meas}	ΔHmeas
1	164.2	-26.6	164.2	-33.09
2	285.0	-8.75	284.5	-9.121

1-28-16 KNO_3 Run DMW 113674

BALANCE: B366

measured	"certified"
① 129.0°C; -58.18 J/g	129.0°C ; 49.47 J/g
② 333.3°C; -102.2 J/g	334.4°C ; 98.35 J/g

COMPUTER CRASH!

2-17-16 Replaced Computer

DMW 113674 Upgraded Software to V6.0

Recalibration starting - Balance calibrated 2/22

BIPHENYL T = 27.3°C %RH = 0.5% @ temp.

G 0.29419

T 0.25828 (w/Lid)

N 0.03591

meas	cert
68.7°C / -114.7	69.2°C / -120.5 J/g

Benzoic Acid

G 0.27262

T 0.24366 (w/Lid)

N 0.02896

meas	cert
121.0°C / -132.9 J/g	122.4°C / -147.4 J/g

$RbNO_3$ ~~meas~~ ~~cert~~

G 0.19832

T 0.16891 started @ 14:45

N 0.02941

~~meas~~ cert	~~cert~~ meas
164.2 / -26.6	163.6 / -33.22
285.0 / -8.75	282.7 / 9.134
N/A	218.7 / 25.05

$Ag_2(SO_4)$ starte 4:00PM

G 0.21938

T 0.16891

N 0.039

meas	cert
421.3°C / -59.25 J/g	426.4 / -51.9 J/g

Good!

WALL CLOCK: CalFile# 040480 exp 08-03-2016

Humidity Monitor 041888 10-25-2016

DMW 113674 2-25-16 %RH=0.5 @ 31.7°C

K_2CrO_4

G 0.19804

T 0.17946 meas 666.2°C / -32.36 cert 668.0 / -37.0 J/g

N 001858

CsCl meas 469.3°C / -16.36 J/g cert 476.0 / -17.2 J/g

G 0.21533

T 0.16958

N 0.04575

2-29-16 DMW 113674

%RH = 0.5% @ 27.7°C

G 0.20627 10:55 AM start ; Running to 895°C

T 0.17849

N 0.02778 meas cert

801.8 / 57.98 J/g 808.0°C / -94.9 J/g

WIPP Calibrations Complete! 2-29-16

Sensitivity & Temperature

3-1-16

Running baseline to 1100°C w/calibrations

Start @ 10:47 AM

KNO_3 calibration check

G 0.21189 T_{ST} 37.6°C

T 0.16897 E_{ST} -6.58 mV

N 0.04292 Time 14:35

MEAS CERT

132.3 / -62.02 J/g 128.7 / -50.0 J/g

3-3-16 Re-DO $BaCO_3$ calib run DMW 113674

G 0.19731 Crush $BaCO_3$ to powder first! %RH= 0.5 @ 30.2°C

T 0.16898 T_{ST}

N 0.02833 E_{ST}

Time

meas cert

803.3°C / 58.83 J/g 808.0°C / -94.9 J/g

3-3-16

KNO_3 Re-Do Calib run. Crush KNO_3 Powder First!!

G: 0.20569

T: 0.17036 E_{ST} = -6.51 mV

N: 0.03533 T_{ST} = 37.2 °C

Time = 2:06 PM

] Data not usable - baseline not completed. OK - will rerun w/out baseline

Heater / Inter-face / Gases off

BALANCE CALIBRATION - complete 3-7-16

3-7-16 113674 DMW

rerunning KNO_3 from 3-3. No baseline.

0.3% RH @ 27.0 °C "cert"

onset = 131.5 °C 128.7 °C

ΔH = 62.07 J/g -50.0

3-8-16 113674 DMW

Baseline to 350 °C T_{ST} 38.2 °C

E_{ST} -6.37 mV

Time 9:43 AM

Good!

Baseline to 550 °C T_{ST}: 38.3 °C

E_{ST} -6.40 mV

Time 13:47

P_{TOT} = 1×10⁻⁶ mbar %RH = 0.6% @ 33.7 °C

GAS ON 8:59

3-9-16 SFWB8-15-1 3716A %RH = 0.5% @ 29.9 °C

G 0.20756 P_{TOT} 9.4×10⁻⁷ mbar

T 0.16909 T_{ST} = 35.4 °C

W 0.03847 E_{ST} - 0.03757 mW/mg

Time Opened: 08:53 Time: 9:21:55

Time Weighed: 08:56 Run to 350 °C - good

☆ Crud in bottom of crucible was sanded off prior to run.

Start Ar ~15% END Ar ~% = 56%

SFWB8-15-1 030716B

G: 0.17599 weighed @ 11:11 T_{ST} 37.5 °C

T: 0.16801 opened @ 11:09 E_{ST} -5.46 mV

N: 0.00798 Time 11:22

SFWB8-15-1 030716B con'd 113674 3/8/16 DMW

†!! Ar@ Start = ~~50.5%~~ 47.3% ↑

A r@ end = not known.

SFWB8-15-1 030716C RNS Surrogate - no additions

G 0.19917 open 14:04 T_{SF} 36.3°C

T 0.16748 weigh 14:06 E_{ST} -4.51 mV

N 0.03169 Time 2:22 PM

Ar@ start ~ 68.0% good

Ar@ end ~ 78.7% & O_2 ~ 6700 ppm

Added ~~~ 0.02 mL~~ ~250-300 ul of 10 M HNO_3 + 0.3 M HF to vial #2 (labelled "ACID") - mixed with surrogate. 4:08 pm

Loosely capped - letting it sit overnight.

GAS ON @ 9:20 AM

3-10-16 DMW 113674 Surrogate + ACID runs

SFWB8-15-1 ACID A opened @ 9:24 AM

G 0.19311 9:27 AM weighed T_{SF} 35.5°C

T 0.16648 E_{SF} -5.09 mV -5.10

N 0.02663 Time 9:46 AM

Ar@ Start ~37% ↑ $P_{TOT} = 9.41 \times 10^{-7}$ mbar

Ar @ end N/A %RH = 0.6% @ 28.5°C

sample looks the same - a little more gooey.

Good run. results pretty similar to dry runs,

MS - BAKING OUT BETWEEN RUNS P_{TOT} = 8.2×10^{-7} mbar

SFWB8-15-1 ACID B open 11:17

G 0.18378 weigh 11:19

T 0.16960 T_{SF} 39.8°C

N 0.01418 E_{SF} -4.91 mV

Ar@ Start 6.8% ↑ Time 11:23

Ar@ end

SFWB8-15-1 ACID C open: 14:05

G 0.18929 weigh 14:07

T 0.16890 T_{ST} 38.3 @ end

N 0.02039 E_{ST} -4.45 mV $P_{TOT} = 9.1 \times 10^{-7}$ mbar

Ar Start : ~10% Time 2:15 pm

Ar End : N/A %RH = 0.6 @ 33.1°C GOOD

3-11-16 DMW 113674 Baseline DSC to 1100 °C

P_{TOT} = 1.1×10⁻⁶ mbar T_{ST} 35.6 °C

saving MCD gas data E_{ST} -6.34 µV

%RH = 0.6 @ 31.5 °C Time 10:26 AM

3-15-16 DMW 113674 %RH = 0.6 @ 34.1 °C

Gas on @ 9:37 P_{TOT} = 8.3×10⁻⁷ mbar

Check calibration $RbNO_3$

G 0.19808 T_{ST} 39.1

T 0.17943 E_{ST} -6.28 µV

W 1865 Time 10:10 AM end

Ar Start ~ 28.9%

O start ~ 4.2%

Ar end ~ 35.1%

O end ~ 3.9%

$RbNO_3$:	cert		meas
	164.2 °C	-26.6	165.9 °C / -31.33
	285 °C	-8.75	286 °C / -8.64

middle: 221.3 / -24.68 J/g

Received 4 samples 1 of SFWB8-15-1

3 of SFWB8-15-4

opened bags @ 15:33

introed @ 15:35 Gas on @ 15:35

%RH = 0.6 @ 31.6 °C P_{TOT} = 9.3×10⁻⁷ mbar

SFWB8-15-4 A opened @ 15:45

G 0.19980 15:47 Ar @ Start ~ 20% O @ Start ~5.2

T 0.16693 Ar @ end O @ end

N 2287 mg

Time = 3:57 PM T_{ST} = 37.0 E_{ST} = -4.28 µV

- Seems to have exploded or combusted suddenly about 9-9.5 minutes into run.
- Lost ~150 mg (lid) & then regained it 90 seconds later?

What Happened: Swelling of sample / Foaming so much that the sample briefly came into contact w/ the Furnace wall. Shrank & withdrew, giving the same weight again.

3-16-16 $p_{tot} = 8.1 \times 10^{-7}$ mbar DMW 113674
Baked out & cleaned System overnight - 1050°C for
30 minutes. %RH = 0.6 @ 30.5°C GAS ON @ 14:49
Still a small amount of residue on top part of
Pt platform. Running it anyway... SFWB8-15-4 B
G 0.18032 open @ 15:01 Time 3:10 PM (15:10)
T 0.16795 weigh @ 15:03 E_{ST} = -5.27 mV
N 1237 T_{ST} = 38.1°C
Hard white/yellow granule component
Ar @ Start ~22% Ar @ end N/A GOOD!
O @ Start ~4.6% O @ end N/A
little swelling - maybe 2X to 3X - lid barely lifted

3-16-16 SFWB8-15-4 C open 16:01
31.33 G 0.18020 ~~16:03~~ 16:06 Time: 4:24 PM
64 T 0.16641 ↑ ↗ E_{ST} : -4.87
N 1379 T_{ST} : 51.7
(removed ~5 mg from OG.) Brown/Yellow slime component
Ar @ Start < 1% Ar @ end swelled ~5X
O @ Start ~7% O @ end
started run kind of HOT ~51.0°C

3-17-16 $p_{tot} = 9.4 \times 10^{-7}$ mbar %RH = 0.6 @ 30.8°C
Gases in @ 10:02 AM SFWB8-15-4 D
G: 0.18133 open @ 10:34 A.M Slime Component
T: 0.16486 Weigh @ 10:35 A.M.
N: 16.47 mg Ar @ Start 37.6% O @ Start 3.9%
Ar @ End O @ End
Time start: 10:53 A.M. Tst: 36.4°C Est: -5.61

3-17-16 DMW 113674 SFWB8-15-4 ACID A
Opened @ 14:36 Slime mostly
G 0.18874 14:40 Ar @ Start 12.2% O @ start
T 0.16956 Ar @ Finish O @ Finish
N 19.18 ← NOT USED - sample continually drying out
tx - entered 17.204 mg into software
T_{ST} = 37.3 16.886 mg @ Start
E_{ST} = -4.83
Time - 2:46 PM

SFWB8-15-4 ACID B GAS on @ 9:47 AM

3-18-16 DMW 113674 P_{TOT} = 8.4×10^{-7} mbar

Opened vial @ 9:58 %RH = 0.6 @ 35.0°C

weigh @ 10:00 → G 0.18612 10:00 AM Ar @ start ~15% Ar @ end = 60%

T 0.16886 O @ start ~5% O @ end = 1.86%

N 0.01726 ← NOT USED

sample dehydrates in air wt @ start: 15.940
used 16.395 mg

T_{ST} 38.8°C
E_{ST} -4.07 μV Good run - same as w/o acid
10:06 AM

SFWB8-15-4 ACID C open @ 11:36 AM

3-18-16 DMW 113674 P_{TOT} 9.2×10^{-7} mbar

G 0.18680 11:38 AM mostly yellow brown goo

T 0.16747 %RH = 0.6 @ 32.2°C

N 0.01933

Ar @ start 10% Ar @ end = 83% E_{ST} -4.17 μV

O @ start 5.8% O @ end = 0.4% T_{ST} 40.8°C

Time 11:42 AM

Mass of sample probably close to 15 mg

good!

3-21-16 DMW 113674 Calib. Check $RbNO_3$

G 0.19651 %RH = 0.6 @ 35.2°C

T 0.17031 P_{TOT} = ~1×10^{-6} mbar

N 0.02620 = 8.5×10^{-7} @ 14:52

Saving MCD gas data.

Ar @ end 37.7% T_{ST} = 37.5°C

O @ end 3.65% E_{ST} = -6.33 μV

14:58

meas	cert
166.0°C / -31.48 ~~[illegible]~~ J/g	164.2°C / -26.6 J/g
286.0°C / -8.679 J/g	285.0°C / 8.75 J/g

DMW 113674
3-23-16

DMW 113674 3-23-16 $P_{TOT} = 9.4 \times 10^{-7}$ mbar
GAS ON 10:49 %RH: 0.6 @ 30.4°C
SFWB8-15-2 A opened vial @ 11:26AM
G 0.19071 11:31AM T_{ST} 36.6
T 0.16744 E_{ST} -4.61
N 0.02327 Time 11:37AM
Ar@start 17% wt ~ 22.5 → 22.47 mg (approx)
O@start 5% Good Run!

SFWB8-15-2 B
Opened vial 14:30 T_{ST} 37.3
G 0.19346 14:32 E_{ST} ~~-4.93~~ 4.99
T 0.17942 Time 14:38
N 1404 Ar@Start ~20% O@Start ~5%
Ar@end ~73% O@end 1.0%
Good Run 22.820 @start

- Added 100 μL of 10m HNO_3 / 0.3 mHF to SFWB8-15-2 @ 15:37

SFWB8-15-2 C 3rd Run opened @ 16:01
G 0.18030 @ 16:03 T_{ST} 41.7
T 0.16641 E_{ST} -4.52
N 1389 Time 16:06
Ar@Start ~5% Ox @ Start ?
Ar@ Finish 73% Ox @ end 1.0%
Good! SFWB8-15-2 ACID A
3-24-16 Acid Samples - Sample + Acid sat overnight - no change.
$P_{TOT} = 9.4 \times 10^{-7}$ mbar %RH = 0.6% @ 30.7, Gas on @ 10:05
G 0.19630 10:52AM E_{ST} -4.24
T 0.16795 (open @ 10:49AM) T_{ST} 36.2°C
N 28.35 (used 24.941) Time 10:00 wt = 24.530 @start
Ar@Start ~15% O@Start 5.3% Started MS before TGA!
Ar@end O@end
Sample dehydrating during prep & loading.
2 white beads in snot. Beads soft & mushy after acid exposure.
Run ABORTED - no more >20mg runs!
Mass change indicated unusual activity.

3-24-16 DMW 113674

SFWB8-15-2 ACID A2 P_{TOT} = 9.4×10^{-7} mbar

opened @ 14:05 - very gooey! %RH: 0.6 @ 31.1 °C

G 0.18378 14:10 E_{ST} -4.52

T 0.16484 T_{ST} 36.9

N 0.01894 Time 14:17

weight used 18.292 mg 18.074

Ar @ Start 28.5% Ar @ end 63.46% - 63.64%

O @ Start ? O @ end 1.70% - 1.74%

yellow stuff only - good run!

3-25-16 DMW 113674 P_{TOT} = 9.5×10^{-7} mbar

CAS on: 10:27 AM SFWB8-15-2 ACID B %RH = 0.6 @ 30.1 °C

Opened @ 10:31 AM - white kernel sample

.18540 @10:32 G 0.18590 10:34 AM T_{ST} 36.0 °C

T 0.16958 E_{ST} -4.83 mV

N 0.01559 Time 10:42

Ar @ Start ~34.5% Ar @ end >60%

O @ Start ~4.1%, O @ end <2.3%

Good! Unusual peak in enthalpy!

SFWB8-15-2-ACID C opened vial 13:28

0.17450 @13:31 G 0.17500 13:30 E_{ST} -5.58 uV

T 0.16688 T_{ST} 37.4 °C

N 0.00812 Time 1:36

Ar @ start >16% Ar @ end ~70%

O @ start ~5.3% O @ end ~1.25%

analyzing white blobs - looking for strange exotherm again. No strange exotherm!

Good!

everything off for weekend

MONDAY 3-28-16 DMW 113674

Baking out MS - pumping down

Interfaces on

3-29-16 DMW 113674
- cleaned pans & lids
- stopped bakeout P_{TOT} = 8.7×10^{-7} mbar (no gas flow)

%RH = 0.5 @ 30.5°C

Calibration Check w/ Ag_2SO_4

G 0.20877 Ar@Start 21.7% O@start 5%
T 0.17031 Ar@end 33.9% O@end 4.07%
N 0.03846 T_{st} 38.8°C
E_{st} −6.29
Time 10:01

Cert / meas
426.4°C / −51.9 427.2°C / −51.47 J/g

3-30-16 Received SNM Spike @ 9:51 MDT
DMW 113674 P_{TOT} = 9.4×10^{-7} mbar
B366 up @ 9:26AM

1) Added 200 uL of spike to all 3 surrogates @ 11:45 - 11:55

2) Mixed spike into surrogate w/a wooden stick new stick for each.

3) witnessed by Paul DeBurgomaster
(Gas on @ 11:57 AM)

200 uL of spike contains $\sim$ ~~31.2 mg~~ 3.12 mg Am ← 3.12 mg
$\sim$ ~~55.4~~ mg Pu ← 5.54 mg

GAS ON @ 13:17

SFWB8-15-4 PUAM1 %RH = 0.6 @ 36.8°C
G 0.18669 13:30 vial re-opened @ 13:29
T 0.16639 13:27 E_{st} −4.28
N 0.02030 T_{st} 36.3°C Loaded 1 white
Time 13:35 kernel + some
Ar@Start >13.5% O@start >5.3% yellow-brown
Ar@end ~~33~~.41% O@end $\sim$3.4% slime

☆ Good Run! Considerable foaming.

→ opened SFWB8-15-2 PUAM1 @ 14:58 ←
G 0.18044 15:00 T_{st} 40.9°C 2 small kernels
T 0.16796 E_{ST} −4.50 mV + slime
N 0.01248 Time 15:04
A@start $\sim$12% O@Start $\sim$5.4%
Ar@end 53.7% O@end $\sim$2.5% GOOD RUN!

16:00 - 3.30.16 Plant in MODE 2
Forced Exit.

4-6-16 DG 107967
RbNO3
G: 0.19700
T: 0.16748
N: 24.52 mg

%RH 0.6 @ 33.0 °C
P_{TOT} = 8.8×10^{-7} mbar
end
Ar % @ start 10.6% 15.2%
O % @ start 5.7% 5.4%
E_{ST} -6.35 mV
T_{ST} 37.3 °C
Time = 14:33

RbNO3

Cert	Meas	
164.2 / -26.6	165.9 °C / -30.5	Good
285.0 / -8.75	285.9 °C / -7.905	

4-7-16 Samples Received & Intro-ed @ 10:13
DMW 113674
P_{TOT} = 9.7×10^{-7} mbar %RH = 0.6% @ 30.4 °C
GAS on @ 10:15 AM
Adding Spike (200 ul) to Surrogates 10:18-10:28
1) witnessed by Daniel J. Garcia
3.12 mg Am 5.54 mg Pu 2) mixed w/ wooden stick - used new stick for each
→ 3) Spike = 3H ~~31.2 mg Am~~, ~~55.4 mg Pa~~
PuAm1 - SFWB8-15-1 @ 13:39
G 0.19160 opened @ 13:36 - closed 1338
T 0.16888 T_{ST} 36.2
N 0.02212 ← @ 13:39 E_{ST} -4.69
22.384 used @ 13:44 Time 13:47
21.994 @ Start 13:47
Ar @ Start 18.75 % Ar @ Finish —
O @ Start 5.2 % O @ Finish —
Good! - All yellow goo
SFWB8-15-4 PuAm2 4-7-16 DMW
G 0.17552 16:02 opened 16:00
T 0.16481 T_{ST} 36.4 °C
N 0.01071 E_{ST} -4.89 mV
@ START 9.962 Time 16:09

SFWB8-15-4 PuAm2 - cont'd
Ar @ start 14.6% Ar @ end 51.7%
O @ start 5.5% O @ end 2.58%
2 white blobs + 500
Good!

4-8-16 DMW 113174 P_{TOT} = $9.6 \cdot 10^{-7}$ mbar
Gas on @ 9:20 AM %RH = 0.6% @ 30°C

SFWB8-15-2 PuAm2 opened @ 9:33 AM
G 0.19217 9:36 E_{ST} = -5.31 mV
T 0.16957 T_{ST} = 35.3 °C
N 0.02260 @ 9:40 wt = 22.504 Time 9:41
Ar @ Start = 7.3 Ar @ end 49.1%
O @ start: 6.2 O @ end 2.76%
wt @ start 22.174
NOTE: opened vent @ 9:42 ~ 1:05 into run
Good!

SFWB8-15-[illegible] PuAm2 opened vial @ 11:24 AM
G 0.20287 T_{ST} 37.1
T 0.17983 E_{ST} -5.48
~~N 0.02079~~ Time 11:34
22.68 mg 11:28 AM
19.960 mg 11:31 ← used 19.662 mg
Ar @ start ~~15~~ 17.9 Ar @ end 58.8%
O @ start 5.4 O @ end 2.27%
Run OK!

~~Pu A~~ SFWB8-15-4 PuAm3 %RH = 0.4 @ 30.9 °C open! 13:52
Brand new unused Pt-Rh pan/lid P_{TOT} = 9.7×10^{-7} mbar
G 0.18602 13:54 E_{ST} -4.60
T 0.16787 T_{ST} 36.5
N 0.01815 used 17.830 13:59 Time 1:58 (14:01)
@ start 17.532
Ar % @ start 16.6 Ar % @ end 69.5 one large white kernel
O % @ start 5.48 O % @ end 1.20
Good Run!!

DMW SFWB8-15-2 PuAm3 4-8-16
113674

G 0.17825 15:31 E$_{ST}$ -4.21 mV
T 0.16706 T$_{ST}$ ~~-4.21~~ 38.8°C
N 0.01119 Time 3:34 pm
10.694 15:35
Ar @start 15.6% Ar@end 78.6%
O @start 5.3% O@end ~0.53%
single white kernel
Good!

SFWB8-15-1 PuAm 3 - couldn't run - got
Kicked out of lab @ 5pm

4-11-16 %RH = 0.4 @ 25.4 °C
Baking & pumping down MS
everything looks OK
4-12-16 new surrogates in @ 9:50 AM
Seal broken @ 10:30 AM
Introed in-line @ 10:37 AM
P$_{TOT}$ 8.5×10^{-7} mbar GAS on @ 10:41 AM
%RH = 0.6% @ 32.6°C
KNO$_3$ std. Run E$_{ST}$ -6.38 mV
G 0.20779 T$_{ST}$ 37.6°C
T 0.16750 Time 11:04
N ~~4037~~ 3999
Ar@Start 21.93% O@start 4.56%
Ar@end 40.27% O@end 3.54%

1- 130.7°C -60.81 J/g
2- 335.3°C -97.75 J/g

10:46 AM
NOTE ADDED 200 ul of Pu-Am Spike to SFWB8-15-1
Added 200 uL of HNO$_3$ + HF acid to SFWB8-15-2
(10:53 AM)
Added an additional 100 ul of Pu-Am spike to
SFWB8-15-1 AFTER the run SFWB8-15-1 PuAm3

4-12-16 DMW 113674

SFWB8-15-1 PuAm3 opened @ 14:00

G 0.18845 (14:02) E_{ST} ~~-3.99~~ V -4.03

T 0.17030 T_{ST} 36.7°C

N 0.01815 Time 14:06

Ar@Start 18% O@start 5.1%

Ar@end 54.8 O@end 2.37

used 17.338 (14:08) Sample appears to be self-heating when placed in carrier!

Good Run.

SFWB8-15-2 200uL of acid only open@ 15:50

G 0.19311 15:52 E_{ST} -4.33

T 0.16886 T_{ST} 37.8

N 0.02425 Time 16:04

22.848 15:57 → 22.128 @ 16:03 → 22.094 @ST

Ar@start 50.56 O@start 2.63

Ar@end O@end

- no little ~~exo~~ exo. rxns. between 85-105°C none at all, in fact.

Fe Nitrate into acid...

~~Cr nitrate into acid~~ ..

4-14-16 P_{TOT} = 9.52×10⁻⁷ mbar RH: 0.6 @ 26.1°C

SFWB8-15-4 400 very nasty looking yellow slime w/ bubbles.

G ~~0.19022~~ 0.18983 15:17

T ~~0.16688~~ 0.16960 15:22 white kernels broken down

N 0.02023 GAS ON @ 14:49

T_{ST} 35.6°C Ar@start 25.2 % Ar@end 42.4

E_{ST} -3.30 O@start 4.5 % O@end 3.26

Time ~~15:23~~ 15:24 $\boxed{2.439}$

1.61+

NOTE: Dissolved 0.82 g of $Fe(NO_3)_3 \cdot 9H_2O$ in the remaining 20 mL of HNO_3 + HF to make a ~3M $FeNO_3$ solution. Added 200uL to SFWB8-15-4 & stirred w/ wooden stick

@ 4:15 pm

running this sample FRIDAY

opened @ 9:43 AM

4-15-16 SFWB8-15-4 FeNO P_{TOT} = 9.5x10^{-7} mbar

G 0.18281 Time 9:56 Am % RH = 0.6 @ 29.0°C

T 0.16482 E_{ST} = -3.65 mV Gas On @ 9:40 AM

N 0.01799 9:45 AM T_{ST} = 34.9°C

→ 16.768 @ 9:54 Am (used) Wt @ start 16.634

Ar @ start 29.6 O @ start 4.27

Ar @ end 64 % O @ end 1.68 %

OK run - very tiny exo pk @ 108.7°C

NO_2 = 32+14 = 46

N_2O = 28+16 = 44

dissolving a SS 304(?) hex nut in acid - no good

wt = 1.20112 g

GAS on @ 10:57 Am -12.294 mg

200 ul Pu-Am spike dried in DSC crucible

GW = 0.17678 ~ 0.007 mg

Ar @ start 16 % O @ start 4 %

Ar @ end O @ end

T_{ST} 40.2°C

E_{ST} -6.54 mV

Time 11:40 AM

4-21-16 - BALANCE CALIBRATED

Los Alamos
NATIONAL LABORATORY
EST. 1943

Form 2033
Reference No:
SQAD-16-TA55-ACHAR-003

The Software Owner RLM must retain completed forms as a record.

Safety/Non-Safety Software Determination, Categorization, and Software Risk Level (SRL)

Part 1: Document the rationale supporting the reasonable probability that the software may be safety software, or risk significant software.

1.1 Excluding personal productivity software that does not provide calculation output (e.g. e-mail software, presentation software), indicate whether the software is or will be used in connection with the design, analysis and/or operation of:

☒ a nuclear (including radiological) facility (Ref. LANL Nuclear Facility List or Conduct of Operations Resources website), or

☐ an accelerator, live-firing range, biological hazard facility, high explosive facility, or moderate- or high- chemical hazard facility as determined using SBP111-1, *Facility Hazard Categorization and Documentation*; or

☐ LANL's Essential Functions as described in EPP-COOP-006, *LANL COOP [Continuity of Operations] Plan.*

Provide supporting comments (as necessary to document the selection above):

Manufacturer-supplied, instrument-specific software is used to operate, collect and process data from ML-4 scientific instrumentation located within a PF-4 glovebox. The software is not used for design, analysis of, or operation of, a Nuclear Facility.

Part 2: Document the software information, software application(s) and software function(s). A separate form may be used for each software item or one form may be used for multiple software items.

2.1 Provide software name(s) Netzsch Measure / Proteus	2.2 Provide software version(s) Any of the following: 4.3.1 / 17-05-2004 for Windows 2000 or XP; 4.5.8 / 11.08.2009 for Win XP; or 6.1.0 / 02.09.2015 for Win XP or Win 7	2.3 Indicate software owner (SO) David M. Wayne	2.4 Indicate SO Organization MET-1

2.5 Provide a description of the specific facility application(s) to sufficient detail to allow the software to be readily traceable to the point(s) of application within the facility. Include technical area (TA) and building number; or, site-wide or Facility Operating Directorate (FOD)-wide use. Add other information as required

Instrumentation and software located in TA-55, PF-4 and in PF3 . Software has no 'facility application.'

2.6 Indicate System, Structure or Components (SSCs) controlled or affected by the software. Indicate NA if not applicable

Simultaneous Thermal Analyzer

2.6.1 Provide SSC name(s): Netzsch STA 409 PC 'Luxx'

2.6.2 Provide functional requirement(s) of the software associated with the SSC:

software used to operate, maintain, calibrate, and process thermal analysis data obtained using the Netzsch STA 409 PC 'Luxx.'

2.6.3 Provide reference document(s) describing the SSC/software:

Netzsch: Operating Instructions for STA Apparatus STA 409 PC Luxx

Netzsch Software Manual – Netzsch Proteus Software for Win XP or 7, versions: 4.2, 4.3, (18.03.2002), 4.8.5 (11.08.2009) or 6.1.0 (02.09.2015).

Provide supporting comments (as required):
see PMT2-MPR-DOP-015

2.7 Indicate facility classification, design, or analysis controlled or affected by the software. Indicate NA if not applicable

NA

2.7.1 Provide facility classification, design or analysis name:

2.7.2 Provide software functional requirement(s) associated with the facility classification, design or analysis:

2.7.3 Provide reference document(s) describing the facility classification, design, or analysis:

Provide supporting comments (as required):

2.8 Indicate the hazard control, Safety Management Program (SMP) and or technical safety requirements (TSRs) controlled or affected by the software. Indicate NA if not applicable

NA

2.8.1 Provide the hazard control, SMP and/or TSR name:

2.8.2 Provide the software functional requirement(s) for the hazard control, SMP and/or TSR:

2.8.3 Provide reference document(s) describing the hazard control, SMP and/or TSR:

Provide supporting comments (as required):

Part 3: Determine whether the software type is (1) safety software; or (2) non-safety software and the associated category for each type.

3.1 Check **one** of the following (3.1.1 through 3.1.5) to determine one of the two software types (safety software or non-safety software) and one of the associated 5 categories for each type (i.e. Categories include SSS, SHADS or SMACS for safety software; and, Risk Significant or Commercially Controlled for non-safety software).

Note: If software is determined to be safety software or risk significant software, complete all parts of this form. If software is determined to be commercially controlled software, complete all parts of this form except for Part 4.

3.1.1 Safety software: SSS ☐

This is software for a nuclear (including radiological) facility that performs, or will perform a safety function as part of a Structure, System, and Component (SSC) and is cited in either (a) a Department of Energy (DOE)-approved documented safety analysis; or, (b) an approved hazard analysis per DOE P 450.4A, *Integrated Safety Management Policy* and 48 Code of Federal Regulations (CFR) 970-5223-1, *Integration of Environment, Safety, and Health into Work Planning and Execution.* This is safety software and is categorized as Safety System Software (SSS).

Provide supporting comments (as required):

3.1.2 Safety software: SHADS ☐

This is software that is used, or will be used to classify, design, or analyze nuclear (including radiological) facilities. This software is not part of an SSC, but helps to ensure the proper accident or hazards analysis of nuclear (including radiological) facilities or an SSC that performs a safety function. This is safety software and is categorized as Safety and Hazard Analysis Software and Design Software (SHADS).

Provide supporting comments (as required):

3.1.3 Safety software: SMACS ☐

☐ This is software that performs or will perform a hazard control function in support of nuclear (including radiological) facility radiological safety management programs (SMPs) or technical safety requirements (TSRs). This is safety software and is categorized as Safety Management and Administrative Controls Software (SMACS).

Provide supporting comments (as required):

☐ This is software that performs, or will perform a control function in support of a nuclear (including radiological) facility necessary to provide adequate protection from nuclear (including radiological) facility radiological hazards. It supports eliminating, limiting, or mitigating nuclear hazards to workers, the public, or the environment as addressed in 10 CFR 830, *Nuclear Safety Management,* 10 CFR 835, *Occupational Radiation Protection,* and the Department of Energy Acquisition Regulation (DEAR) Integrated Safety Management System (ISMS) clause 48 CFR 970.5223-1, *Integration of Environment, Safety, and Health into Work Planning and Execution*. This is safety software and is categorized as Safety Management and Administrative Controls Software (SMACS).

Provide supporting comments (as required):

3.1.4 Non-safety software: Risk Significant ☐	This is software that is, or will be used for any of the purposes that safety software is used for only such purposes are in or for an accelerator, live-firing range, biological hazard facility, high explosive facility, or moderate- or high- chemical hazard facility OR, failure of the software would prevent LANL from performing Essential Functions *as described in EPP-COOP-006, LANL COOP [Continuity of Operations] Plan.* This is non-safety software and is categorized as Risk Significant software. Provide supporting comments (as required):
3.1.5 Non-safety software: Commercially Controlled ☒	This is software that is not, or will not be used for any of the above purposes in 3.1.1–3.1.4. Such software may be acquired (including commercial off the shelf (COTS)) or designed software. Examples of this software include personal productivity software (e.g., Microsoft PowerPoint, Oracle Project Primavera, MS Outlook, etc.) and other types of software (e.g., some business accounting systems, facility personnel comfort temperature monitoring systems).This is non-safety software and is categorized as Commercially Controlled software. Proceed to Part 5. Provide supporting comments (as required):

Part 4: Determine the Software Risk Level (SRL).

4.1 Complete this section for safety software and risk significant (non-safety) software. Check **only one** of the following to determine the SRL. Text shown in *[brackets]* is applicable to safety software only.

SRL 1 ☐	4.1.1 This level includes software applications that meet one or more of the following criteria. Failure of the software could: - *[Compromise a limiting condition for operation].* - *[Cause a reduction in the safety margin for a safety SSC that is cited in a DOE approved documented safety analysis.]* - Cause a reduction in the safety margin for other systems such as toxic or chemical protection systems that are cited in either (a) a DOE approved documented safety analysis or (b) an approved hazard analysis per DOE P 450.4A, *Integrated Safety Management Policy*, and the DEAR ISMS clause (48 CFR 970.5223-1, *Integration of Environment, Safety, and Health into Work Planning and Execution*). - Result in non-conservative safety analysis, design, or misclassification of facilities or SSCs. Provide supporting comments (as required):
SRL 2 ☐	4.1.2 This level includes *[safety]* software applications that do not meet SRL 1 criteria, but meet one or more of the following criteria: - *[Safety management databases used to aid in decision making whose failure could impact safety SSC operation.]* - Software failure that could result in incorrect analysis, design, monitoring, alarming, or recording of hazardous exposures to workers or the public. - *[Software failure could compromise the defense-in-depth capability for a nuclear (including radiological) facility.]* Provide supporting comments (as required);
SRL 3 ☐	4.1.3 This level includes software applications that do not meet SRL 2 criteria, but meet one or more of the following criteria. Failure of the software could: - Cause a potential violation of regulatory permitting requirements. - Affect environment, safety, health monitoring, or alarming systems. - Affect the safe operation of an SSC. Provide supporting comments (as required):

Part 5: Attest to compliant completion, review and approve.

5.1 As the Software Owner (SO), I have determined the software type, category, and as appropriate, SRL, in accordance with P1040, *Software Quality Management* and the instructions associated with this form. Provide SO Name/Z No. (print): David M. Wayne / 113674	Signature	Date 11-23-15
5.2 As the Software Owner Responsible Line Manager (SO RLM), I have reviewed and approve the determination of the software type, category and, as appropriate, SRL for the software as described on this form. Provide SO RLM Name/Z No. (print): Kent Abney / 101670	Signature	Date 11/23/15
5.3 As the ☑ Facility Design Authority Representative (FDAR) for my representative facilities, or, as the ☐ LANL Design Authority (DA), I have reviewed and approve the determination of the software type, category and, as appropriate, SRL for the software as described on this form. Check one and provide Name/Z No. (print): Richard Hemphill 083862 **Note:** The Responsible Associate Director (RAD) is authorized to review and approve Form 2033s (rather than the FDAR or DA) for software applications where, as determined by the FDAR or DA, the FDAR or DA does not have the knowledge and/or a reasonable connection to the software.	Signature	Date 12/3/15

11-23-15

Los Alamos
NATIONAL LABORATORY
EST.1943

Form 2033
Reference No:
SQAD-16-TA55-ACHAR-002

The Software Owner RLM must retain completed forms as a record.

Safety/Non-Safety Software Determination, Categorization, and Software Risk Level (SRL)

Part 1: Document the rationale supporting the reasonable probability that the software may be safety software, or risk significant software.

1.1 Excluding personal productivity software that does not provide calculation output (e.g. e-mail software, presentation software), indicate whether the software is or will be used in connection with the design, analysis and/or operation of:

☒ a nuclear (including radiological) facility (Ref. LANL Nuclear Facility List or Conduct of Operations Resources website), or

☐ an accelerator, live-firing range, biological hazard facility, high explosive facility, or moderate- or high- chemical hazard facility as determined using SBP111-1, *Facility Hazard Categorization and Documentation*; or

☐ LANL's Essential Functions as described in EPP-COOP-006, *LANL COOP [Continuity of Operations] Plan.*

Provide supporting comments (as necessary to document the selection above):

Manufacturer-supplied, instrument-specific software is used to operate, collect and process data from ML-4 scientific instrumentation located within a PF-4 glovebox. The software is not used for design, analysis of, or operation of, a Nuclear Facility.

Part 2: Document the software information, software application(s) and software function(s). A separate form may be used for each software item or one form may be used for multiple software items.

2.1 Provide software name(s) Pfeiffer ThermoStar Suite (Quadstar 32-Bit)	2.2 Provide software version(s) 7.03, for Windows 2000 or XP	2.3 Indicate software owner (SO) David M. Wayne	2.4 Indicate SO Organization MET-1

2.5 Provide a description of the specific facility application(s) to sufficient detail to allow the software to be readily traceable to the point(s) of application within the facility. Include technical area (TA) and building number; or, site-wide or Facility Operating Directorate (FOD)-wide use. Add other information as required

Instrumentation and software located in TA-55, PF-4 and in PF-3. Software has no 'facility application.'

2.6 Indicate System, Structure or Components (SSCs) controlled or affected by the software. Indicate NA if not applicable

Quadrupole Mass Spectrometer

2.6.1 Provide SSC name(s): Pfieffer ThermoStar GSD301T3

2.6.2 Provide functional requirement(s) of the software associated with the SSC:

calibration, maintenance, and operation of a Pfeifer quadrupole mass spectrometer for gas analysis

2.6.3 Provide reference document(s) describing the SSC/software:

Pfeiffer Vacuum (BG 805 951 BE [0110]); OmniStar / ThermoStar Gas Analysis System, Operating Instructions

Provide supporting comments (as required):
see PMT2-MPR-DOP-015

2.7 Indicate facility classification, design, or analysis controlled or affected by the software. Indicate NA if not applicable

NA

2.7.1 Provide facility classification, design or analysis name:

2.7.2 Provide software functional requirement(s) associated with the facility classification, design or analysis:

2.7.3 Provide reference document(s) describing the facility classification, design, or analysis:

Provide supporting comments (as required):

2.8 Indicate the hazard control, Safety Management Program (SMP) and or technical safety requirements (TSRs) controlled or affected by the software. Indicate NA if not applicable

NA

2.8.1 Provide the hazard control, SMP and/or TSR name:

2.8.2 Provide the software functional requirement(s) for the hazard control, SMP and/or TSR:

2.8.3 Provide reference document(s) describing the hazard control, SMP and/or TSR:

Provide supporting comments (as required):

Part 3: Determine whether the software type is (1) safety software; or (2) non-safety software and the associated category for each type.

3.1 Check **one** of the following (3.1.1 through 3.1.5) to determine one of the two software types (safety software or non-safety software) and one of the associated 5 categories for each type (i.e. Categories include SSS, SHADS or SMACS for safety software; and, Risk Significant or Commercially Controlled for non-safety software).

Note: If software is determined to be safety software or risk significant software, complete all parts of this form. If software is determined to be commercially controlled software, complete all parts of this form except for Part 4.

3.1.1 Safety software: SSS ☐		This is software for a nuclear (including radiological) facility that performs, or will perform a safety function as part of a Structure, System, and Component (SSC) and is cited in either (a) a Department of Energy (DOE)-approved documented safety analysis; or, (b) an approved hazard analysis per DOE P 450.4A, *Integrated Safety Management Policy* and 48 Code of Federal Regulations (CFR) 970-5223-1, *Integration of Environment, Safety, and Health into Work Planning and Execution.* This is safety software and is categorized as Safety System Software (SSS). Provide supporting comments (as required):
3.1.2 Safety software: SHADS ☐		This is software that is used, or will be used to classify, design, or analyze nuclear (including radiological) facilities. This software is not part of an SSC, but helps to ensure the proper accident or hazards analysis of nuclear (including radiological) facilities or an SSC that performs a safety function. This is safety software and is categorized as Safety and Hazard Analysis Software and Design Software (SHADS). Provide supporting comments (as required):
3.1.3 Safety software: SMACS ☐	☐	This is software that performs or will perform a hazard control function in support of nuclear (including radiological) facility radiological safety management programs (SMPs) or technical safety requirements (TSRs). This is safety software and is categorized as Safety Management and Administrative Controls Software (SMACS). Provide supporting comments (as required):
	☐	This is software that performs, or will perform a control function in support of a nuclear (including radiological) facility necessary to provide adequate protection from nuclear (including radiological) facility radiological hazards. It supports eliminating, limiting, or mitigating nuclear hazards to workers, the public, or the environment as addressed in 10 CFR 830, *Nuclear Safety Management,* 10 CFR 835, *Occupational Radiation Protection*, and the Department of Energy Acquisition Regulation (DEAR) Integrated Safety Management System (ISMS) clause 48 CFR 970.5223-1, *Integration of Environment, Safety, and Health into Work Planning and Execution.* This is safety software and is categorized as Safety Management and Administrative Controls Software (SMACS). Provide supporting comments (as required):

3.1.4 Non-safety software: Risk Significant ☐	This is software that is, or will be used for any of the purposes that safety software is used for only such purposes are in or for an accelerator, live-firing range, biological hazard facility, high explosive facility, or moderate- or high- chemical hazard facility OR, failure of the software would prevent LANL from performing Essential Functions *as described in EPP-COOP-006, LANL COOP [Continuity of Operations] Plan.* This is non-safety software and is categorized as Risk Significant software. Provide supporting comments (as required):
3.1.5 Non-safety software: Commercially Controlled ☒	This is software that is not, or will not be used for any of the above purposes in 3.1.1–3.1.4. Such software may be acquired (including commercial off the shelf (COTS)) or designed software. Examples of this software include personal productivity software (e.g., Microsoft PowerPoint, Oracle Project Primavera, MS Outlook, etc.) and other types of software (e.g., some business accounting systems, facility personnel comfort temperature monitoring systems).This is non-safety software and is categorized as Commercially Controlled software. Proceed to Part 5. Provide supporting comments (as required):

Part 4: Determine the Software Risk Level (SRL).	
4.1	Complete this section for safety software and risk significant (non-safety) software. Check **only one** of the following to determine the SRL. Text shown in *[brackets]* is applicable to safety software only.
SRL 1 ☐	4.1.1 This level includes software applications that meet one or more of the following criteria. Failure of the software could: ▪ *[Compromise a limiting condition for operation].* ▪ *[Cause a reduction in the safety margin for a safety SSC that is cited in a DOE approved documented safety analysis.]* ▪ Cause a reduction in the safety margin for other systems such as toxic or chemical protection systems that are cited in either (a) a DOE approved documented safety analysis or (b) an approved hazard analysis per DOE P 450.4A, *Integrated Safety Management Policy*, and the DEAR ISMS clause (48 CFR 970.5223-1, *Integration of Environment, Safety, and Health into Work Planning and Execution*). ▪ Result in non-conservative safety analysis, design, or misclassification of facilities or SSCs. Provide supporting comments (as required):
SRL 2 ☐	4.1.2 This level includes *[safety]* software applications that do not meet SRL 1 criteria, but meet one or more of the following criteria: ▪ *[Safety management databases used to aid in decision making whose failure could impact safety SSC operation.]* ▪ Software failure that could result in incorrect analysis, design, monitoring, alarming, or recording of hazardous exposures to workers or the public. ▪ *[Software failure could compromise the defense-in-depth capability for a nuclear (including radiological) facility.]* Provide supporting comments (as required);
SRL 3 ☐	4.1.3 This level includes software applications that do not meet SRL 2 criteria, but meet one or more of the following criteria. Failure of the software could: ▪ Cause a potential violation of regulatory permitting requirements. ▪ Affect environment, safety, health monitoring, or alarming systems. ▪ Affect the safe operation of an SSC. Provide supporting comments (as required):

Part 5: Attest to compliant completion, review and approve.

5.1 As the Software Owner (SO), I have determined the software type, category, and as appropriate, SRL, in accordance with P1040, *Software Quality Management* and the instructions associated with this form. Provide SO Name/Z No. (print): David M. Wayne / 113674	Signature	Date 11-23-15
5.2 As the Software Owner Responsible Line Manager (SO RLM), I have reviewed and approve the determination of the software type, category and, as appropriate, SRL for the software as described on this form. Provide SO RLM Name/Z No. (print): Kent Abney / 101670	Signature	Date 11/23/15
5.3 As the ☑ Facility Design Authority Representative (FDAR) for my representative facilities, or, as the ☐ LANL Design Authority (DA), I have reviewed and approve the determination of the software type, category and, as appropriate, SRL for the software as described on this form. Check one and provide Name/Z No. (print): Richard Hemphill 083862 **Note:** The Responsible Associate Director (RAD) is authorized to review and approve Form 2033s (rather than the FDAR or DA) for software applications where, as determined by the FDAR or DA, the FDAR or DA does not have the knowledge and/or a reasonable connection to the software.	Signature	Date 12/3/15

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