We put science to work.™

Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

A U.S. DEPARTMENT OF ENERGY NATIONAL LABORATORY • SAVANNAH RIVER SITE • AIKEN, SC

Analysis of Waste Isolation Pilot Plant (WIPP) Underground and MgO Samples by the Savannah River National Laboratory (SRNL)

H. M. Ajo

L. W. Brown

C. J. Coleman

S. L. Crump

C. C. Diprete

D. P. Diprete

A. A. Ekechukwu

F. Fondeur

C. M Gregory

M. A. Jones

D. M. Missimer

P. E. O'Rourke

T. L. White

J. E. Young

December 2014

SRNL-STI-2014-00617

SRNL.DOE.GOV

DISCLAIMER

This work was prepared under an agreement with and funded by the U.S. Government. Neither the U.S. Government or its employees, nor any of its contractors, subcontractors or their employees, makes any express or implied:

1. warranty or assumes any legal liability for the accuracy, completeness, or for the use or results of such use of any information, product, or process disclosed; or
2. representation that such use or results of such use would not infringe privately owned rights; or
3. endorsement or recommendation of any specifically identified commercial product, process, or service.

Any views and opinions of authors expressed in this work do not necessarily state or reflect those of the United States Government, or its contractors, or subcontractors.

Printed in the United States of America

Prepared for
U.S. Department of Energy

SRNL-STI-2014-00617
Revision 0

Keywords: *WIPP, Analysis*

Retention: *Permanent*

Analysis of Waste Isolation Pilot Plant (WIPP) Underground and MgO Samples by the Savannah River National Laboratory (SRNL)

December 2014

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

REVIEWS AND APPROVALS

COORDINATING AUTHOR:

5/7/2015

J. E. Young
Analytical Development

Date

APPROVAL:

5/7/2015

C. M. Gregory
Analytical Development

Date

5/7/15

M. J. Barnes
Analytical Development

Date

EXECUTIVE SUMMARY

Analysis of the recent WIPP samples are summarized in this report

WIPP Cam Filters 4, 6, 9 (3, 7, 11 were analyzed with FAS-118 in a separate campaign)
WIPP Drum Lip R16 C4
WIPP Standard Waste Box R15 C5
WIPP MgO R16 C2
WIPP MgO R16 C4
WIPP MgO R16 C6
LANL swipes of parent drum
LANL parent drum debris
LANL parent drum IAEA Swipe
Unused “undeployed” Swheat
Unused “undeployed” MgO
Masselin cloth “smears”

Analysis showed that the MgO samples were very pure with low carbonate and water content. Other samples showed the expected dominant presence of Mg, Na and Pb.

Parent drum debris sample was mildly acidic.

Interpretation of results is not provided in this document, but rather to present and preserve the analytical work performed. The WIPP Technical Analysis Team is responsible for result interpretation which will be written separately.

TABLE OF CONTENTS

LIST OF ABBREVIATIONS vii

1.0 Introduction 1

2.0 Experimental Procedure 1

3.0 Results and Discussion 1

3.1 Dissolution
3.2 Elemental – ICP-ES
3.3 Elemental – ICP-MS
3.4 Anions – Ion Chromatography
3.5 Wet Chemistry – pH, acidity and alkalinity
3.6 Organics
3.7 Electron Microscopy
3.8 X-ray Fluorescence
3.9 X-ray Diffraction
3.10 Radiochemistry

Attachments 46

Attachment 1 - Data Table
Attachment 2 - Sample ID and Chain of Custody Cross Reference

LIST OF ABREVIATIONS

SRNL	Savannah River National Laboratory
FTIR	Fourier-Transformed Infrared Spectroscopy
KBr	Potassium Bromide
TEA	Triethanolamine
GC-MS	Gas Chromatography – Mass Spectrometry
ICP-ES	Inductively Coupled Plasma – Emission Spectroscopy
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry
SEM	Scanning Electron Microscopy
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

1.0 Introduction

The Savannah River National Laboratory (SRNL) received a series of high priority samples from the Waste Isolation Pilot Plant (WIPP) in New Mexico beginning after the detection of a radiological release on February 14, 2014. The samples were associated with a drum breach in Panel 7 Room 7 at WIPP and consisted of Masselin Cloth swipes, Radiological Control Operations (RCO) smears (wipes), Fixed Air Sample (FAS) filter paper, and Constant Air Monitor (CAM) filter papers in a cartridge, bulk samples from the lip of 16-4, bulk samples (2) from 15-5 and MgO samples from 16-2, 16-4 and 16-6.

The samples were received and analyzed by SRNL's Analytical Development (AD) Section.

2.0 Experimental Procedure

Instrumental procedures are described within the Results and Discussions section for the individual analyses.

3.0 Results and Discussions

Analytical results of the recent WIPP samples are summarized in the section below, and expanded with analytical data on the attached set of data tables.

3.1 Dissolutions

WIPP Samples Dissolution Methods

Acid dissolutions were carried out in sealed Teflon pressure vessels at a temperature of 115°C using a mixture of 4mL concentrated HNO_3, 2 mL concentrated HF, and 2 mL concentrated HCL for a period of at least six hours. If solids remained after the hot acid treatment they were removed by filtration and washed with de-ionized water. The filtrate and wash solutions were combined and diluted to 50 mL with de-ionized water.

Sodium peroxide fusions were carried out in zirconium crucibles with 2g of sodium peroxide at a temperature of 675°C for 10 minutes. The flux residue after cooling was dissolved with de-ionized water and concentrated HNO_3 and diluted to 100 mL with de-ionized water to yield clear solutions in each case. The higher dilution volume used for the sodium peroxide fusions was to ensure complete dissolution and removal of the flux residue from the crucible and to lower the sodium ion concentration prior to spectroscopic analysis.

De-ionized water leaches were carried out with two different methods: (1) Hot water leach in which the sample was heated in a Teflon pressure vessel overnight at 115° C. The solids were removed by filtration and washed with de-ionized water. The filtrate and wash solutions were combined and diluted to 25 mL with de-ionized water; and (2) Room temperature leach in which the sample was leached for four days in a Teflon centrifuge tube. The solids were removed by filtration and washed with de-ionized water. The filtrate and wash solutions were combined and diluted to 25 mL with de-ionized water.

Summary of WIPP Sample Digestion Data and Methods			
Sample I.D.	**Sample wt. (g)**	**Final volume of Digestion (mL)**	**Method**
300313030 R-14C-6 IC	**0.9598**	**25**	**Hot Distilled Water Leach-overnight heating at 115 ° C in sealed Teflon vessel; filter, wash solids, and dilute filtrate to 25 mL**
300313031 Undeployed MgO	**1.0378**	**25**	**Hot Distilled Water Leach overnight heating at 115 ° C in sealed Teflon vessel; filter, wash solids, and dilute filtrate to 25 mL**
300313598 CAM Filter 4, Section MA	**0.0284**	**50**	**Hot Mixed Acid-overnight heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF**
300313599 CAM Filter 6, Section MA	**0.0249**	**50**	**Hot Mixed Acid-overnight heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF**
300313600 CAM Filter 9, Section MA	**0.0328**	**50**	**Hot Mixed Acid-overnight heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF**
300313602 CAM Filter 4, Section PF	**0.0247**	**100**	**Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300313603 CAM Filter 6, Section PF	**0.0365**	**100**	**Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300313604 CAM Filter 9, Section PF	**0.0379**	**100**	**Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300313726 69120-A-MA	**0.5218**	**50**	**Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF**
300313729 69120-H-MA	**0.5148**	**50**	**Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF**
300313733 69120-A-PF	**0.4817**	**100**	**Ramp up temperature then Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300313734 69120-H-PF	**0.4850**	**100**	**Ramp up temperature then Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300313736 69120-B-MA	**0.6968**	**50**	**Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4**

			mL HNO_3/2 mL HCl/2mL HF. Let stand at room temp for 4 days. Solids removed by filtration and filtrate and wash solutions diluted to 50 mL
300313741 69120-B-PF	**0.4317**	**100**	**Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300313743 69120-B-IC	**0.6901**	**20**	**Room temperature leach in 20 mL DI water for 4 days; filter. Solution used for both IC and titration methods.**
300313744 69120-B-TITR	**0.6901**	**20**	**Room temperature leach in 20 mL DI water for 4 days; filter. Solution used for both IC and titration methods.**
300313910 Sample-1-MA	**0.1855**	**50**	**Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL.**
300313911 Sample-2-MA	**0.1863**	**50**	**Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL.**
300313924 Sample- 2-PF	**0.2168**	**100**	**Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300314333 Unused Swheat-PF	**0.3012**	**100**	**Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300314335 Unused Swheat-MA	**0.3930**	**100**	**Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 100 mL.**
300314375 WIPP #3 (spare 15-5)	**0.1264**	**50**	**Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Let stand at room temp for 4 days. Solids removed by filtration and filtrate and wash solutions diluted to 50 mL**

300314377 WIPP #3 (spare-15-5)	**0.0779**	**100**	**Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3**
300314454 WIPP Sample #2	**0.1366**	**20**	**Room temperature leach in 20 mL DI water for 4 days; filter. Solution used for both IC and titration methods.**

3.2 Elemental – ICP-ES

Inductively Coupled Plasma – Emission Spectrometry

ICP-ES General: The inductively coupled plasma spectrometer used in this laboratory (Teledyne-Leeman Prodigy-XP) provides multiple element analysis by measuring the light emitted when a liquid sample is desolvated, atomized, and excited by argon plasma. To accomplish the analysis, a liquid sample is introduced into a nebulizer where a small percentage, 2 to 4 percent, is turned into an aerosol. The aerosol is carried by an argon stream into the inductively coupled argon plasma (750-1500 watts) with a maximum temperature of 10,000 K. The aerosol is converted to atoms and ions which emit energy in the form of light.

The light emitted is dispersed by an echelle grating. To separate the orders and create a two-dimensional diffraction pattern, the echelle grating is combined with a cross-dispersing element. A Schmidt Cross Disperser is used in the ultraviolet range (165-375 nm) and a prism is used in the visible range (375-782 nm).

Two Segmented-Array Charge-Coupled-Device Detectors (SCD) are used. The detectors are made of a silicon chip that consists of linear subarrays comprised of pixels. There are a total of 224 subarrays on each detector and each subarray covers a spectral range of 0.1 to 0.4 nm. Each subarray has a photosensitive area, storage area and the readout register. Photons strike the photosensitive area and are converted to photoelectrons. The photoelectrons are pulled away from the surface, moved to the storage register and stored as an electrical charge. The integration time is the length of time these electrons are allowed to accumulate in the storage register. When the integration is complete, the charge in the storage register is transferred into the output register by a charge in the electrical potential. The charge is then moved to the output electronics and sent to the signal process electronics.

Multiple emission lines are used to quantitate each element. During data analysis, those emission lines that show interference by the sample matrix are eliminated for the analysis, and the remaining calculated concentrations are averaged. Emission line interference may be positive or negative (suppression or elevated background subtraction).

The ICP-ES is limited to aqueous samples or solid samples that can be dissolved in acid. The ICP-ES and ICP-Mass Spectrometry (ICP-MS) are complimentary techniques. The –ES method is generally better for lighter elements and the –MS method is better for heavier elements.

ICP-ES Task Specific: Unusual behavior was noted in the Na/Mg ratios were inverted, while some of the more minor component ratios remained relatively constant. No other abnormalities were noted.

3.3 Elemental – ICP-MS

Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

The Inductively Coupled Plasma Mass Spectrometer provides multi-element analyses of aqueous solutions based on the measurement of atomic species from their ions created in the plasma. The high temperature plasma (~10,000 K) ionizes metallic species, and the ions are separated through a quadrupole mass filter. The detector (electron multiplier) measures the signal for calibration and analysis at ppb levels with a 20 % method uncertainty.

Seventeen digested samples were analyzed by Inductively Coupled Plasma Mass Spectrometry (ICPMS). The samples are listed below:

- CAM Filter 4 Mixed Acid 300313598 and Peroxide Fusion 300313602,
- CAM Filter 6 Mixed Acid 300313599 and Peroxide Fusion 300313603,
- CAM Filter 9 Mixed Acid 300313600 and Peroxide Fusion 300313604,
- LANL Parent Drum IAEA Swipes 300313726,729 and Peroxide Fusion 300313733,734,
- WIPP Sample #1 R16 C4 Mixed Acid 300313910,
- WIPP Sample #2 R15 C5 Mixed Acid 300313911 and Peroxide Fusion 300313924,
- LANL Parent Drum Debris Mixed Acid 300313736 and Peroxide Fusion 300313741, and
- WIPP Sample #3 R15 C5 Mixed Acid 300314375 and Peroxide Fusion 300314377.

Analysis was performed on an Agilent 7700x ICPMS, equipped with a He gas collision cell for polyatomic interference discrimination. The mass spectrometer was mass calibrated and tuned before analysis using Procedure L16.1 ADS-1578, Inductively Coupled Plasma – Mass Spectrometer Elemental and Isotopic Analysis for Aqueous Liquid Samples Agilent 7700x. The digested samples were diluted in 2% nitric acid and spiked with internal standard. Instrument dilution factors were prepared such that the concentration of the analytes did not exceed the concentration of the maximum calibration standard (25 ppb) at the discretion of the task supervisor and to achieve acceptable internal standard recoveries in the 80-120% range. The following instrument dilution factors were performed on the digested samples: 100x for 300313598-600,602-604, 10x for 300313726,729, 100x for 300313733,734, 10x for 300313910, 100x for 300313911,924, 1000x for 300313736,741, 100x for 300314375, and 500x for 300314377.

The routine method on the ICPMS for these samples covered the isotopes with a mass-to-charge ratio (m/z) at 51, 59, 69, 71, 78, and most masses in the 84 – 248 m/z range. The concentration at each mass was measured; natural abundance corrections for elemental concentrations were not calculated due to potential fission products in the radiological samples (e.g., Cs-137 interferes with natural Ba-137). For this project, elemental concentrations were noted only when a natural abundance of the element's isotopes was measured. For the CAM filters, the customer was mostly interested in the actinides present, Bi, W, and La from the glovebox glove. For the IAEA swipes and LANL Parent Drum Debris solids, the customer was mostly interested in the radioactive isotopes but also in the materials in the drum and how they compared to the known content of the drum. For the Sample #1 R16 C4 (Lip sample), the customer was interested in the elements that were in the highest concentration and if they were similar to known contents in the drum. For Sample #2 and Sample #3 R15 C5, the customer was mostly interested in Bi, W, and La from the glovebox glove. The mixed acid and the peroxide fusion digested samples contained different concentrations for the same isotopes. The peroxide fusion was a complete digestion (residue was noted for the mixed acid digestions) and should be used to provide a better quantitation of the samples' contents.

The CAM Filter 4 Mixed Acid contained elemental Pb at 971 µg/filter, 0.260 µg/filter m/z 235 and 4.92 µg/filter m/z 238, 9.59 µg/filter m/z 239, 6.04 µg/filter m/z 181, 3.10 µg/filter m/z 71, and trace levels (< 3 µg/filter) at the other masses with a typical reporting limit (RL) of < 0.2 µg/filter. The CAM Filter 4 Peroxide Fusion contained elemental Pb at 1770 µg/filter, elemental Sb at 14.5 µg/filter, 0.561 µg/filter m/z 235 and 10.3 µg/filter m/z 238, 6.26 µg/filter m/z 239, 6.13 µg/filter m/z 71, and trace levels (< 3 µg/filter) at the other masses with a typical RL < 0.4 µg/filter.

The CAM Filter 6 Mixed Acid contained elemental Pb at 775 µg/filter, 0.204 µg/filter m/z 235 and 4.06 µg/filter m/z 238, 3.85 µg/filter m/z 239, and trace levels (< 3 µg/filter) at the other masses with a typical RL < 0.2 µg/filter. The CAM Filter 6 Peroxide Fusion contained elemental Pb at 2400 µg/filter, elemental Sb at 24.8 µg/filter, 0.753 µg/filter m/z 235 and 14.5 µg/filter m/z 238, 9.14 µg/filter m/z 239, 8.28 µg/filter m/z 71, 3.46 µg/filter m/z 114, 3.14 µg/filter m/z 59, and trace levels (< 3 µg/filter) at the other masses with a typical RL < 0.4 µg/filter.

The CAM Filter 9 Mixed Acid contained elemental Pb at 1030 µg/filter, 0.296 µg/filter m/z 235 and 5.58 µg/filter m/z 238, 5.62 µg/filter m/z 239, 3.35 µg/filter m/z 71, and trace levels (< 3 µg/filter) at the other masses with a typical RL < 0.2 µg/filter. The CAM Filter 9 Peroxide Fusion contained elemental Pb at 2270 µg/filter, elemental Sb at 23.4 µg/filter, 0.739 µg/filter m/z 235 and 13.9 µg/filter m/z 238, 9.53 µg/filter m/z 239, 7.87 µg/filter m/z 71, 3.23 µg/filter m/z 59, 3.20 µg/filter m/z 114, and trace levels (< 3 µg/filter) at the other masses with a typical RL < 0.4 µg/filter.

The 69120_A LANL Parent Drum IAEA Swipe Mixed Acid contained elemental Pb at 372 µg/swipe, 0.194 µg/swipe m/z 235 and 4.74 µg/swipe m/z 238, 1.16 µg/swipe m/z 239, and trace levels (< 1 µg/swipe) at the other masses with a typical RL < 0.02 µg/swipe; no significant levels were noted for 69120_H. The 69120_A LANL Parent Drum IAEA Swipe Peroxide Fusion contained elemental Pb at 294 µg/swipe, 3.40 µg/swipe m/z 238, 1.06 µg/swipe m/z 239, with a typical RL < 0.4 µg/swipe; no significant levels were noted for 69120_H; the actinides (including m/z 240 and 241) were < 0.4 µg/swipe. The LANL Parent Drum Debris Mixed Acid contained elemental Pb at 8.4 wt%, 224 ppm m/z 235 and 5370 ppm m/z 238, 338 ppm m/z 69; 293 ppm m/z 71, 25.2 ppm m/z 239, 21.0 ppm m/z 236, and trace levels (< 10 ppm) at the other masses with a typical RL < 0.7 ppm. The LANL Parent Drum Debris Peroxide Fusion contained elemental Pb at 11 wt%, elemental Hf at 554 ppm, 134 ppm m/z 235 and 3490 ppm m/z 238, 553 ppm m/z 239, 494 ppm m/z 69, 424 ppm m/z 71, 148 ppm m/z 241, 53.6 ppm m/z 89, 41.8 ppm m/z 240, 28.6 ppm m/z 106, 24.8 ppm m/z 138, 16.1 ppm m/z 59, 14.7 ppm m/z 236, 14.1 ppm m/z 110, 10.7 ppm m/z 107, and trace levels (< 10 ppm) at the other masses with a typical RL < 2.3 ppm.

The Sample #1 R16 C4 (Lip sample) Mixed Acid contained elemental Ba at 39.8 ppm (although not of significant interest), 21.9 ppm m/z 51, 8.39 ppm m/z 88, 3.22 ppm m/z 181, and trace levels (< 1 ppm) at the other masses with a typical RL < 0.03 ppm. Not enough material was available to perform a peroxide fusion on Sample #1 R16 C4 (Lip sample) Peroxide Fusion.

The Sample #2 R15 C5 Mixed Acid contained elemental Pb at 0.64 wt%, 19.3 ppm m/z 235 and 311 ppm m/z 238, 36.2 ppm m/z 239, 31.7 ppm m/z 138, 14.3 m/z 181, 10.0 ppm m/z 71, and trace levels (< 10 ppm) at the other masses with a typical RL < 0.3 ppm. The Sample #2 R15 C5 Peroxide Fusion contained elemental Pb at 0.77 wt%, elemental Hf at 0.12 wt%, elemental Ga at 129 ppm, elemental Ba at 104 ppm, elemental Sn at 95.0 ppm, 19.6 ppm m/z 235 and 324 ppm m/z 238, 185 ppm m/z 239, 62.9 ppm m/z 106, 30.9 ppm m/z 110, 22.7 ppm m/z 108, 21.8 ppm m/z 107, 19.7 ppm m/z 88, 17.7 ppm m/z 241, 13.9 ppm m/z 240, 11.8 ppm m/z 181, 10.5 ppm m/z 112, and trace levels (< 10 ppm) at the other masses with a typical RL < 0.5 ppm. The Sample #3 R15 C5 Mixed Acid contained elemental Pb at 0.39

wt%, 5.12 ppm m/z 235 and 105 ppm m/z 238, and trace levels (< 10 ppm) at the other masses with a typical RL < 0.4 ppm. The Sample #3 R15 C5 Peroxide Fusion contained elemental Pb at 0.61 wt%, 8.93 ppm m/z 235 and 187 ppm m/z 238, 72.3 ppm m/z 239; 37.5 ppm m/z 71, 11.5 ppm m/z 51, and trace levels (< 10 ppm) at the other masses with a typical RL < 7 ppm.

3.4 Anions – Ion Chromatography

WIPP LANL Parent Drum solid debris (TC 66288 – 300313743) and WIPP sample #2 (R15-C5)(TC 66439 300314454)

Ion chromatography was used to determine the water-soluble anions in the solid debris from the parent drum, a Swheat sample (as a control) and the debris sample from R15 C5. The Dionex RFIC-3000 Ion Chromatography system consisted of an ASDV autosampler, a gradient pump, and a suppressed conductivity detector. Software control of the system and data acquisition was through Dionex Chromeleon v.6.8. We operate under Manual L16.1, Procedure ADS-2310 "Analysis of Ions in Solutions using a Dionex ICS3000 Ion Chromatography System". Samples are diluted with DI water to within the calibration curve range of 1 to 50 mg/L, analyzed, and reported. The samples were extracted with water as described above and analyzed by the half hour method described in Table 1.

Table 1. Half Hour IC Method

Anion Method	
Injection	50 μL
Flow rate	1.0 mL/min
Stop Time	32 min
Guard Column	IonPac AG-19 4x50 mm P/N 062887
Analytical Column	IonPac AS-19 4x250 mm P/N 062885
Suppressor	Self-Regenerating Suppressor (ASRS) 300 P/N 064554
Mobile Phase	20-40 mMol KOH Gradient
Calibration Curve (1σ % Unc.)	1 mg/L to 50 mg/L, r = >0.995 (10%)
Retention Time of Fluoride	4.2 min
Retention Time of Formate	5.0 min
Retention Time of Chloride	6.5 min
Retention Time of Nitrite	7.8 min
Retention Time of Bromide	9.8 min
Retention Time of Nitrate	11.0 min
Retention Time of Sulfate	15.1 min
Retention Time of Oxalate	17.7 min
Retention Time of Phosphate	23.5 min

Table 2. WIPP LANL parent drum solid debris

Cust ID	Fluoride	Formate	Chloride	Nitrite	Bromide	Nitrate	Phosphate	Sulfate	Oxalate
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
TC 66288-WIPP LANL Parent Drum (blank)	<290	<290	<290	<290	<1500	<290	<290	<290	<290
TC 66288-WIPP LANL Parent Drum	667	638	899	<290	<1500	126000	<290	290	2640

Figure 1 and Table 2 shows nitrate as the major analyte in addition to minor amounts of oxalate, fluoride, formate, and chloride. A small peak next to fluoride has a similar retention time as acetate as shown in Figure 2.

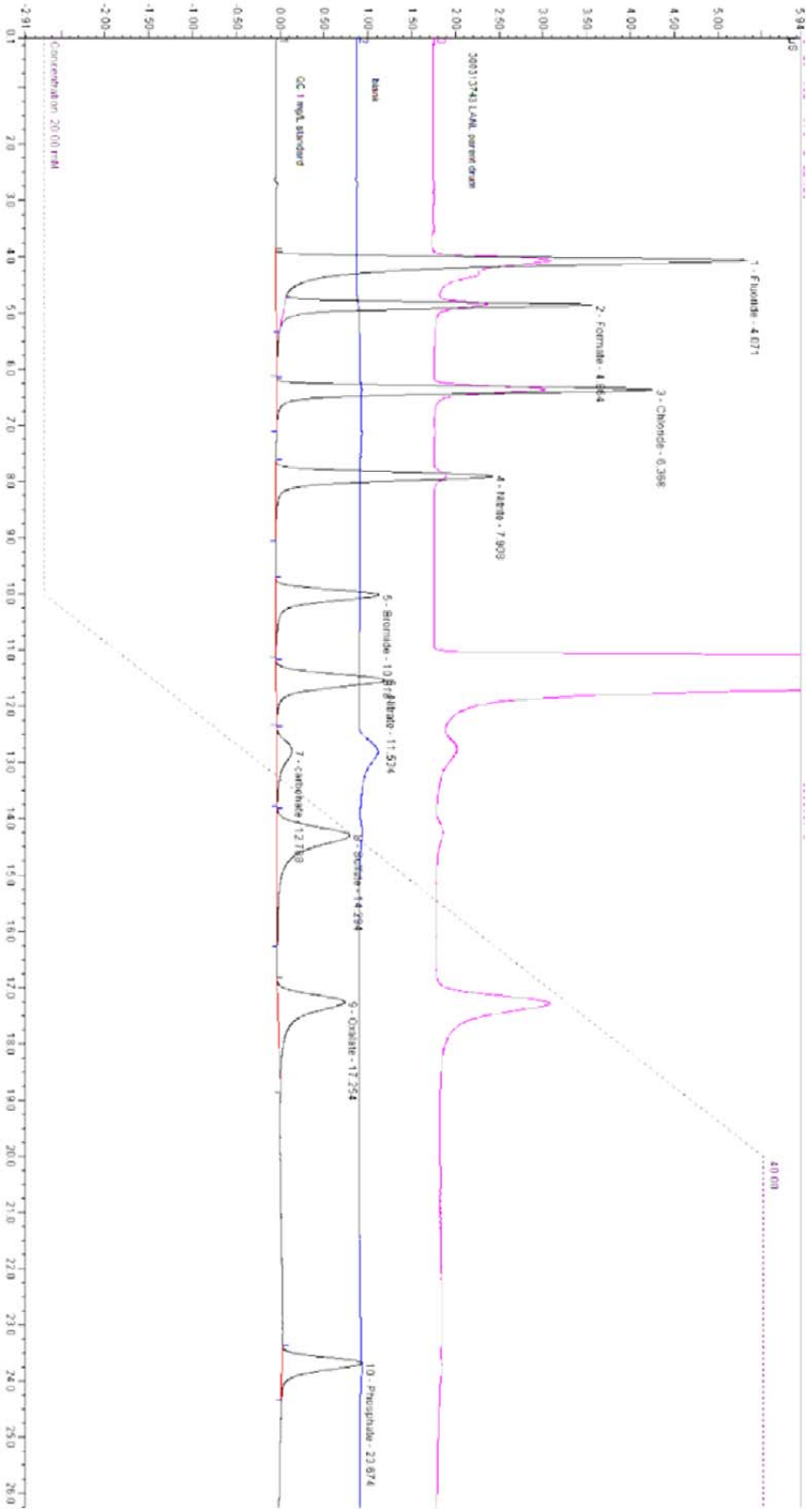

1 - Fluoride - 4.071
2 - Formate - 4.464
3 - Chloride - 6.398
4 - Nitrite - 7.909
5 - Bromide - 10.816
6 - Nitrate - 11.524
7 - Sulfate - 12.789
8 - Oxalate - 14.294
9 - Oxalate - 17.254
10 - Phosphate - 23.974
40.00
GC 1 mg/L standard
Concentration: 20.00 mM

Figure 1. TC 66288 WIPP LANL parent drum solid debris water leachate, blank, and 1 mg/L IC standard.

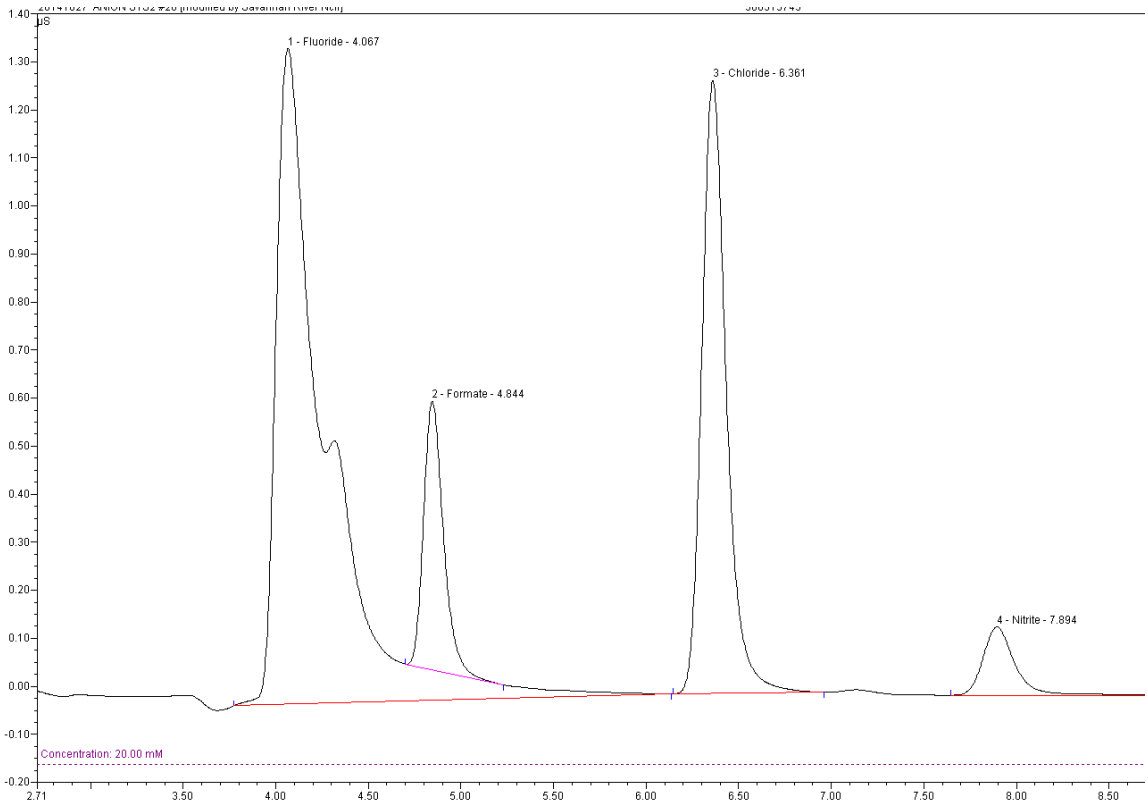


Figure 2. TC 66288 WIPP LANL parent drum solid debris water leachate showing a peak at 4.30 min with a similar retention time to acetate.

WIPP sample #2 (R15-C5) shown in Figure 3 and Table 3 shows nitrate, chloride, nitrite, and oxalate as the major analytes in addition to minor amounts of sulfate and phosphate. A large carbonate peak was observed in the chromatogram. A small peak next to fluoride has a similar retention time as acetate as shown in Figure 4.

Table 3: TC 66439 WIPP sample #2 (R15-C5)

Cust ID	Fluoride	Formate	Chloride	Nitrite	Bromide	Nitrate	Phosphate	Sulfate	Oxalate
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
TC 66439-WIPP #2(R15-C5)water leach blank	<1460	<1460	<1460	<1460	<7300	<1460	<1460	<1460	<1460
TC 66439-WIPP #2(R15-C5)water leach	<1460	<1460	13100	13100	<7300	34600	1610	3100	13100

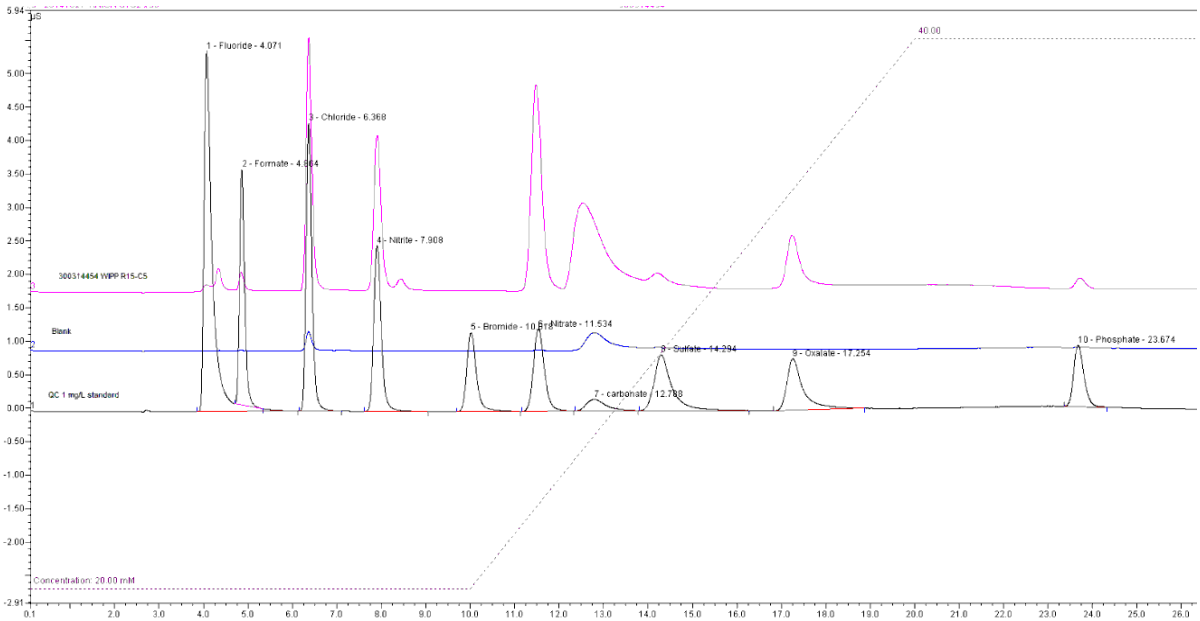


Figure 3: TC 66439 WIPP sample #2 (R15-C5) water leach, blank, and 1 mg/L IC standard.

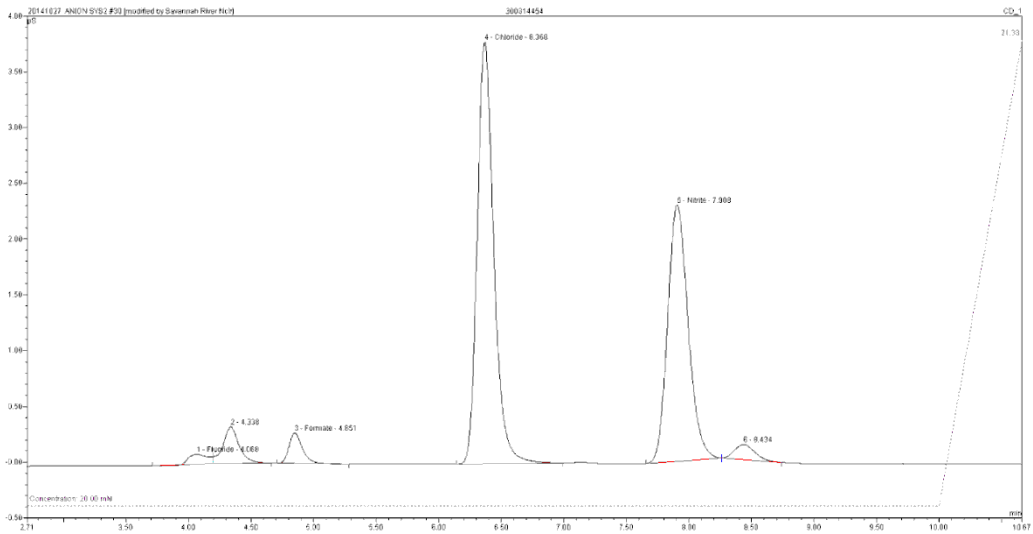


Figure 4: TC 66439 WIPP sample #2 (R15-C5) water leachate showing a peak at 4.3 min with a similar retention time to acetate.

WIPP sample #2 (R15-C5) with Acetate Standard and Swheat Leachate (TC 66523 300314962)

Sample 300314454 (0.1366 g; 4 days) and Swheat (0.5014 g; 3 days) were leached in 20 mL of ambient deionized water and analyzed by the one hour method described in Table 4 to identify acetate.

Table 4. One Hour IC Method

Anion Method	
Injection	50 μL
Flow rate	1.0 mL/min
Stop Time	60 min
Guard Column	IonPac AG-19 4x50 mm P/N 062887
Analytical Column	IonPac AS-19 4x250 mm P/N 062885
Suppressor	Self-Regenerating Suppressor (ASRS) 300 P/N 064554
Mobile Phase	7-45 mMol KOH Gradient
Calibration Curve (1σ %Unc.)	1 mg/L to 50 mg/L, r = >0.995 (10%)
Retention Time of Fluoride	6.1 min
Retention Time of Acetate	6.8 min
Retention Time of Formate	7.9 min
Retention Time of Bromate	9.2 min
Retention Time of Chloride	10.6 min
Retention Time of Nitrite	13.0 min
Retention Time of Chlorate	15.0 min
Retention Time of Bromide	16.0 min
Retention Time of Nitrate	17.9 min
Retention Time of Sulfate	25.0 min
Retention Time of Oxalate	28.9 min
Retention Time of Iodide	32.9 min
Retention Time of Chromate	38.2 min
Retention Time of Phosphate	41.5 min
Retention Time of Perchlorate	50.1 min

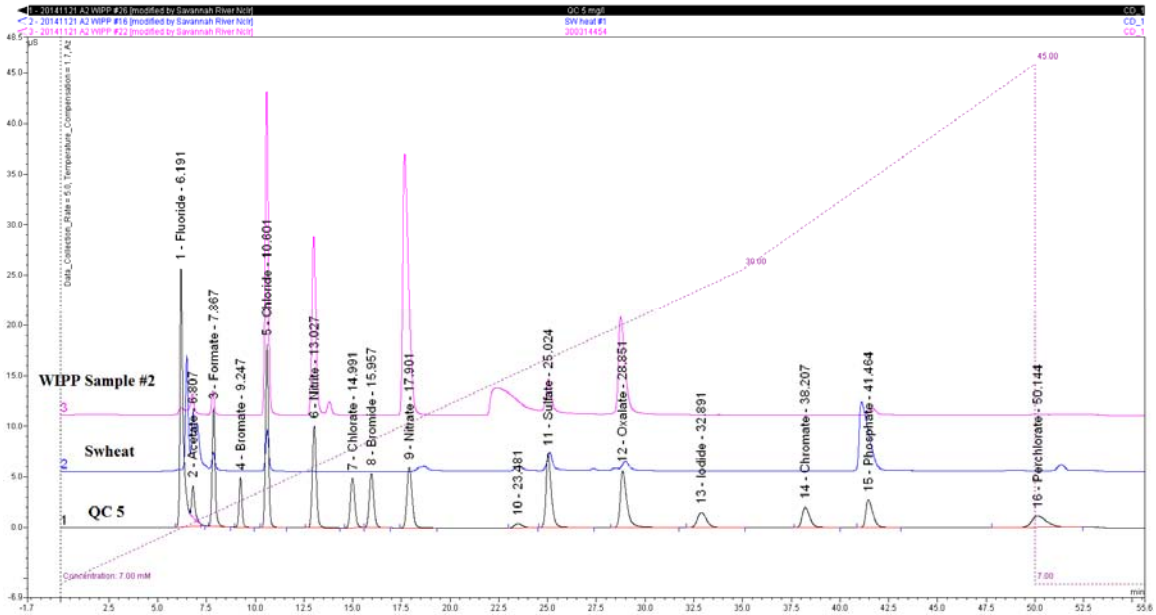


Figure 5: One hour IC method showing a QC 5 standard, Swheat leach, and WIPP sample #2. An acetate peak is present in the Swheat and WIPP Sample #2.

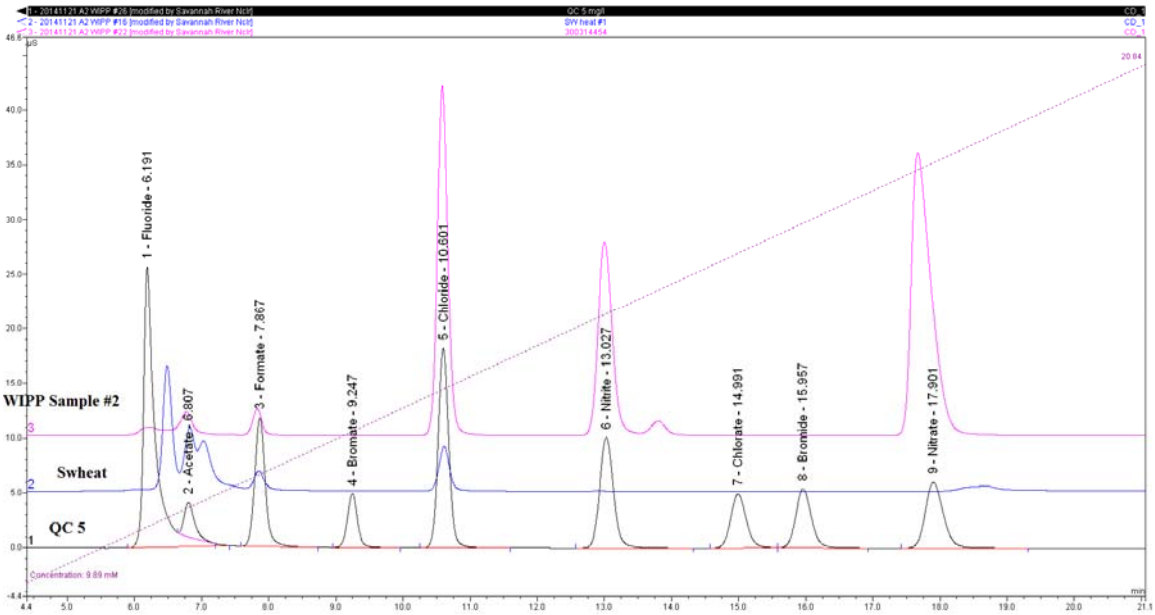


Figure 6: Expanded Figure 5 chromatogram. Note acetate in Swheat leachate and WIPP Sample #2 leachate.

Figure 5 shows an appreciable amount of carbonate at 23.4 minutes present in WIPP Sample #2 leachate. Figure 6 shows acetate in both Swheat leachate and WIPP Sample #2 leachate. The peaks were not fully resolved and thus were integrated manually to give estimated values. Adjusting for the sample weight, the acetate peak (6.8 min) is estimated to be slightly higher in the WIPP sample #2 (17500 mg/Kg) than the Swheat sample (12000 mg/Kg). In addition, an unknown peak is present at ~13.8 min in the WIPP Sample #2. Peaks boardering acetate in the Swheat sample could be due to

the presence of other monoacids such as glycolic acid. Table 5 summarizes the analytes of interest and selected analytes not found. The fluoride detection limit was raised due to interferences.

Table 5: TC 66439 WIPP sample #2 (R15-C5) Long Method

Cust ID	Fluoride	Formate	Chloride	Nitrite	Bromide	Nitrate	Phosphate	Sulfate	Oxalate
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
Swheat blank	<40	<40	<40	<40	<200	<40	<40	<40	<40
Swheat Leachate	<400	332	480	<40	<200	240	5050	435	240
TC 66439-WIPP #2(R15-C5)blk	<146	<146	<146	<146	<730	<146	<146	<146	<146
TC 66439-WIPP #2(R15-C5)	<730	1314	12800	14500	<730	35300	1610	3360	13100

3.5 Wet Chemistry – pH, Acidity, and Alkalinity

Carbonate analyses on the sample leachates were performed using an OI Analytical 1030D Total Carbon Analyzer. Inorganic carbon was measured by acidification followed by infrared detection of evolved carbon dioxide gas. Organic carbon was measured by wet chemical oxidation followed by infrared detection of the evolved carbon dioxide gas. The sample size used was 5 mL, rendering a detection limit of 0.8 ug/mL. All samples were analyzed in triplicate. Quality control standards and blanks were run before and after each set of samples.

Total acid determination was performed using a Radiometer TIM 870 auto-titration system. 2 mL aliquots of the sample leachates were run in duplicate. Total acid was determined by titrating the sample with 0.01 N sodium hydroxide to an inflection point close to pH 7. Free acid content (H+) was estimated by examining the inflection points on the titration curve. The system was calibrated prior to analysis using a 3 point calibration (pH 4, 7, and 10) protocol. Check standards (1.0 N hydrochloric acid) were run before and after each set of samples.

Total base determination was performed using a Radiometer TIM 870 auto-titration system. 2 mL aliquots of the sample leachates were run in duplicate. Total base was determined by titrating the sample with 0.1 N hydrochloric acid to an inflection point close to pH 7. Free hydroxide content (OH-) was estimated by examining the inflection points on the titration curve. The system was calibrated prior to analysis using a 3 point calibration (pH 4, 7, and 10) protocol. Check standards (2.2 N total base) were run before and after each set of samples.

pH measurements were made on the undiluted samples using a Piccolo HI 1290 Amplified electrode pH meter by HANNA Instruments. Calibration of the pH meter was verified using pH 4, 7, and 10, buffers.

3.6 Organics

Gas Chromatography / Mass Spectrometry (GC/MS) analysis was employed to identify organic compounds in the samples. Analytical separations were carried out on an Agilent 6890 gas

chromatograph, equipped with a 25 m J&W DB-5MS column, with 0.20 mm diameter and 0.33 um film thickness. Characterization and quantification was performed using an Agilent 5973 mass selective detector.

Each sample was weighed then extracted three times with methylene chloride at room temperature. The samples were then dried and weighed to quantify the amount of material extracted. Each extract was concentrated to one mL, spiked with internal standard (see Table 1 below), then analyzed by GC/MS.

Blanks were analyzed before and after each set of samples. Internal standard analytes were used for quantification of sample analytes detected.

Refer to Page 78 of the Slide Package Section, SRNL Analytical Results Summary on WIPP TAT Investigative Samples from the WIPP Underground Sampling of August 2014 and the LANL Parent Drum for a complete discussion of the Organics results.

3.7 Scanning Electron Microscopy

Samples were examined by a LEO S440 scanning electron microscope (SEM) using 30 kV accelerating voltage. The SEM is completely enclosed in a glovebox, capable of both secondary electron and backscatter electron imaging, and equipped with a liquid nitrogen cooled Oxford Instruments Si(Li) energy dispersive spectrometer (EDS). All particles were mounted on carbon sticky tape on aluminum stubs and evaporatively carbon coated. The numbers in the images, with a "+" sign in the case of a spot and with pink outlines in the case of an area, identify the location of the EDS spectrum in the image. The EDS spectra have a number in the upper right hand corner of the spectrum that corresponds to the location of the spectrum in the image. All the images in this section are backscatter images, in which the higher atomic number locations in the image appear brighter and the lower atomic number locations appear darker.

SAMPLE_1_SEM 300313918 was a solid chunk of material taken from WIPP Drum Lip 16-4 with dimensions of 1-3 millimeters. A backscatter image showing the surface of a particle and corresponding EDS spectrum are shown in Figure 1. Higher magnification images revealed that the surface of this particle appeared to be composed of many smaller particles a few microns to tens of microns in size as was commonly seen with the many other magnesium oxide samples previously examined. Contrast in backscatter imaging mode did not reveal any large compositional differences. This was confirmed by EDS results which showed that the surface of the millimeter sized particle appeared to be magnesium oxide with traces of silicon and calcium, as is typical for magnesium oxide particles, based on our rather extensive examination of such particles.

SAMPLE_2_SEM 300313919 was material from WIPP Standard Waste Box 15-5. A backscatter image and EDS spectrum are shown in Figure 2. This material was fairly heterogeneous. Particles a few microns to tens of microns in size were commonly seen. In backscatter imaging, bright particles contained mostly lead, with small amounts of aluminum, phosphorous, iron, and occasionally silicon as measured by EDS. The particles that did not appear bright in backscatter imaging and appeared to make up most of the sample appeared grey and contained mostly magnesium, oxygen, and sodium with small amounts of potassium, calcium and chlorine as measured by EDS.

ARCHIVE B SOLID DEBRIS 300313607 was from LANL parent drum debris. A backscatter image and EDS spectrum are shown in Figure 3. These solid chunks of debris were a few millimeters in size and contained predominantly lead, with small amounts of sodium, magnesium, aluminum, potassium, calcium, and iron. Lead appeared bright in backscatter imaging and appeared to be the dominant surface, although there appeared to be scattered particles darker in contrast about and upon the lead particles.

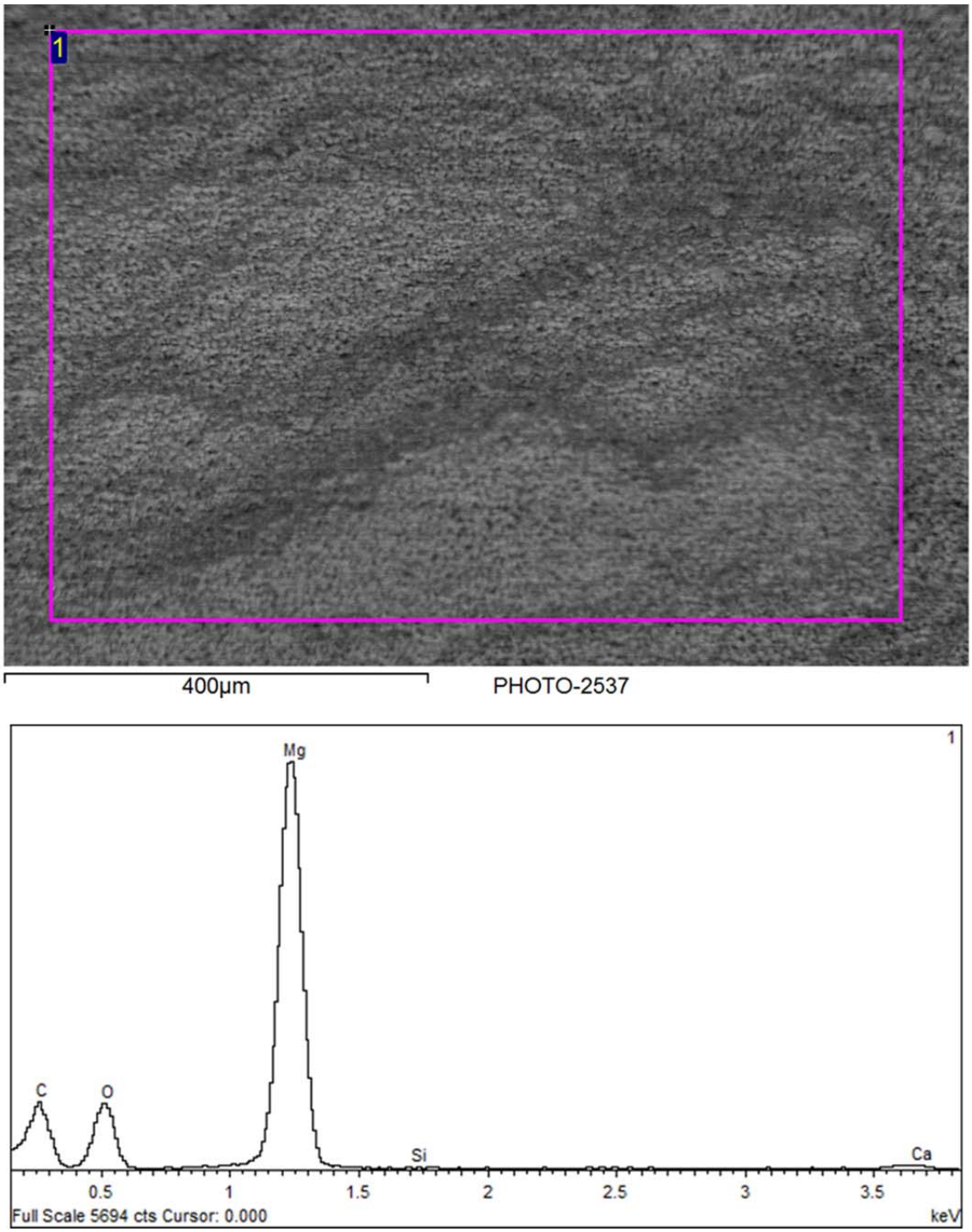
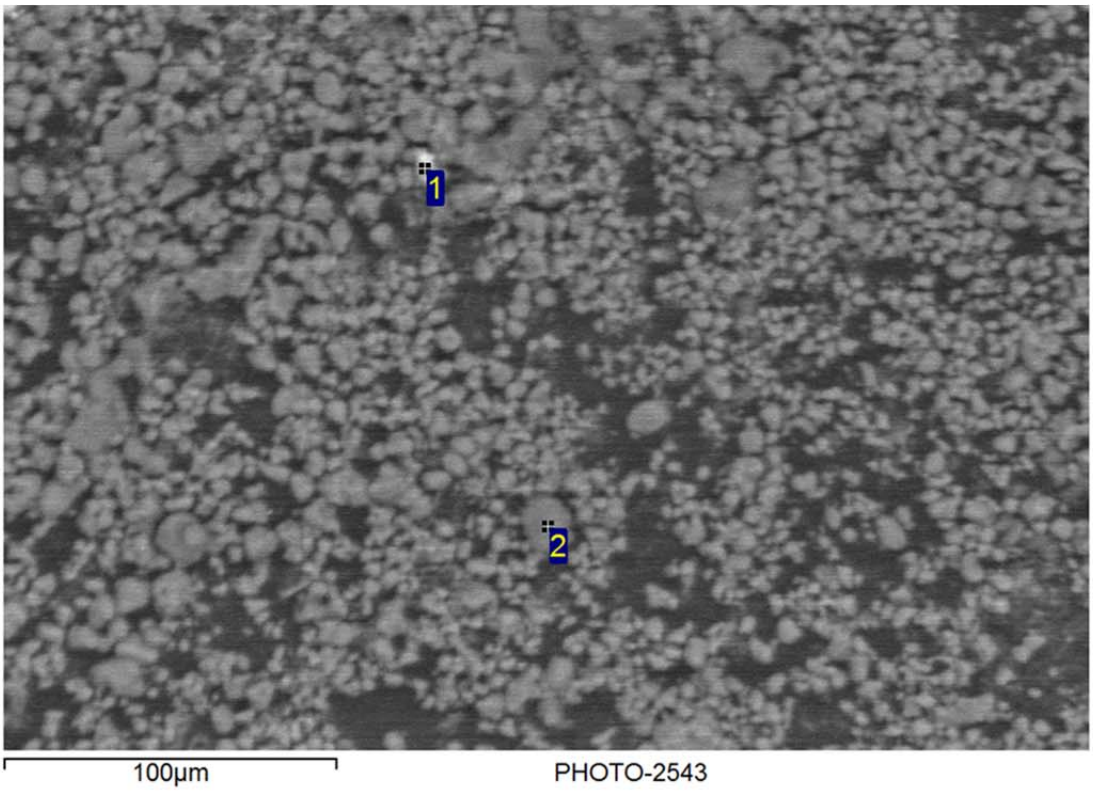


Figure 1 SAMPLE_1_SEM 300313918 (WIPP Drum Lip 16-4) backscatter image with energy dispersive spectroscopy (EDS) spectrum 1. From the corresponding EDS spectrum shown below the image, this particle appears to be magnesium oxide with traces of silicon and calcium, as is typical for magnesium oxide particles.



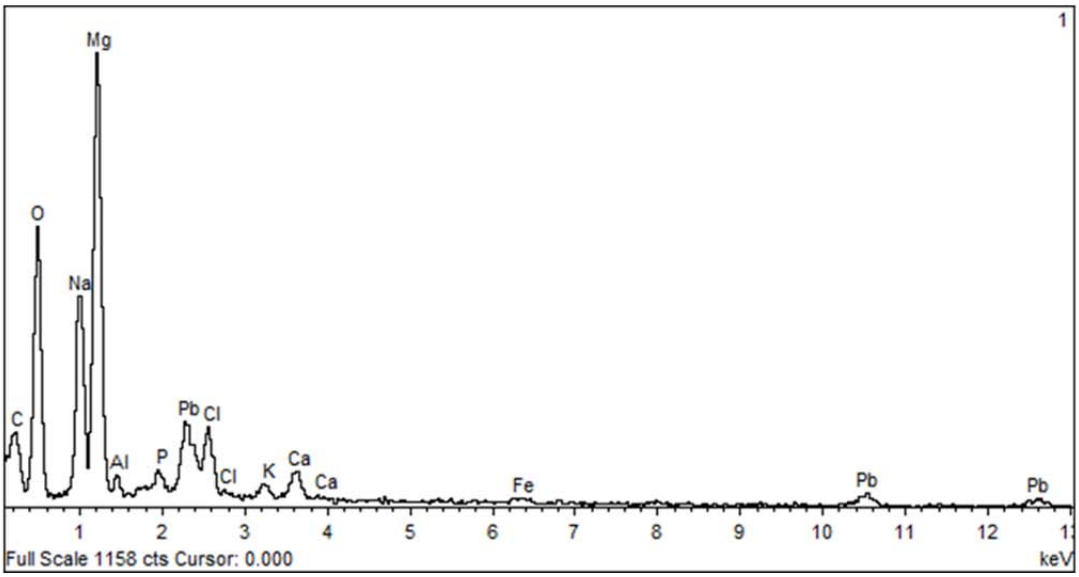
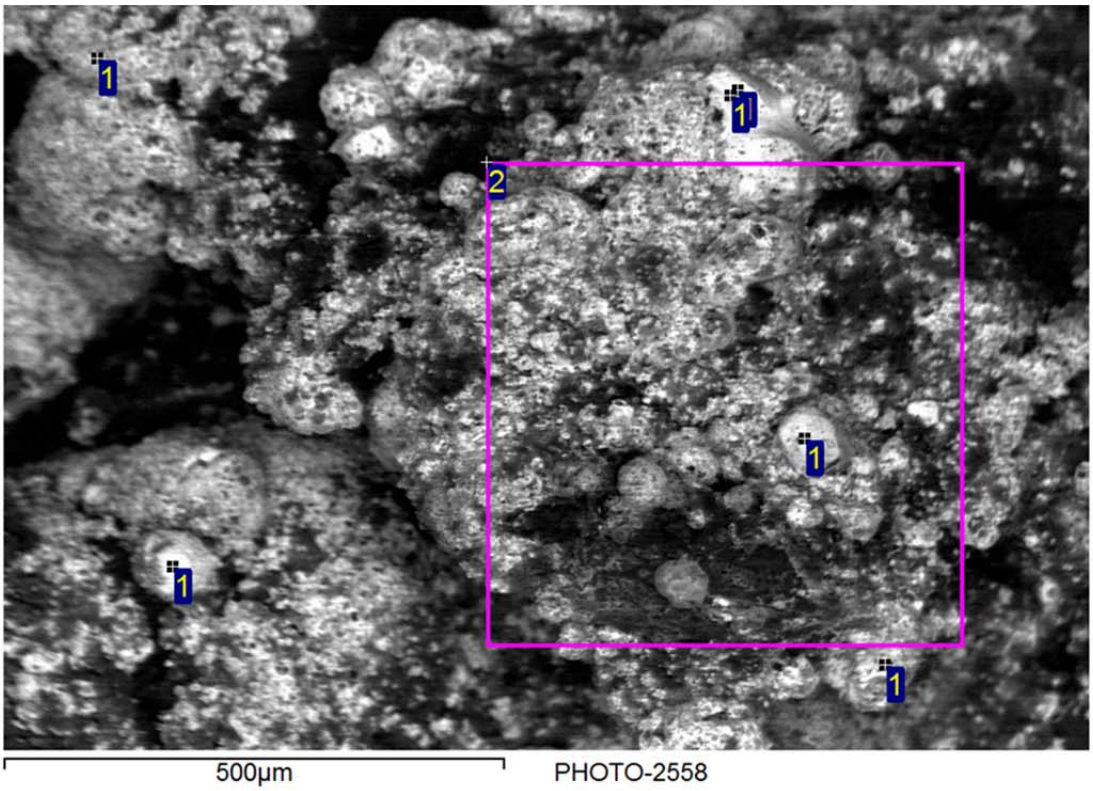


Figure 2 SAMPLE_2_SEM 300313919 (WIPP Standard Waste Box 15-5) backscatter image with energy dispersive spectroscopy (EDS) spectrum 1. In backscatter imaging, bright particles that show up contain mostly lead, with small amounts of aluminum, phosphorous, iron and occasionally silicon, as shown in the corresponding EDS spectrum 1.



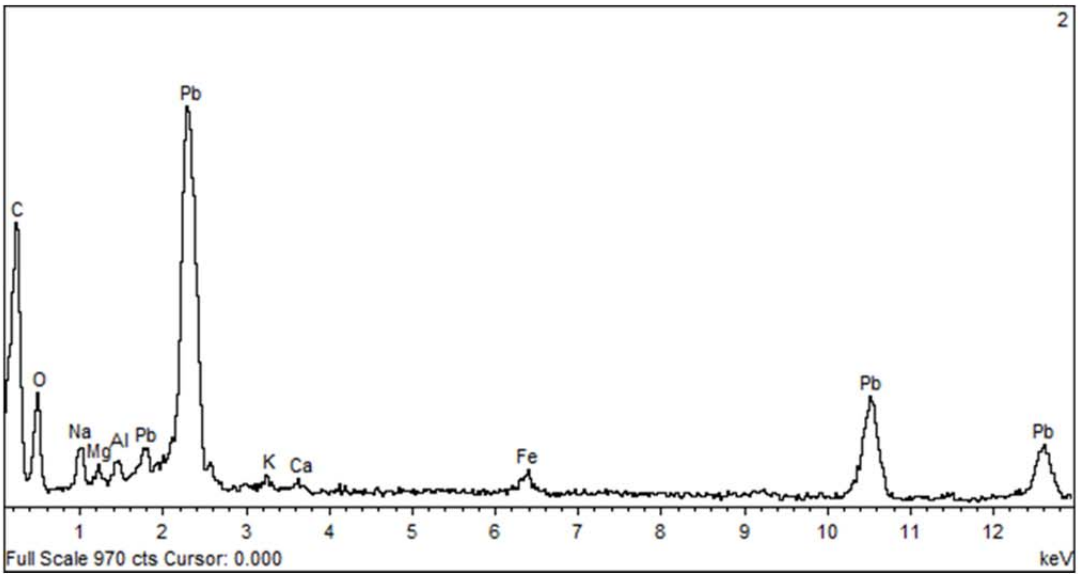


Figure 3 ARCHIVE B SOLID DEBRIS 300313607 (LANL parent drum debris) backscatter image with energy dispersive spectroscopy (EDS) spectrum 2. This chunk of debris contains predominantly lead, with small amounts of sodium, magnesium, aluminum, potassium, calcium, and iron, as seen from the EDS spectrum of area 2.

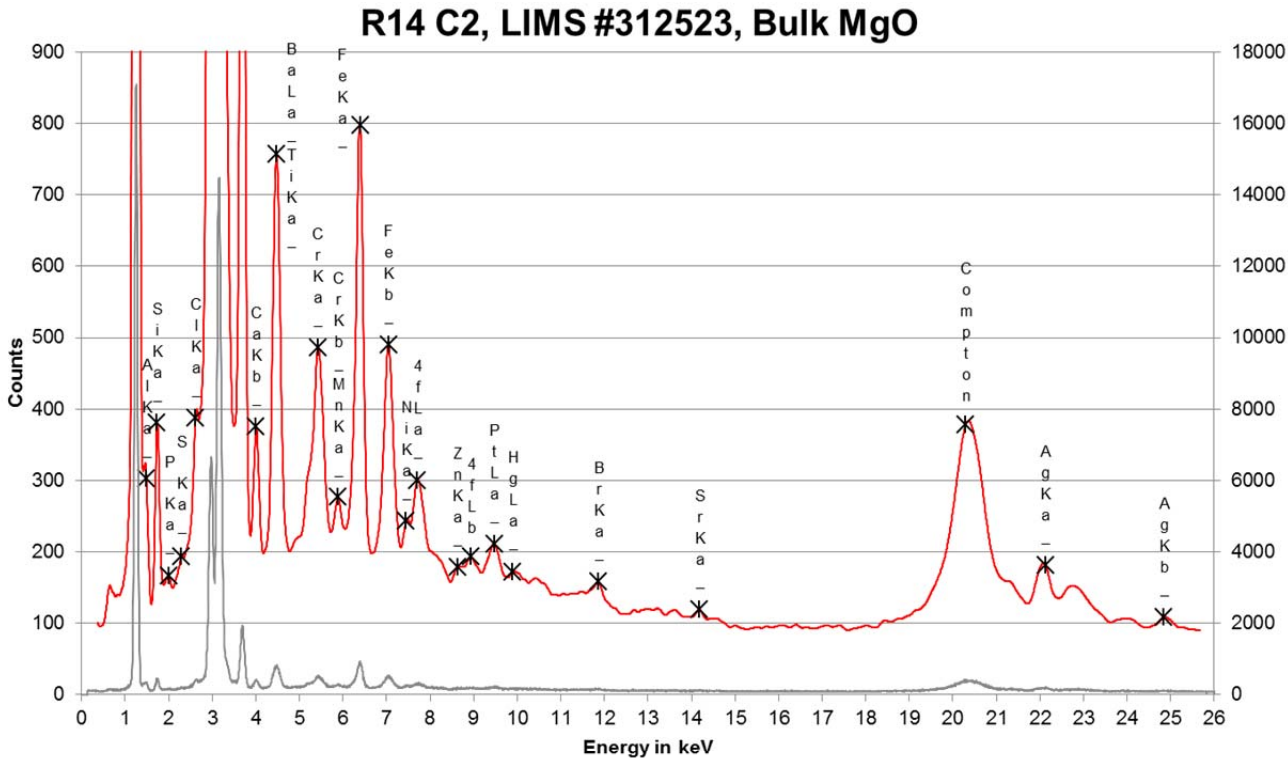
3.8 X-Ray Fluorescence

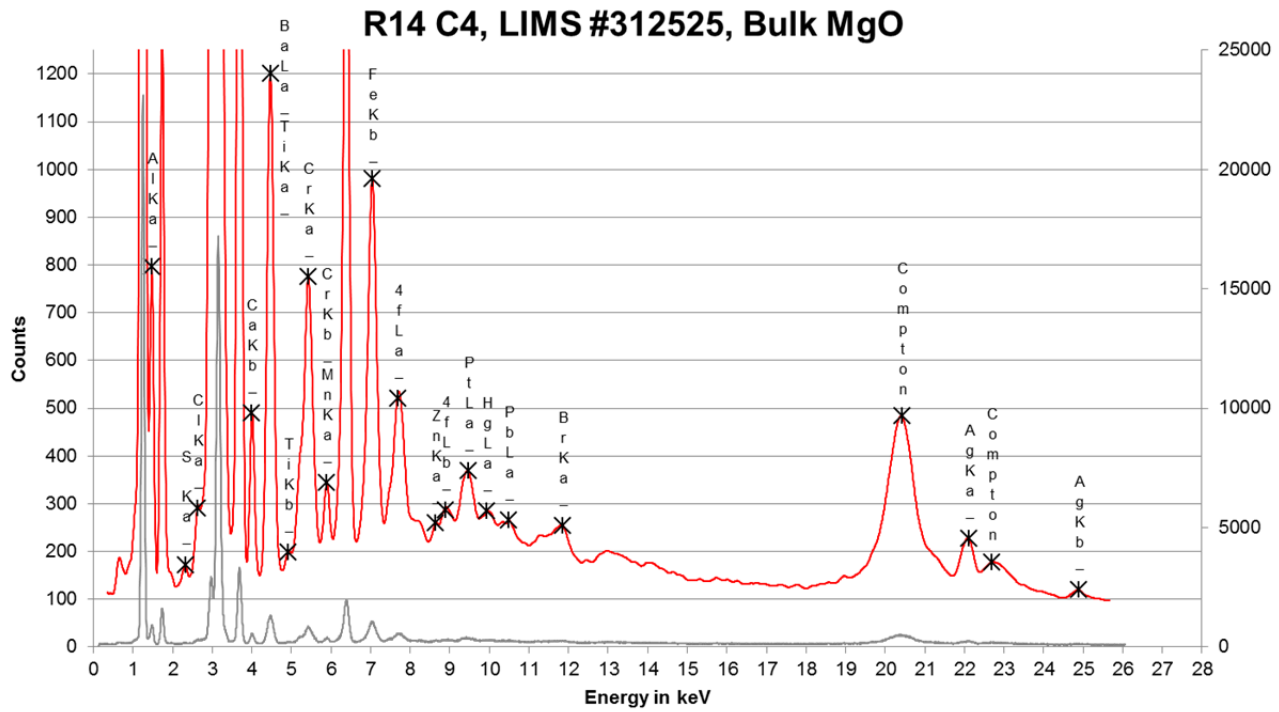
The Amptek system is a computer-controlled x-ray fluorescence spectrometer used for non-destructive elemental analyses of elements from Na (Z=11) to U (Z=92). Basically, a sample is irradiated with x-rays, which cause the loss of inner shell electrons from the constituent atoms. These inner orbital vacancies can be filled by electrons in a higher energy shell. To maintain conservation of energy, an x-ray photon, whose energy is the difference between the two levels, is produced. The energy of each emitted x-ray photon is well-defined and characteristic of the element from which it originated. The rate at which x-ray photons are emitted by a particular element is related to the concentration of the element.

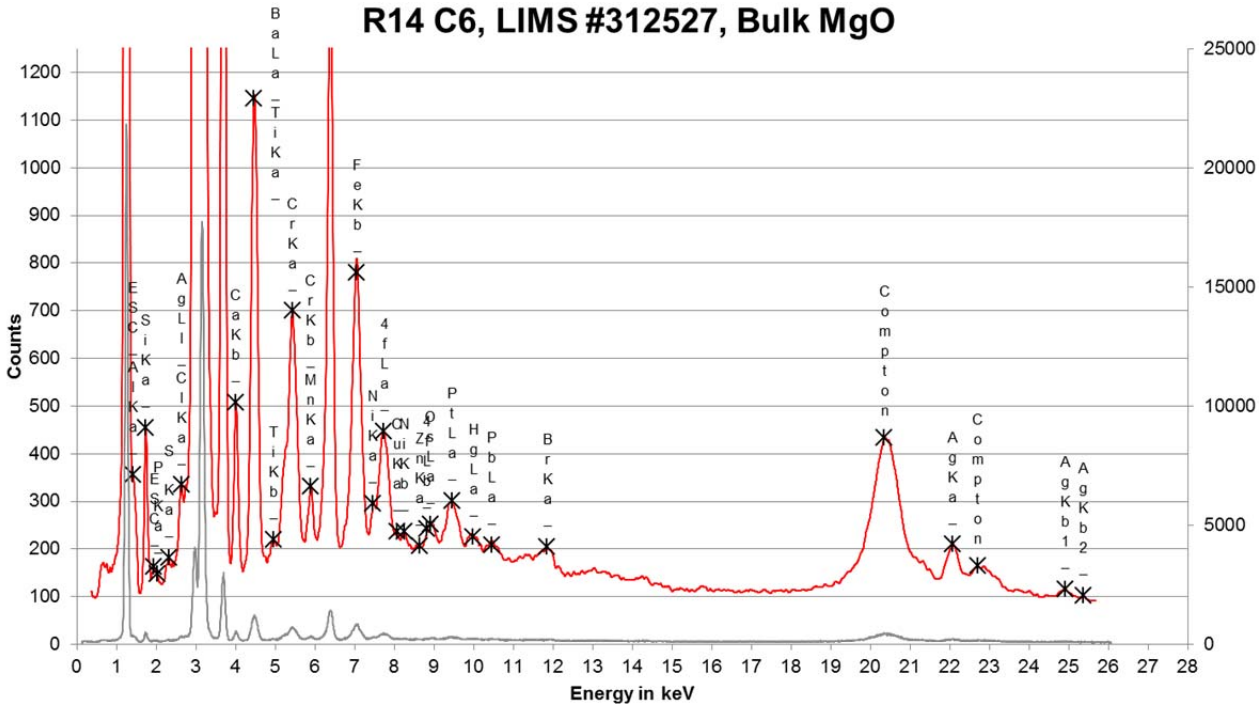
The Amptek system features an energy-dispersive lithium-drifted silicon detector and a silver transmission target miniature x-ray tube. The sample is fluoresced by the characteristic and brehmstrahlung x-rays emitted from the tube. Filters may be inserted between the x-ray tube and the collimator to reduce background and improve detection limits for some elements.

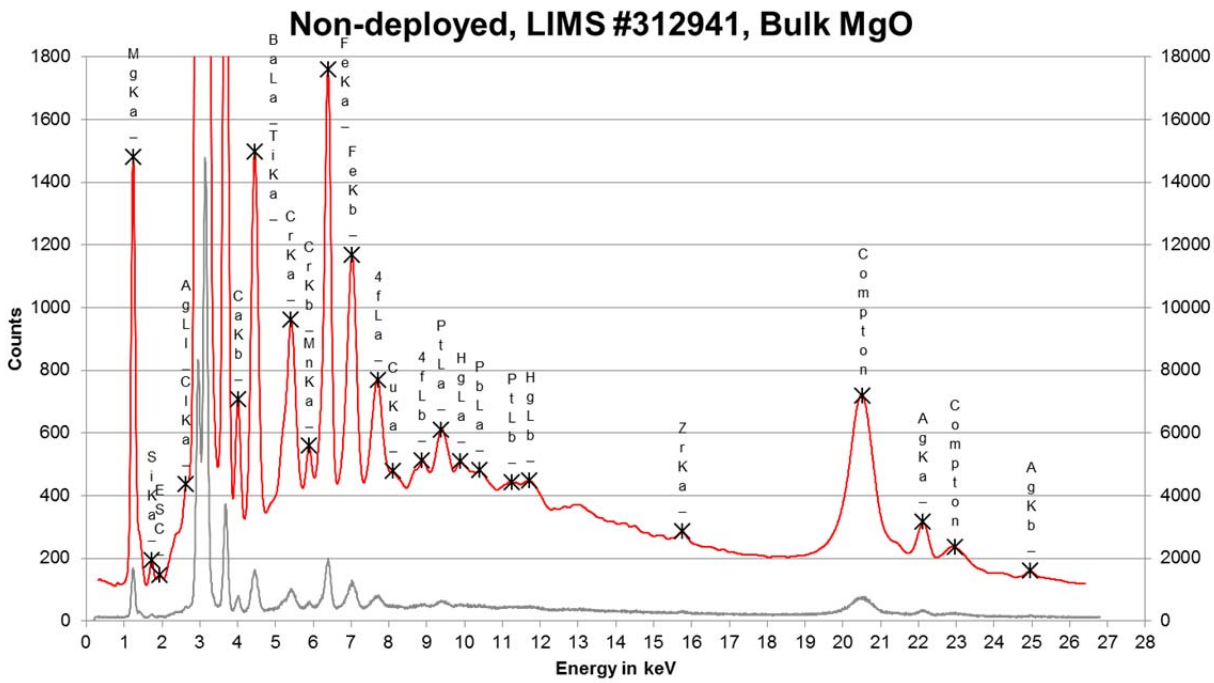
A sample, mounted onto a 2” x 2” square plate Plexiglas sample holder with a 4 µm thick ultralene film, is inserted into the SRNL designed vacuum sample chamber for radioactive samples, and positioned underneath the x-ray tube and spectrometer by a spring-loaded backing plate. A shield door, which covers the sample access area, must be slid into place and engage a microswitch before the x-ray tube can be energized.

The MgO samples were composed of powder and mm-sized pellets. Both morphologies were independently analyzed by X-ray fluorescence to determine if there were any differences. Samples (R-14 C-2 XPD {Powder, Bulk MgO}, R-14 C4 XPL {Pellet, Bulk MgO}, R14 C6 {Powder, Bulk MgO}, and MgO Ref#2 {Non Deployed MgO}; 30031523, 300312525, 30031527, and 300312941) were examined by XRF analysis. All samples show presence of 4s and 3d elements (Fe, Ca, with Ni, Cr, Ti, Zn) and trace amounts of 4f and 5d elements. All the MgO samples including the powder and the pellets showed similar elements were present.


R14 C2, LIMS #312523, Bulk MgO
Counts
Energy in keV
Al Ka
Si Ka
P Ka
S Ka
Cl Ka
Ca Kb
Ba La
Ti Ka
Cr Ka
Cr Kb
Mn Ka
Fe Ka
Fe Kb
4f NL Ka
Zn Ka
4f Lb
Pt La
Hg La
Br Ka
Sr Ka
Compton
Ag Ka
Ag Kb


R14 C4, LIMS #312525, Bulk MgO
Counts
Energy in keV
Al Ka
Cl Ka
S Ka
Ca Kb
Ba La
Ti Kb
Ti Ka
Cr Ka
Cr Kb
Mn Ka
Fe Kb
4f La
Zn Ka
4f Lb
Pt La
Hg La
Pb La
Br Ka
Compton
Ag Ka
Compton
Ag Kb

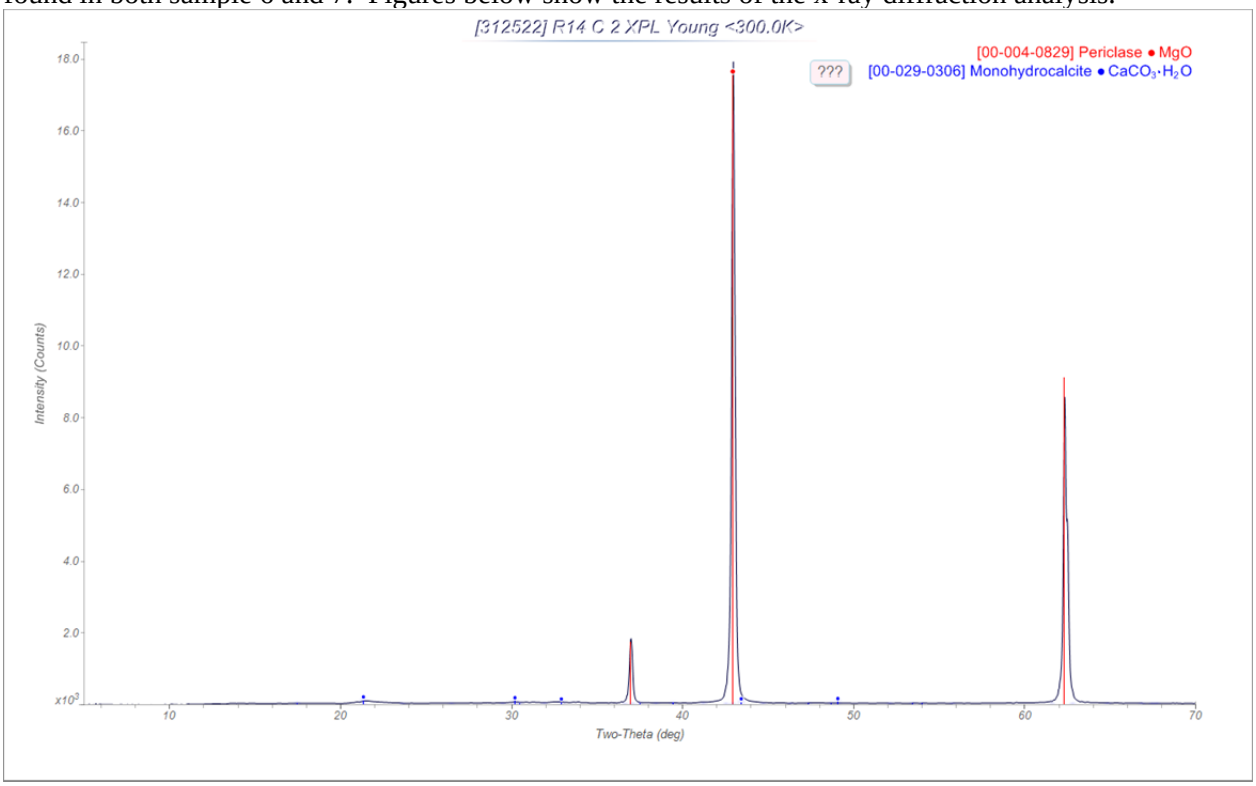


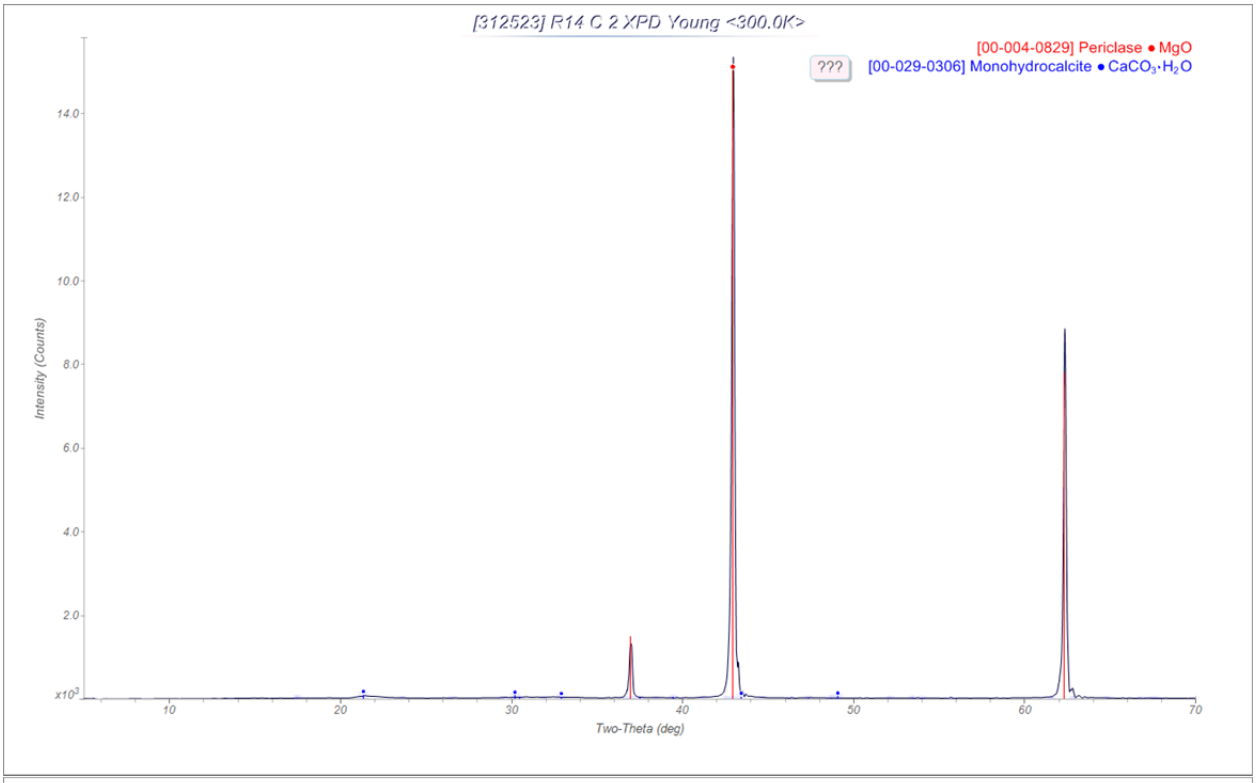


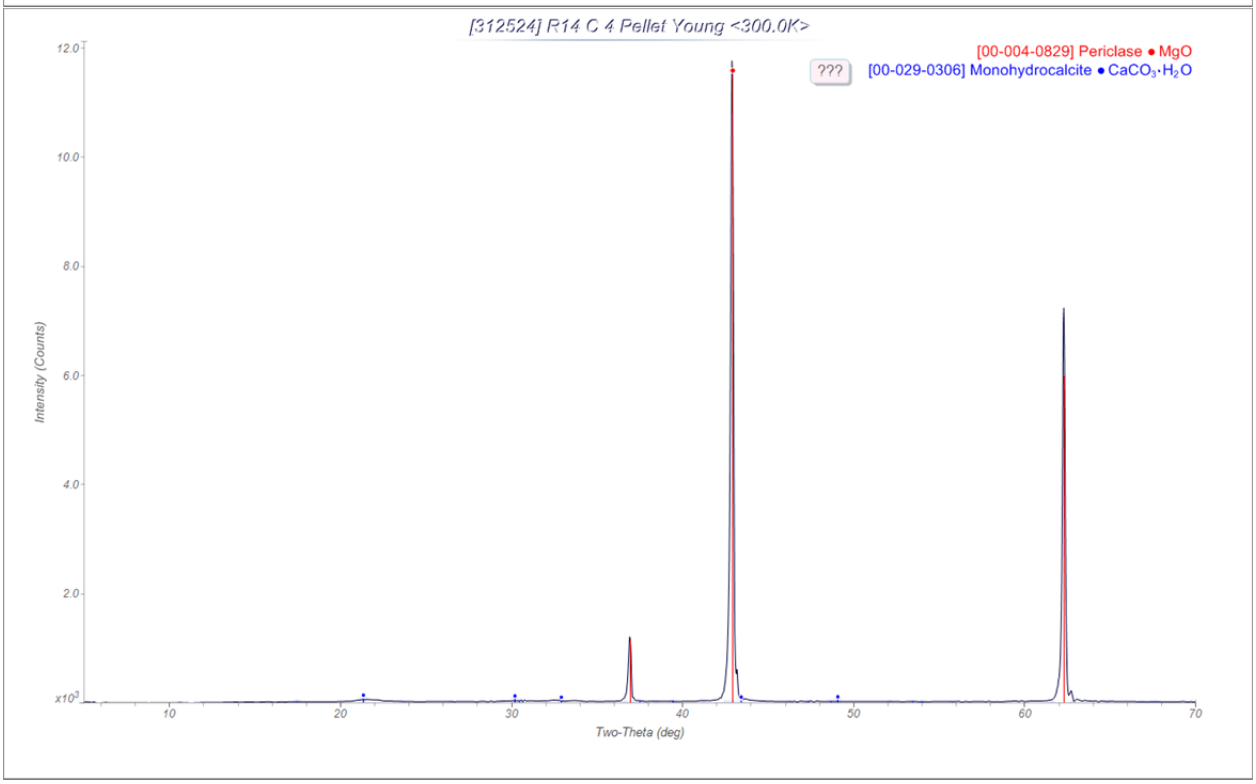
A portion of the magnesium oxide and particles taken from the area of the breached drum Panel 7, room 7, WIPP sample 2; R15 C5 (300313811), and was the spectrum below shows that lead, Pb, was detected in the sample.

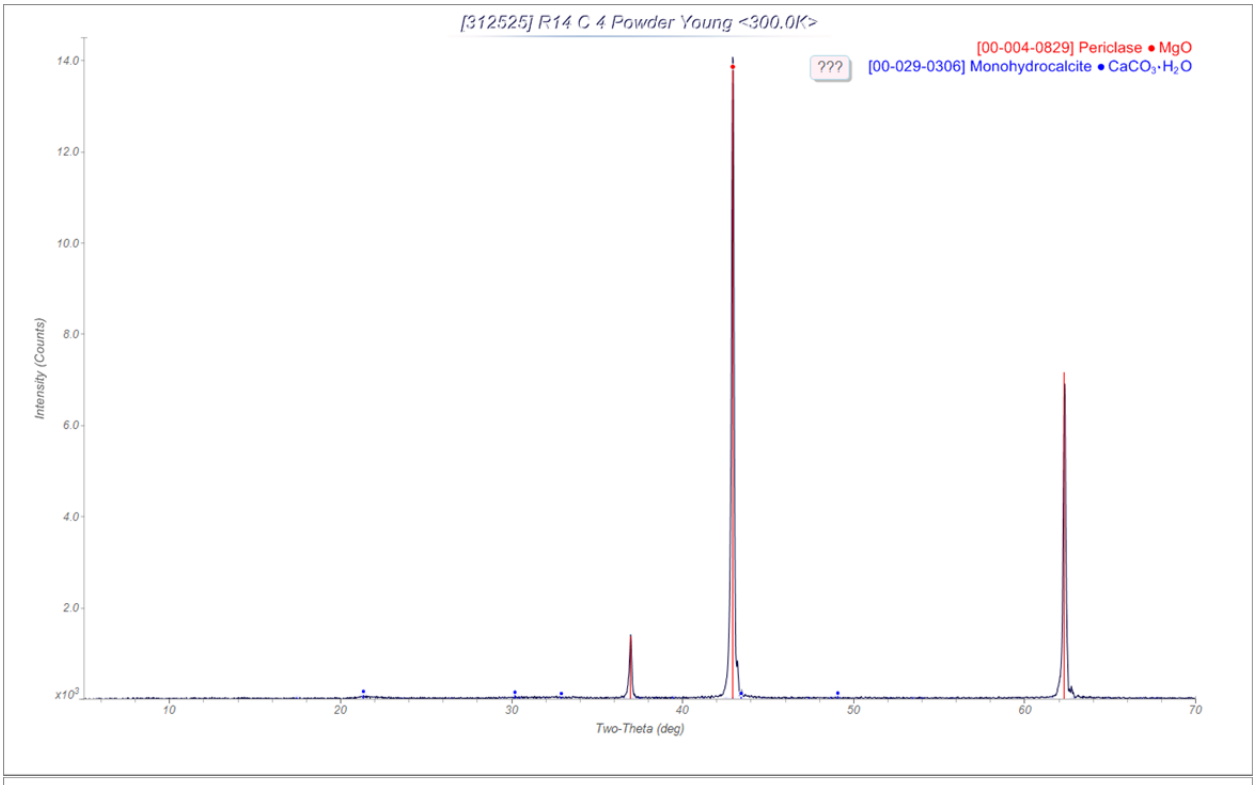
3.9 X-Ray Diffraction

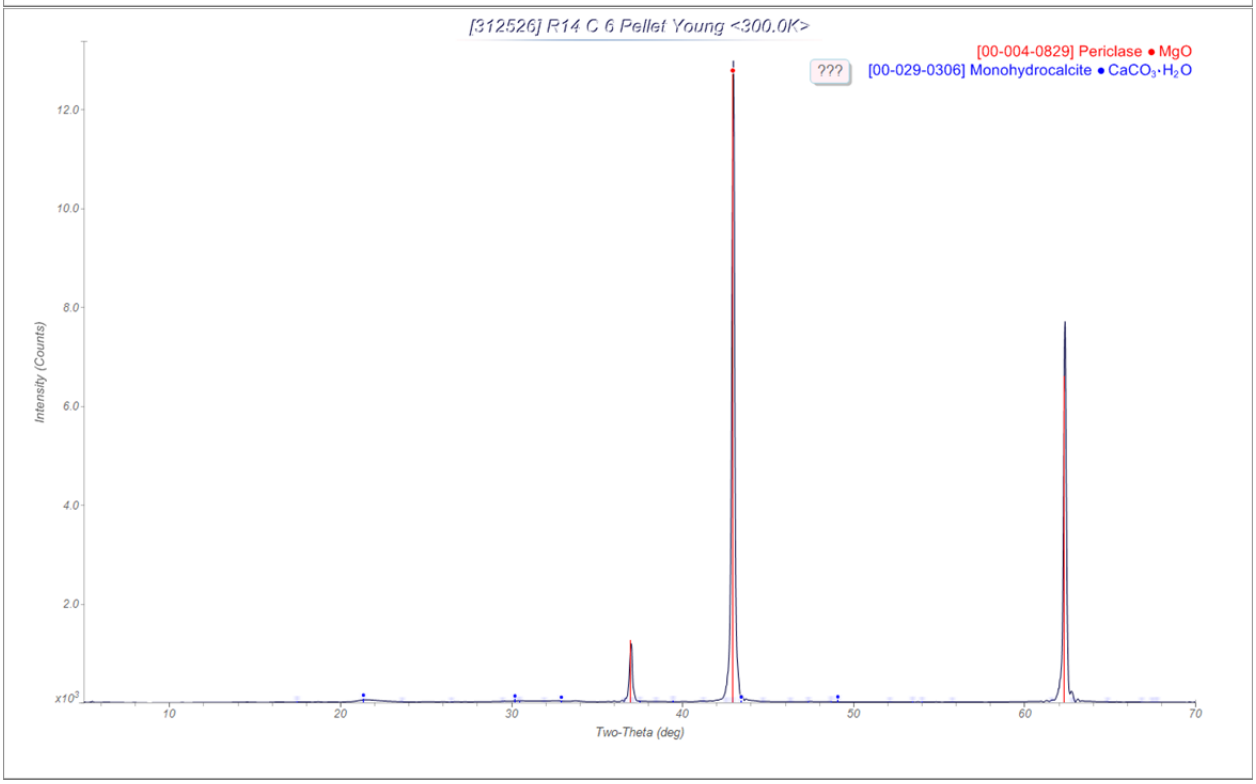
The X-ray Diffraction Laboratory of the SRNL examined eleven WIPP samples (R-14 C-2 XPD {Powder, Bulk MgO}, R-14 C4 XPL {Pellet, Bulk MgO}, R-16 C-6 XPD {Powder, Bulk}, R-16 C4 {Sticky Tape}, Mgo Ref#2 {Non Deployed MgO}, #2, 6, and 7; 300312522, 300312523, 300312524, 300312525, 300312526, 300312527, 300312748, 30031941, 300313811, 300313920, and 313921). Sample 2 was the debris sample taken from R-15 C-5. Samples 6 and 7 were magnesium oxide from the super sack at the floor of the waste face. Sample 6 was from the surface and sample 7 was from a depth of 6 inches.. The solids (particles and powder) from the three samples were ground in an agate mortar/pestle and adhered to a 2" by 2" piece of plate glass with a mixture of 10% Collodion/Amyl Acetate. The samples were run on a Bruker D8 Advance x-ray diffraction instrument with a 2θ range of 5 to 70°, a step size of 0.02^{o}, and a dwell time of 1 second. Search-match identification was performed with Jade software from Materials Data Inc. and the International Centre of Diffraction Database. The analysis of the powder, pellet and the non-depolyed MgO samples were consistent with periclase. Monohydrocalcite was detected but the match is questionable. No magnesium hydroxide, magnesium carbonate, or other reaction intermediates were identified. Nitratine, Trona, and Periclase were found on sample 2 from R15 C5. Periclase was found in both sample 6 and 7. Figures below show the results of the x-ray diffraction analysis.

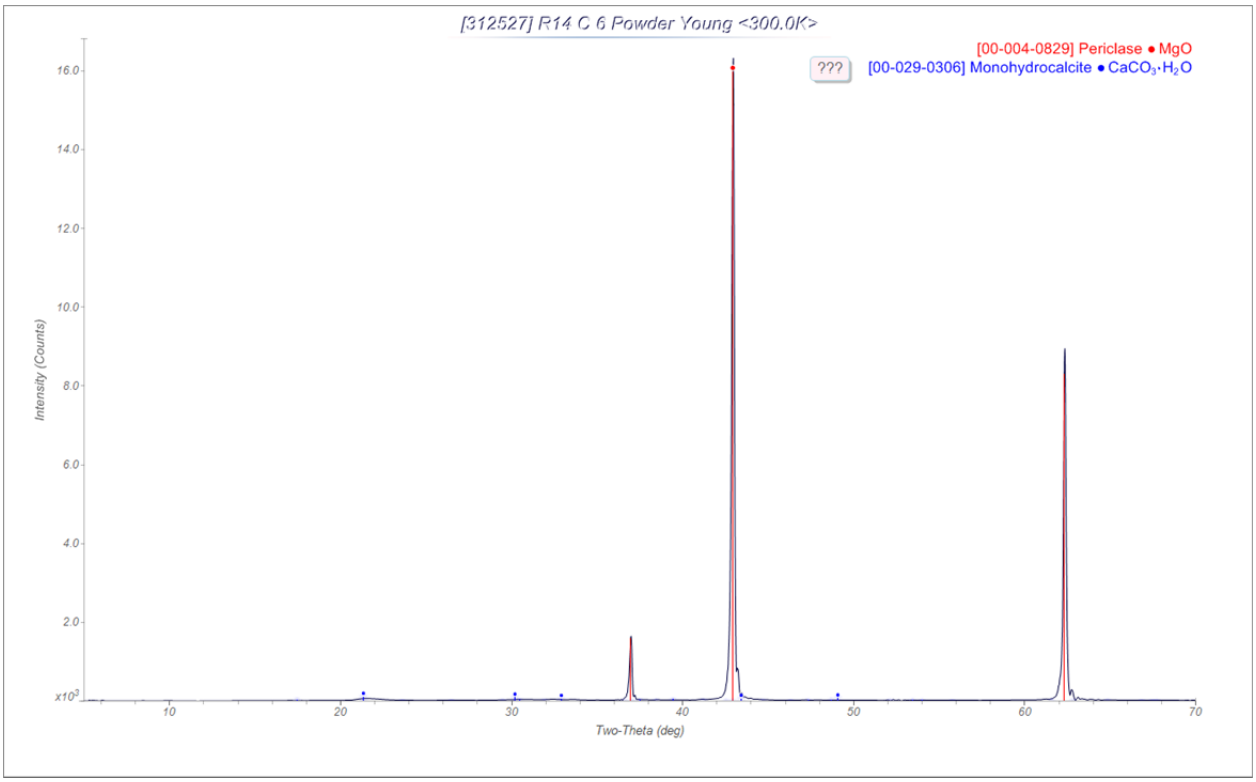


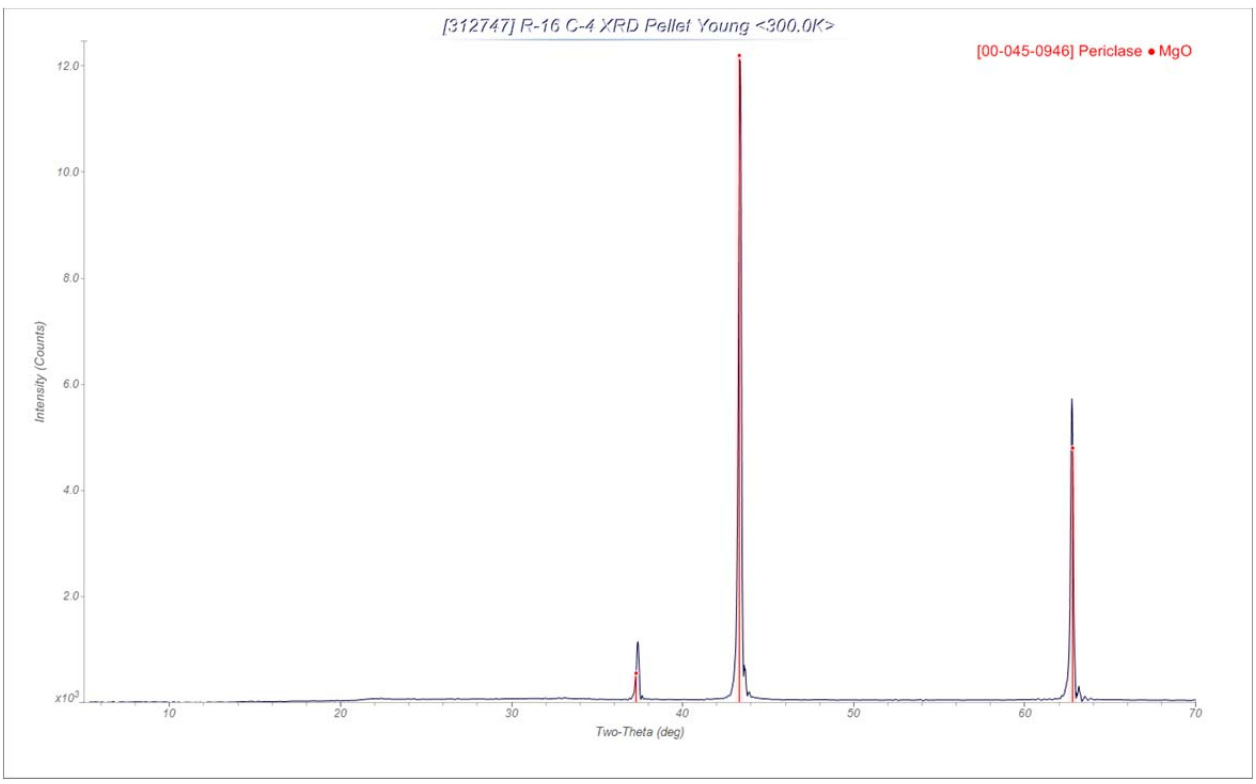

[312523] R14 C 2 XPD Young <300.0K>
[00-004-0829] Periclase • MgO
???
[00-029-0306] Monohydrocalcite • $CaCO_3 \cdot H_2O$
Intensity (Counts)
x10³
14.0
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)

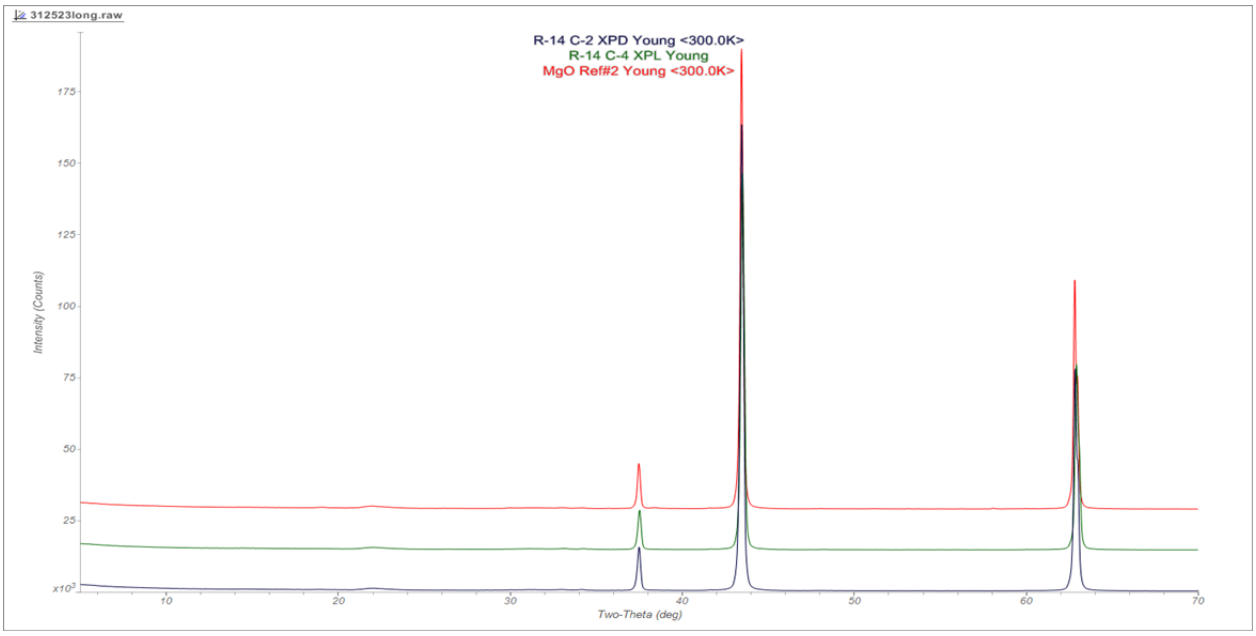

[312524] R14 C 4 Pellet Young <300.0K>
[00-004-0829] Periclase • MgO
???
[00-029-0306] Monohydrocalcite • $CaCO_3 \cdot H_2O$
Intensity (Counts)
x10³
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)

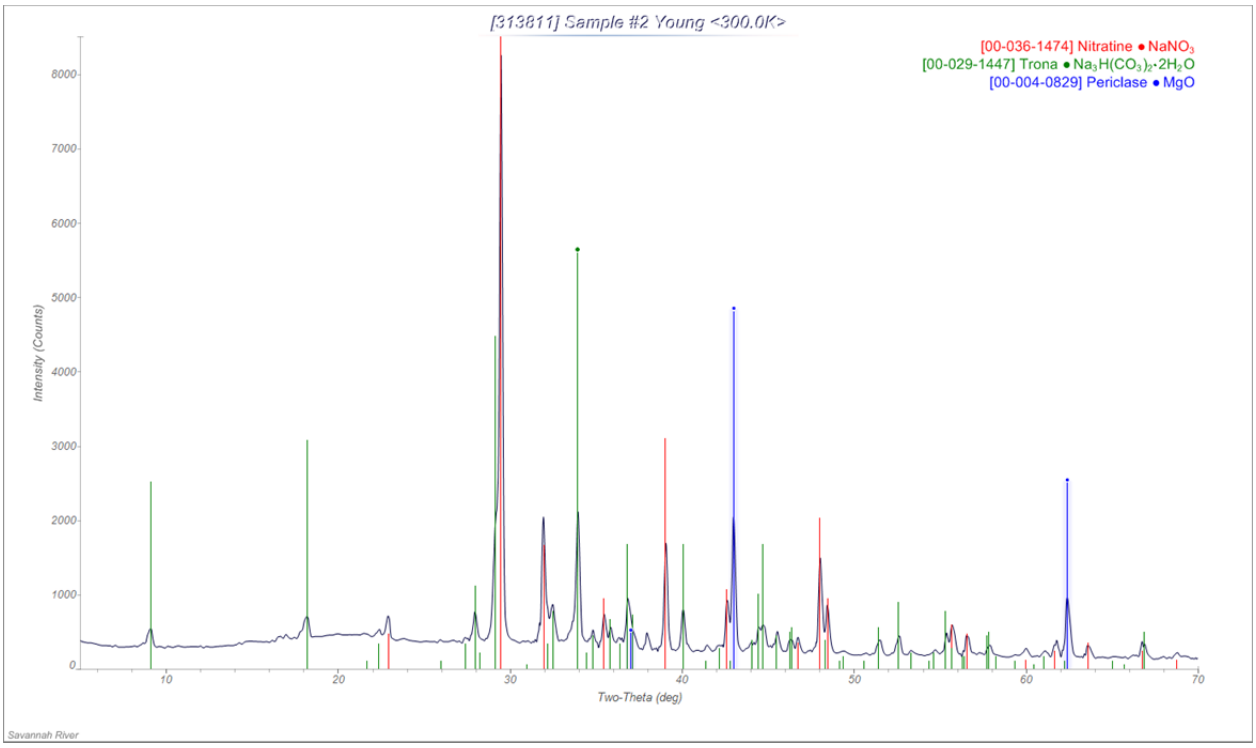

[312525] R14 C 4 Powder Young <300.0K>
[00-004-0829] Periclase • MgO
???
[00-029-0306] Monohydrocalcite • CaCO3·H2O
Intensity (Counts)
x10^3
14.0
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)

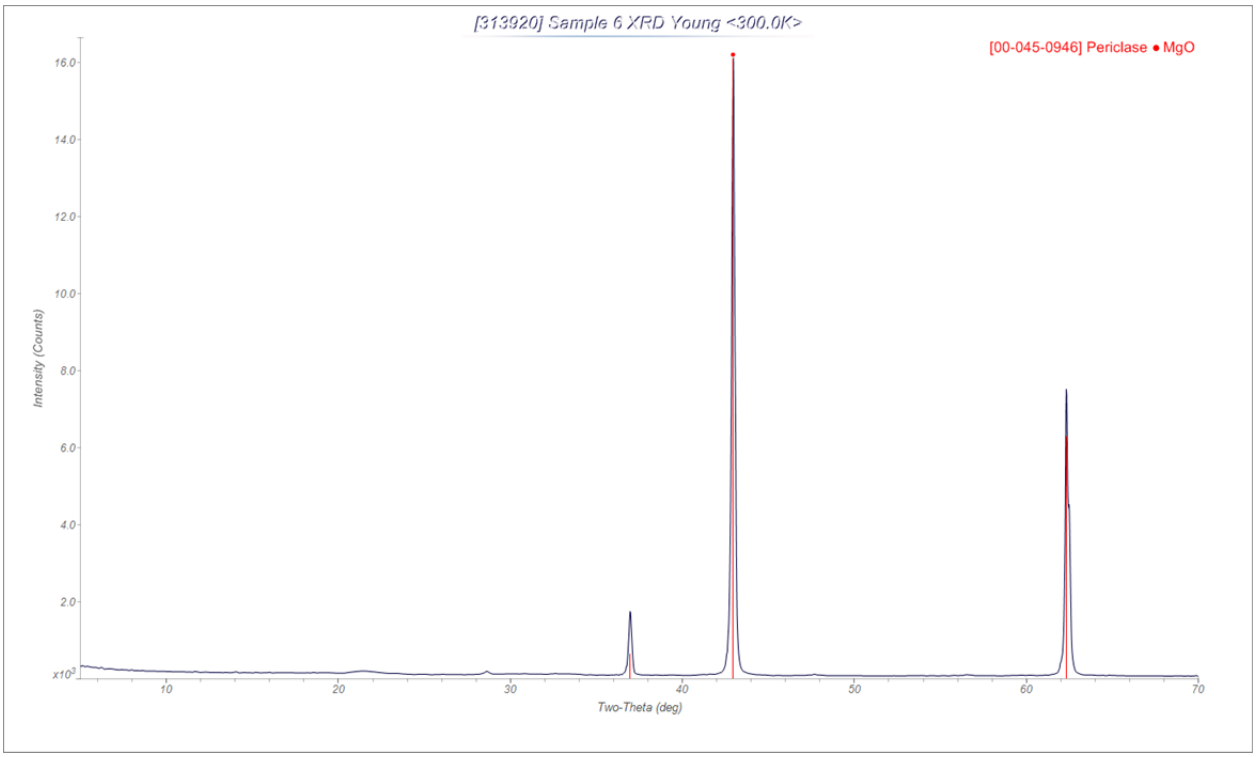

[312526] R14 C 6 Pellet Young <300.0K>
[00-004-0829] Periclase • MgO
???
[00-029-0306] Monohydrocalcite • CaCO3·H2O
Intensity (Counts)
x10^3
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)

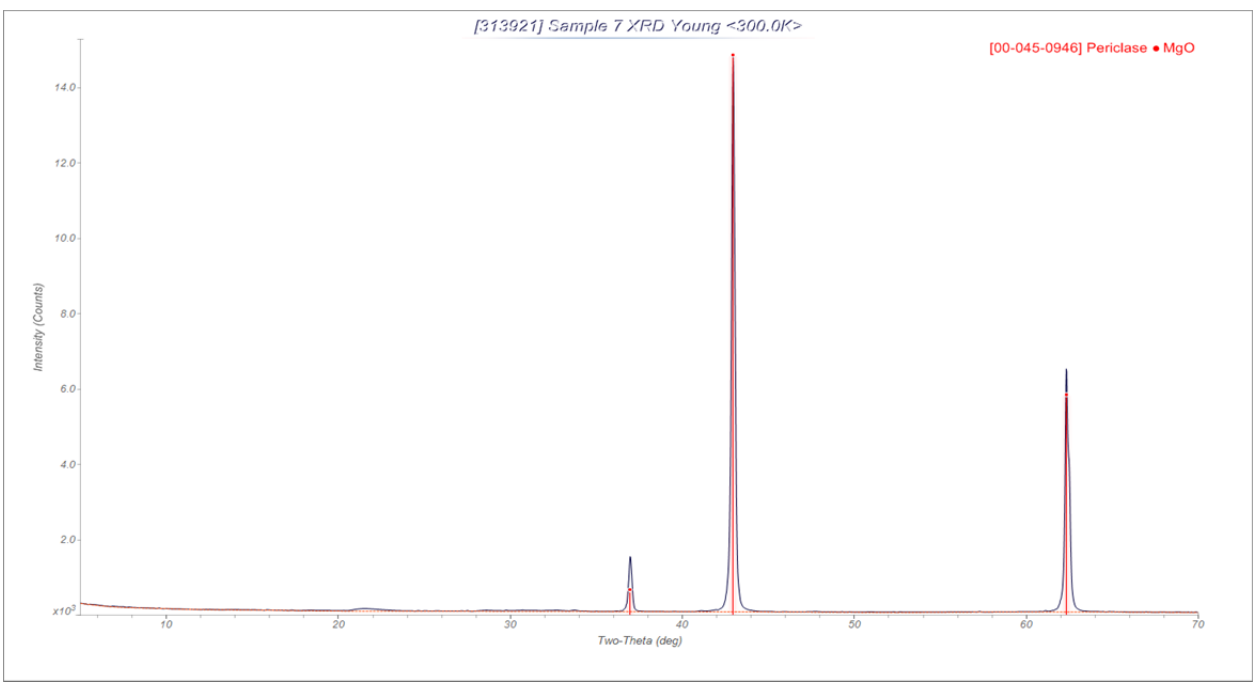

[312527] R14 C 6 Powder Young <300.0K>
[00-004-0829] Periclase • MgO
???
[00-029-0306] Monohydrocalcite • $CaCO_3 \cdot H_2O$
Intensity (Counts)
x10³
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)


[312747] R-16 C-4 XRD Pellet Young <300.0K>
[00-045-0946] Periclase • MgO
Intensity (Counts)
x10³
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)


312523long.raw
R-14 C-2 XPD Young <300.0K>
R-14 C-4 XPL Young
MgO Ref#2 Young <300.0K>
Intensity (Counts)
x10³
Two-Theta (deg)


[313811] Sample #2 Young <300.0K>
[00-036-1474] Nitratine • NaNO3
[00-029-1447] Trona • Na3H(CO3)2·2H2O
[00-004-0829] Periclase • MgO
Intensity (Counts)
Two-Theta (deg)
Savannah River


[313920] Sample 6 XRD Young <300.0K>
[00-045-0946] Periclase • MgO
Intensity (Counts)
x10³
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)


[313921] Sample 7 XRD Young <300.0K>
[00-045-0946] Periclase • MgO
Intensity (Counts)
x10³
14.0
12.0
10.0
8.0
6.0
4.0
2.0
10
20
30
40
50
60
70
Two-Theta (deg)

3.10 Radiochemistry

ADD Radiochemistry WIPP TAT Data Summary
Two surface wipe samples from Panel 7 Rooms 1 (ADS # 300311201) and 6 (ADS # 300311203) were analyzed to determine their respective Am-241 to Pu ratios. Stained areas of the Masslin were excised and then digested off the Masslin with a mixture of HNO3/HCl/HF. The dissolution was assayed by gamma for an Am-241 value, and by a plutonium thenoyltrifluoroacetone (TTA) extraction followed by an alpha spectrometry assay. The data is provided below. Alpha spectrometry provided the activity of the sum of Pu-239 and Pu-240 (the alphas cannot be resolved) and the Pu-238 activity. I provided am Am-241/Pu-239 ratio assuming the Pu-240 enrichment was 7%, which is what we have been observing on the CAM filters.

ADS LIMS #	Sample ID	Am-241 (dpm/sample)	1 Sigma %Unc	Pu-239/240 (dpm/sample)	1 Sigma %Unc	(
300311201	Panel 7 Room 1	3.94E+04	5.00%	5.50E+03	5.43%	
300311203	Panel 7 Room 6	7.86E+04	5.00%	9.85E+03	5.52%	
				Assuming 7% Pu-240 enrichment		
ADS LIMS #	Sample ID	Am-241 (dpm/sample)	1 Sigma %Unc	Pu-239 (dpm/sample)	Am-241/Pu-239 Activity Ratio	
300311201	Panel 7 Room 1	3.94E+04	5.00%	4.31E+03	9.14E+00	
300311203	Panel 7 Room 6	7.86E+04	5.00%	7.72E+03	1.02E+01	

Gamma Screening Analyses were conducted on as received samples of MgO received from WIPP on 6/5/14. The results of those screenings are provided below.

ADS Sample ID	300312402	
Customer ID	R_16 C_4	
Radionuclide	Activity (DPM/Sample)	1 Sigma %Uncertainty
Am-241	3.63E+03	25%
ADS Sample ID	300312403	
Customer ID	R_14 C_4	
Radionuclide	Activity (DPM/Sample)	1 Sigma %Uncertainty
Am-241	1.20E+03	25%
ADS Sample ID	300312404	
Customer ID	R_14 C_6	

Radionuclide	Activity (DPM/Sample)	1 Sigma %Uncertainty
Am-241	< 2.55E+02	MDA
ADS Sample ID	300312405	
Customer ID	R_14 C_2	
Radionuclide	Activity (DPM/Sample)	1 Sigma %Uncertainty
Am-241	1.14E+03	25%

The Archive A IAEA swipe (ADS 300313605) was assayed by gamma spectrometry as received, the results of that analysis follow.

ADS ID	300313605	
Customer ID	Archive A, IAEA swipe	
Radionuclide	Activity(uCi/Sample)	1 Sigma %Unc
Am-241	1.26E+00	20%
Pu-239	1.34E-01	20%
Am-243/Np-239	1.78E-04	20%
Np-237/Pa-233	1.11E-05	20%

The IAEA Swipe was sectioned, one section was digested using a peroxide fusion, one section was digested using a mixed acid digestion, and two sections were archived. All portions were assayed by gamma prior to dissolution. The results of those gamma assays follow.

ADP FILE/NOTEBOOK REF	SRNL-NB-2012-00058 PAGE 41		
Sample ID	300313726		
Comment	69120_A_MA		
Nuclide	dpm/sample	1 Sigma Uncertainty	
Am-241	6.24E+05	5.00%	
Sample ID	300313727		
Comment	69120_A_ARC1		
Nuclide	dpm/sample	1 Sigma Uncertainty	
Am-241	3.21E+04	5.00%	
Sample ID	300313728		
Comment	69120_A_ARC2		
Nuclide	dpm/sample	1 Sigma	

		Uncertainty
Am-241	2.12E+04	5.00%
Sample ID	300313733	
Comment	69120_A_PF	
Nuclide	dpm/sample	1 Sigma Uncertainty
Am-241	4.03E+05	5.00%
Sample ID	300313734	
Comment	69120_H_PF	
Nuclide	dpm/sample	1 Sigma Uncertainty
Am-241	<1.30E+02	MDA

The LANL Archive B Solid Debris sample (ADS LIMS # 300313607) was too radioactive to be analyzed directly on the detector as received. The sample was subdivided into 9 bottles, each was assayed quickly by gamma spectrometry to establish how homogeneous the americium contamination was across the kitty litter. The sub-aliquots were also too radioactive to be assayed directly on the HPGe gamma spectrometer, and had to be assayed through a cadmium filter. All results are corrected for that attenuation.

Sample ID	313607 - 1	
Comment	0.440g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	9.10E+08	5.00%
Sample ID	313607 - 2	
Comment	0.708g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	1.47E+09	5.00%
Sample ID	313607 - 3	
Comment	0.391g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	1.82E+09	5.00%
Sample ID	313607 - 4	
Comment	0.441g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	1.34E+09	5.00%
Sample ID	313607 - 5	

Comment	0.662g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	1.12E+09	5.00%
Sample ID	313607 - 6	
Comment	0.756g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	9.40E+08	5.00%
Sample ID	313607 - 7	
Comment	0.346g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	1.68E+09	5.00%
Sample ID	313607 - 8	
Comment	0.451g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	8.79E+08	5.00%
Sample ID	313607 - 9	
Comment	0.454g	
Nuclide	dpm/g	1 Sigma Uncertainty
Am-241	1.60E+09	5.00%

One subsample of 300313607 was assayed for an extended period of time through a cadmium filter. All results are corrected for the attenuation of that cadmium filter.

ADS LIMS # 30313607		
Radionuclide	Activity (uCi/g)	1 Sigma%Uncertainty
Pa-233	2.41E-03	5%
Np-239	5.75E-02	5%
Pu-239	2.31E+01	5%
Am-241	4.23E+02	5%

Some additional calculations on the LANL Archive B Solid Debris sample (ADS LIMS # 300313607) were carried out for the TAT, utilizing the ICP-MS data set and comparing it to the gamma spectrometry data set. The results of that data reduction follow.

	Activity Ratio	
Am-241/Pu-239	1.84E+01	
Am241/Np-237	1.76E+05	

Am-241/Am-243	7.39E+03	
	Mass ratio	
Pu-240/239	7.44E-02	
	% Pu-240 Enrichment	
%Pu-240	6.93%	
	% U-235 Enrichment	U-235/U-238
U-235 enrichment	3.99%	4.18E-02
	mass ratio	Activity ratio (ignoring Pu-241)
Am-241/Pu-239	2.73E-01	1.51E+01

Sample Archive D Glass Swipe (ADS LIMS # 300313610) was assayed directly by gamma spectrometry as received. The results follow.

Sample ID	300313610	
Comment	NA	
Nuclide	dpm/sample	1 Sigma %Uncertainty
Pu-239	4.56E+05	20%
Am-241	4.46E+06	20%

A set of MGO samples received from WIPP on 8/15/14, from chain of custody 9572. The samples were MGO from the top of drum 68660 and from the SWB R15C5. The samples were identified as Sample #1 (ADS LIMS #300313810), Sample #2 (ADS LIMS #300313811), Sample #3 (ADS LIMS #300313812), Sample #6 (ADS LIMS #300313813), and Sample #7 (ADS LIMS #300313814). The samples were screened by gamma spectrometry as received. The results of those assays follow. Some of the samples had to be counted through cadmium filters.

Sample ID	300313810	
Comment	NA	
Nuclide	dpm/sample	1 Sigma Uncertainty
Am-241	1.14E+04	20%
Sample ID	300313811	
Comment	Couted Raised on ER +Cd plate	
Nuclide	dpm/sample	1 Sigma Uncertainty
Am-241	1.24E+08	20%
Sample ID	300313812	
Comment	NA	

Nuclide	dpm/sample	1 Sigma Uncertainty
Am-241	1.71E+07	20%
Sample ID	300313813	
Comment	NA	
Nuclide	dpm/sample	1 Sigma Uncertainty
Am-241	4.74E+01	20%
Sample ID	300313814	
Comment	NA	
Nuclide	dpm/sample	1 Sigma Uncertainty
Am-241	<1.71E+02	MDA

Sample #2 SWB R15C5 ADS LIMS #300313811

Sample #2 was subdivided into 6 subsamples and assayed for extended periods of time by gamma spectrometry.

		Am-241		Pu-239		Am-243		Np-239		Pa-233	
	Mass(g)	uCi/g	1S %Unc	uCi/g	1S %Unc	uCi/g	1S %Unc	uCi/g	1S %Unc	uCi/g	1S %Unc
313811 #1	0.237	4.75E+01	2.30	9.42E+00	5.46	5.24E-03	7.52	5.32E-03	4.18	6.08E-04	9.12
313811 #2	0.203	9.49E+01	2.30	1.91E+01	5.64	1.12E-02	7.52	1.11E-02	4.18	9.71E-04	9.12
313811 #3	0.237	4.84E+01	1.94	8.98E+00	3.41	5.69E-03	5.00	5.65E-03	3.02	5.86E-04	5.58
313811 #4	0.235	4.94E+01	2.24	9.06E+00	5.93	5.97E-03	7.36	5.91E-03	4.05	4.19E-04	10.8
313811 #5	0.400	4.05E+01	1.87	6.96E+00	5.60	5.47E-03	4.52	5.13E-03	2.80	4.62E-04	4.66
313811 #6	0.448	5.26E+01	2.08	8.69E+00	4.77	7.00E-03	6.60	6.36E-03	3.27	5.31E-04	7.26

Some additional calculations on the Sample #2 SWB R15C5 ADS LIMS #300313811 were carried out for the TAT, utilizing the ICP-MS data set and comparing it to the gamma spectrometry data set. The results of that data reduction follow.

					125keV/129 keV
	Am-241/Pu-239	Am241/Am243	Am-243/Np-239	Am-241/Np-237	Am-241/Pu-239
	Activity Ratio	Activity Ratio	Activity Ratio	Activity Ratio	Activity Ratio
313811 Subsample 1	5.04E+00	9.05E+03	9.85E-01	7.80E+04	5.11
313811 Subsample	4.97E+00	8.48E+03	1.00E+00	9.77E+04	4.87

2					
313811 Subsample 3	5.39E+00	8.51E+03	1.01E+00	8.26E+04	5.27
313811 Subsample 4	5.45E+00	8.27E+03	1.01E+00	1.18E+05	5.53
313811 Subsample 5	5.82E+00	7.42E+03	1.07E+00	8.78E+04	4.68
313811 Subsample 6	6.06E+00	7.51E+03	1.10E+00	9.92E+04	5.95
average	5.45E+00	8.21E+03	1.03E+00	9.38E+04	5.24E+00

	Mass ratio	
Pu-240/239	7.54E-02	
	% Pu-240 Enrichment	
%Pu-240	7.01%	
	% U-235 Enrichment	U-235/U-238 Ratio
U-235 enrichment	5.67%	6.07E-02
	mass ratio	Activity ratio (ignoring Pu-241)
Am-241/Pu-239	9.60E-02	5.32E+00

Sample #3 SWB R15C5 ADS LIMS #300313812
Sample #3, the second sample from R15C5 was also assayed for extended count times. The results of those assays follow.

Sample ID	300313812	SWB Sample#3
Comment	NA	
Nuclide	dpm/sample	1 Sigma Uncertainty
Np-237/Pa-233	1.77E+02	1.36%
Am-243/Np-239	1.86E+03	1.99%
Pu-239	1.54E+06	1.74%
Am-241	1.71E+07	1.61%

Some additional calculations on the Sample #3 SWB R15C5 ADS LIMS #300313812 were carried out for the TAT, utilizing the ICP-MS data set and comparing it to the gamma spectrometry data set. The results of that data reduction follow.

300313812	
By Gamma	Activity ratio
Am-241/Pu-239	1.11E+01
Am-241/Am-243	9.18E+03

Am-241/Np-237	9.67E+04
ICP-MS	
Am-241/Pu-239	7.54E+00
U-235 Enrichment	4.55%

Extended count time gamma assays of a number of wipe samples taken by the AIB in Vault 7 were requested by the TAT. The results of those assays follow.

ADS ID 300311437
Customer ID Table Chair Waste Face

Radioisotope	Activity (dpm/sample)	uncertainty (1 Sigma %)
Am-241	2.38E5	10%
Am-243/Np-239	3.14E1	13.1%

ADS ID 300311516
Customer ID Slip Sheet

Radioisotope	Activity (dpm/sample)	uncertainty (1 Sigma %)
Am-241	6.21E4	10%
Am-243/Np-239	6.34E0	13.6%

ADS ID 300311199
Customer ID Bag 1_Waste Face Pink

Radioisotope	Activity (dpm/sample)	uncertainty (1 Sigma %)
Am-241	1.77E5	10%
Am-243/Np-239	1.93E1	10%

ADS ID 300311439
Customer ID Slip Sheet Waste Face

Radioisotope	Activity (dpm/sample)	uncertainty (1 Sigma %)
Am-241	1.14E5	10%
Am-243/Np-239	1.32E1	10%
Pu-239	1.14E4	10%

ADS ID 300311515
Customer ID Pink Mat Waste Face

Radioisotope	Activity (dpm/sample)	uncertainty (1 Sigma %)
Am-241	3.04E4	10%
Am-243/Np-239	4.06E0	16%

ADS ID 300311200
Customer ID Waste Face

Radioisotope	Activity (dpm/sample)	uncertainty (1 Sigma %)
Am-241	2.43E4	10%
Am-243/Np-239	4.29E0	15%

ADS ID 300311202
Customer ID Waste Face Table

Radioisotope	Activity (dpm/sample)	uncertainty (1 Sigma %)
Am-241	5.26E4	10%
Am-243/Np-239	5.29E0	10%
Pu-239	3.36E3	16%

Additional gamma assays of a number of CAM filters submitted initially by the AIB were requested by the TAT. Additionally, ICP-MS data was taken on CAM samples previously analyzed for the AIB was requested by the TAT, and those results were compared to the earlier gamma assays. The results of the assays follow.

Sample ID	300311041 CAM Filter #3	
Comment	NA	
Nuclide	dpm/filter	1 Sigma Uncertainty
Np-237/Pa-233	3.42E+02	10.0%
Np-239	6.83E+02	10.0%
Pu-239	3.64E+05	10.0%
Am-241	5.68E+06	10.0%
Am-243	6.98E+02	10.0%
Sample ID	300311046 CAM Filter #8	
Comment	NA	
Nuclide	dpm/filter	1 Sigma Uncertainty
Np-237/Pa-233	5.44E+01	11.2%
Am-243/Np-239	9.29E+02	10.0%
Pu-239	3.52E+05	15.3%
Am-241	7.81E+06	10.0%

	Sample ID	300311042 CAM Filter #4	
	Comment	NA	
	Nuclide	dpm/filter	1 Sigma Uncertainty
ICP-MS	U-235	1.76E+00	20%
ICP-MS	U-238	5.04E+00	20%
gamma	Np-237/Pa-233	2.80E+01	7.40%
	Am-243/Np-239	7.92E+02	5.00%
	Pu-239	4.52E+05	10.00%
	Pu-240	1.22E+05	20%
	Am-241	7.13E+06	5.00%
	Pu-241	1.23E+06	17.10%
	Am-243	7.82E+02	5.00%
	U-235 Enrichment	5.16%	
	Pu-240 Enrichment	6.87%	

	Sample ID	300311044 CAM Filter #6	
	Comment	NA	
	Nuclide	dpm/filter	1 Sigma Uncertainty
ICP-MS	U-235	1.50E+00	20%
ICP-MS	U-238	4.48E+00	20%
	Np-237/Pa-233	3.58E+01	5.6%
	Np-239	7.02E+02	5.0%
	Pu-239	3.43E+05	10%
ICP-MS	Pu-240	9.57E+04	20%
	Am-241	6.58E+06	5.8%
	Am-243	7.51E+02	5.0%
ICP-MS	U-235 Enrichment	4.95%	
ICP-MS	Pu-240 Enrichment	7.08%	
	Sample ID	300311047 CAM Filter #9	
	Comment	NA	
	Nuclide	dpm/filter	1 Sigma Uncertainty
ICP-MS	U-235	1.69E+00	20%
ICP-MS	U-238	4.96E+00	20%
	Np-237/Pa-233	4.45E+01	5.0%
	Np-239	7.47E+02	5.0%
	Pu-239	5.46E+05	10%
ICP-MS	Pu-240	1.49E+05	20%
	Am-241	6.80E+06	5.0%
	Am-243	7.89E+02	5.0%
ICP-MS	U-235 Enrichment	5.03%	
ICP-MS	Pu-240 Enrichment	6.93%	

Atttachments:

Attachment 1 – Data Packages

Attachment 2 – Sample ID and Chain of Custody Cross Reference

Distribution:

M. J. Barnes, 773-A
L. H. Connelly, 773-A
R. H. Young, 773-A
C. M. Gregory, 773-A
D. P. DiPrete, 773-A
J. E. Young, 773-A
L. W. Brown, 773-A

Savannah River National Laboratory™

OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

We put science to work.™

SRNL Analytical Results Summary on WIPP TAT Investigative Samples from the WIPP Underground Sampling of August 2014 and the LANL Parent Drum

John Young, Chemist, Analytical Development R&D Programs

Clint Gregory, Manager, Analytical Development R&D Programs

WIPP Technical Assistance Team meeting at ORNL

November 17, 2014

Savannah River NUCLEAR SOLUTIONS℠

FLUOR • NEWPORT NEWS NUCLEAR • HONEYWELL

Contents

- Methods
  - Radiochemistry
  - ICP-MS
  - Wet Chemistry
  - ICP-ES
  - XRD/XRF
  - SEM
  - IC
  - GC
  - FTIR
  - TGA-MS
- Samples
  - WIPP R16-C4 top breached drum
  - WIPP R15-C5 standard waste box
  - WIPP R15-C5 standard waste box (alt. loc.)
  - WIPP Supersack MgO surface sample
  - WIPP Supersack MgO 6” depth sample
  - LANL Parent Drum Debris
  - LANL Parent Glass Swipe
  - LANL Parent IAEA Swipe
  - Non Deployed MgO
  - Non Deployed Swheat

Radchem Data

WIPP Sample #2 R15C5 Isotopics

Gamma Results

					125keV/129 keV
	Am-241/Pu-239	Am241/Am243	Am-243/Np-239	Am-241/Np-237	Am-241/Pu-239
	Activity Ratio	Activity Ratio	Activity Ratio	Activity Ratio	Activity Ratio
313811 Subsample 1	5.04E+00	9.05E+03	9.85E-01	7.80E+04	5.11
313811 Subsample 2	4.97E+00	8.48E+03	1.00E+00	9.77E+04	4.87
313811 Subsample 3	5.39E+00	8.51E+03	1.01E+00	8.26E+04	5.27
313811 Subsample 4	5.45E+00	8.27E+03	1.01E+00	1.18E+05	5.53
313811 Subsample 5	5.82E+00	7.42E+03	1.07E+00	8.78E+04	4.68
313811 Subsample 6	6.06E+00	7.51E+03	1.10E+00	9.92E+04	5.95
average	5.45E+00	8.21E+03	1.03E+00	9.38E+04	5.24E+00

ICP-MS Results

	Mass ratio	
Pu-240/239	7.54E-02	
	% Pu-240 Enrichment	
%Pu-240	7.01%	
	% U-235 Enrichment	
U-235 enrichment	5.67%	
	mass ratio	Activity ratio (ignoring Pu-241)
Am-241/Pu-239	9.60E-02	5.32E+00

WIPP Sample #3 R15C5 Isotopics

300313812	
By Gamma	Activity ratio
Am-241/Pu-239	1.11E+01
Am-241/Am-243	9.18E+03
Am-241/Np-237	9.67E+04
ICP-MS	
Am-241/Pu-239	7.54E+00
	Weight %
U-235 Enrichment	4.55%

WIPP LANL Parent Drum Debris

Gamma Results Through Cd Filter

	Activity Ratio
Am-241/Pu-239	**1.84E+01**
Am241/Np-237	**1.76E+05**
Am-241/Am-243	**7.39E+03**

ICP-MS (MA)

	Mass ratio	
Pu-240/239	7.44E-02	
	% Pu-240 Enrichment	
%Pu-240	6.93%	
	% U-235 Enrichment	U-235/U-238
U-235 enrichment	3.99%	4.18E-02
	mass ratio	Activity ratio (ignoring Pu-241)
Am-241/Pu-239	2.73E-01	1.51E+01

ICP-MS (PF)

	Mass ratio	
Pu-240/239	7.56E-02	
	% Pu-240 Enrichment	
%Pu-240	7.03%	
	% U-235 Enrichment	U-235/U-238
U-235 enrichment	3.67%	3.83E-02
	mass ratio	Activity ratio (ignoring Pu-241)
Am-241/Pu-239	2.68E-01	1.49E+01

Comparison of CAM Data to R15C5 and LANL Parent Drum Debris Data

Quantity	CAM #2	CAM#3	CAM#4	CAM#6	CAM#7	CAM#8	CAM#9	CAM#11	R15C5 SWB#2	R15C5 SWB #3	LANL Parent Drum Debris
Activity Ratio Am-241/Pu-239 (Gamma)	1.74E+01	1.56E+01	1.58E+01	1.92E+01	1.79E+01	2.22E+01	1.25E+01	1.21E+01	5.45E+00	1.11E+01	1.84E+01
Activity Ratio Am-241/Am-243 (Gamma)	9.43E+03	8.32E+03	9.00E+03	9.37E+03	8.79E+03	8.41E+03	9.10E+03	7.66E+03	8.21E+03	9.18E+03	7.39E+03
Activity Ratio Am-241/Np-237 (Gamma)	2.93E+05	1.66E+04	2.55E+05	1.84E+05	2.42E+05	1.43E+05	1.53E+05	2.00E+05	9.38E+04	9.67E+04	1.76E+05
Activity Ratio Am-241/Pu-239 (ICP-MS)	1.49E+01	N/A	1.26E+01	1.26E+01	1.32E+01	N/A	1.09E+01	9.53E+00	5.32E+00	7.54E+00	1.51E+01
U-235 Enrichment(wgt%)	5.24%	N/A	5.16%	4.95%	5.09%	N/A	5.03%	5.07%	5.67%	4.55%	3.99%
Pu-240 Enrichment(wgt%)	7.26%	N/A	6.87%	7.08%	7.20%	N/A	6.93%	6.87%	7.01%	N/A	6.93%

ICP-MS Data

ICP-MS: WIPP Sample #1 R16 C4 Mixed Acid (LIMS 300313910)

R16 C4 Mixed Acid

Sample Preparation

Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL; 0.1855 g in 50 mL

10x IDF in 2% nitric acid with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Ba at 39.8 ppm; 21.9 ppm m/z 51; 8.39 ppm m/z 88;3.22 ppm m/z 181; trace levels (< 1 ppm) at other masses

Typical RL < 0.03 ppm

	300313910 SAMPLE-1, R16 C4 Mixed Acid Digestion	
	2.6954 X	
m/z	ug/g	--
51	2.19E+01	(1.09E+00 %RSD)
59	4.11E-02	(9.44E-01 %RSD)
86	9.95E-01	(3.25E-01 %RSD)
87	7.73E-01	(1.40E+00 %RSD)
88	8.39E+00	(8.61E-01 %RSD)
92	2.84E-01	(4.33E+00 %RSD)
95	9.46E-02	(3.26E+00 %RSD)
96	1.36E-01	(6.56E-01 %RSD)
97	6.61E-02	(6.94E+00 %RSD)
98	1.51E-01	(9.68E+00 %RSD)
100	5.71E-02	(3.76E+00 %RSD)
114	2.80E-02	(1.05E+01 %RSD)
119	4.38E-02	(1.34E+00 %RSD)
121	1.52E-01	(2.67E-01 %RSD)
123	1.30E-01	(1.15E+00 %RSD)
133	7.77E-02	(1.11E+01 %RSD)
134	9.49E-01	(2.79E+00 %RSD)
135	2.59E+00	(9.56E-01 %RSD)
136	3.11E+00	(5.59E-01 %RSD)
137	4.47E+00	(4.60E-01 %RSD)
138	2.87E+01	(2.33E-01 %RSD)
181	3.22E+00	(1.15E+01 %RSD)
206	1.41E-01	(3.39E+00 %RSD)
207	1.19E-01	(4.68E+00 %RSD)
208	2.83E-01	(5.79E+00 %RSD)
238	7.44E-01	(1.67E+00 %RSD)

R16 C4 Peroxide Fusion – not enough material to perform peroxide fusion

ICP-MS: WIPP Sample #2 R15 C5 Mixed Acid (LIMS 300313911) and Peroxide Fusion (LIMS 300313924)

Sample #2 R15 C5 Mixed Acid

Sample Preparation

Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL; 0.1863 g in 50 mL

100x IDF in 2% nitric acid with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 0.64 wt%; 19.3 ppm m/z 235 and 311 ppm m/z 238; 36.2 ppm m/z 239; 31.7 ppm m/z 138; 14.3 m/z 181; 10 ppm m/z 71; trace levels (< 10 ppm) at other masses

Typical RL < 0.3 ppm

Sample #2 R15 C5 Peroxide Fusion

Sample Preparation

Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.2168 g in 100 mL

100x IDF in 2% nitric acid with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 0.77 wt%; elemental Hf at 0.12 wt%; elemental Ga at 129 ppm; elemental Ba at 104 ppm; elemental Sn at 95.0 ppm; 19.6 ppm m/z 235 and 324 ppm m/z 238; 185 ppm m/z 239; 62.9 ppm m/z 106; 30.9 ppm m/z 110; 22.7 ppm m/z 108; 21.8 ppm m/z 107; 19.7 ppm m/z 88; 17.7 ppm m/z 241; 13.9 ppm m/z 240; 11.8 ppm m/z 181; 10.5 ppm m/z 112; trace levels (< 10 ppm) at other masses

Typical RL < 0.5 ppm

	300313911 SAMPLE-2 R15 C5 Mixed Acid Digestion	
	26.8384 X	
m/z	ug/g	--
51	3.08E+00	(9.16E+00 %RSD)
59	1.19E+00	(6.59E+00 %RSD)
71	1.00E+01	(2.01E+00 %RSD)
85	4.72E+00	(8.33E-01 %RSD)
86	1.18E+00	(1.96E+01 %RSD)
87	2.85E+00	(2.38E+00 %RSD)
88	8.87E+00	(3.11E+00 %RSD)
89	2.85E-01	(1.15E+01 %RSD)
95	9.58E-01	(7.40E+00 %RSD)
96	1.02E+00	(9.41E+00 %RSD)
97	6.02E-01	(1.16E+01 %RSD)
98	1.47E+00	(7.49E-01 %RSD)
100	5.28E-01	(4.83E+00 %RSD)
104	3.45E-01	(2.16E-01 %RSD)
107	1.08E+00	(9.79E-02 %RSD)
109	1.00E+00	(6.27E+00 %RSD)
110	3.16E-01	(1.73E+00 %RSD)
111	5.99E-01	(2.32E+00 %RSD)
112	1.24E+00	(1.49E+00 %RSD)
113	7.38E-01	(1.58E+01 %RSD)
114	1.46E+00	(4.12E+00 %RSD)
119	5.79E+00	(7.06E-01 %RSD)
121	2.52E+00	(2.54E+00 %RSD)
123	1.94E+00	(1.71E+00 %RSD)
133	1.46E+00	(1.25E+00 %RSD)
134	1.53E+00	(2.77E+01 %RSD)
135	2.97E+00	(3.11E+00 %RSD)
136	3.78E+00	(1.34E+01 %RSD)
137	5.01E+00	(7.36E-01 %RSD)
138	3.17E+01	(2.37E+00 %RSD)
181	1.43E+01	(1.08E+00 %RSD)
204	8.67E+01	(2.85E-01 %RSD)
206	1.63E+03	(3.92E-01 %RSD)
207	1.38E+03	(3.84E-01 %RSD)
208	3.33E+03	(2.78E-01 %RSD)
234	4.57E-01	(2.93E+00 %RSD)
235	1.93E+01	(7.07E-01 %RSD)
236	2.71E+00	(2.80E+00 %RSD)
238	3.11E+02	(6.29E-01 %RSD)
239	3.62E+01	(8.67E-01 %RSD)
240	2.94E+00	(1.56E+00 %RSD)
241	4.55E+00	(4.27E-01 %RSD)

	300313924 SAMPLE-2 R15 C5 Peroxide Fusion Digestion	
	46.1255 X	
m/z	ug/g	--
51	5.97E+00	(3.23E-01 %RSD)
59	1.04E+00	(1.69E+00 %RSD)
69	7.73E+01	(1.33E+00 %RSD)
71	5.57E+01	(9.41E-01 %RSD)
85	5.21E+00	(5.39E+00 %RSD)
88	1.97E+01	(1.08E+00 %RSD)
89	4.92E+00	(6.03E-01 %RSD)
93	6.73E+00	(3.14E+00 %RSD)
106	6.29E+01	(1.83E-01 %RSD)
107	2.18E+01	(4.90E-01 %RSD)
108	2.27E+01	(1.19E+00 %RSD)
109	2.72E+00	(1.85E+00 %RSD)
110	3.09E+01	(6.19E-01 %RSD)
111	4.48E+00	(2.77E+00 %RSD)
112	1.05E+01	(4.18E-01 %RSD)
113	1.75E+00	(2.57E+00 %RSD)
114	2.74E+00	(4.85E+00 %RSD)
116	1.13E+01	(2.67E-01 %RSD)
117	7.22E+00	(4.32E-01 %RSD)
118	2.30E+01	(1.69E+00 %RSD)
119	1.19E+01	(6.30E-01 %RSD)
120	3.10E+01	(7.73E-01 %RSD)
121	2.09E+00	(5.99E-01 %RSD)
122	6.61E+00	(6.95E-01 %RSD)
123	1.70E+00	(1.67E+00 %RSD)
124	9.36E+00	(1.70E+00 %RSD)
134	4.21E+00	(2.87E+00 %RSD)
135	6.99E+00	(2.26E+00 %RSD)
136	9.37E+00	(5.37E-01 %RSD)
137	1.17E+01	(8.67E-01 %RSD)
138	7.28E+01	(9.40E-01 %RSD)
140	1.60E+00	(5.11E+00 %RSD)
176	5.71E+01	(5.53E-01 %RSD)
177	2.19E+02	(1.26E+00 %RSD)
178	3.18E+02	(1.61E+00 %RSD)
179	1.60E+02	(2.34E-01 %RSD)
180	4.09E+02	(1.57E+00 %RSD)
181	1.18E+01	(1.38E+00 %RSD)
204	1.05E+02	(1.02E-01 %RSD)
206	1.96E+03	(1.43E+00 %RSD)
207	1.67E+03	(7.13E-01 %RSD)
208	4.02E+03	(4.36E-01 %RSD)
234	4.67E-01	(5.83E-01 %RSD)
235	1.96E+01	(1.19E+00 %RSD)
236	2.72E+00	(6.50E-01 %RSD)
237	9.18E-01	(3.65E+00 %RSD)
238	3.24E+02	(9.97E-01 %RSD)
239	1.85E+02	(1.19E+00 %RSD)
240	1.39E+01	(1.33E+00 %RSD)
241	1.77E+01	(1.20E+00 %RSD)

ICP-MS: WIPP Sample #3 R15 C5 Mixed Acid (LIMS 300314375) and Peroxide Fusion (LIMS 300314377)

Sample #3 R15 C5 Mixed Acid

Sample Preparation

Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Let stand at room temp for 4 days. Solids removed by filtration and filtrate and wash solutions diluted to 50 mL; 0.1264 g in 50 mL

100x IDF in 2% nitric acid with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 0.39 wt%; 5.12 ppm m/z 235 and 105 ppm m/z 238; trace levels (< 10 ppm) at other masses

Typical RL < 0.4 ppm

	300314375 WIPP #3 (SPARE 15_5)_MA	
	39.557 X	
m/z	ug/g	--
51	7.20E+00	(1.81E+00 %RSD)
59	6.91E-01	(5.24E+00 %RSD)
85	1.62E+00	(2.22E+00 %RSD)
86	1.06E+00	(7.29E-01 %RSD)
87	1.40E+00	(1.51E+00 %RSD)
88	9.81E+00	(2.62E+00 %RSD)
95	9.59E-01	(6.04E-01 %RSD)
97	5.83E-01	(6.06E+00 %RSD)
98	1.39E+00	(7.39E+00 %RSD)
119	2.10E+00	(3.20E+00 %RSD)
123	1.02E+00	(5.14E+00 %RSD)
204	5.24E+01	(1.21E-02 %RSD)
206	9.71E+02	(1.30E-01 %RSD)
207	8.22E+02	(4.10E-01 %RSD)
208	2.06E+03	(2.38E-01 %RSD)
235	5.12E+00	(5.24E-01 %RSD)
236	7.06E-01	(4.71E-02 %RSD)
238	1.05E+02	(1.90E+00 %RSD)

Sample #3 R15 C5 Peroxide Fusion

Sample Preparation

Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.0779 g in 100 mL

500x IDF in 2% nitric acid with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 0.61 wt%; 8.93 ppm m/z 235 and 187 ppm m/z 238; 11.5 ppm m/z 51; 37.5 ppm m/z 71; 72.3 ppm m/z 239; trace levels (< 10 ppm) at other masses

Typical RL < 7 ppm

	300314377 WIPP #3 (SPARE 15_5)_PF	
	641.8485 X	
m/z	ug/g	--
51	1.15E+01	(1.97E+01 %RSD)
71	3.75E+01	(9.68E-01 %RSD)
111	7.23E+00	(2.42E+00 %RSD)
181	6.98E+00	(5.86E+00 %RSD)
194	7.75E+00	(1.05E+01 %RSD)
196	8.68E+00	(3.71E+00 %RSD)
204	8.21E+01	(4.52E-01 %RSD)
206	1.49E+03	(6.25E-01 %RSD)
207	1.27E+03	(2.06E-01 %RSD)
208	3.20E+03	(4.78E-01 %RSD)
235	8.93E+00	(4.40E+00 %RSD)
238	1.87E+02	(9.59E-01 %RSD)
239	7.23E+01	(1.36E+00 %RSD)
241	9.87E+00	(1.54E+00 %RSD)

ICP-MS: LANL Parent Drum IAEA Swipe Mixed Acid (LIMS 300313726,729) and Peroxide Fusion (LIMS 300313733,734)

IAEA Swipes Mixed Acid

Sample Preparation

Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF; 0.5218 g in 50 mL (A) and 0.5148 g in 50 mL (H)

10x IDF in 2% nitric acid of quarter swipe with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

[69120_A] elemental Pb at 372 µg/swipe; 0.194 µg/swipe m/z 235 and 4.74 µg/swipe m/z 238; 1.16 µg/swipe m/z 239; trace levels (< 1 µg/swipe) at other masses; [69120_H] no significant levels noted

Typical RL < 0.02 µg/swipe

	300313726 69120_A Mixed Acid Digestion IAEA Sample Swipe		300313729 69120_H Mixed Acid Digestion IAEA Blank Swipe	
	2 X		2 X	
m/z	ug/swipe	--	ug/swipe	--
204	5.12E+00	(1.57E-01 %RSD)	< 2.00E-02	(N/A %RSD)
206	9.43E+01	(8.27E-01 %RSD)	2.87E-02	(1.01E+00 %RSD)
207	7.94E+01	(2.27E-01 %RSD)	2.20E-02	(1.01E+01 %RSD)
208	1.95E+02	(6.11E-01 %RSD)	5.50E-02	(7.62E+00 %RSD)
235	1.94E-01	(7.73E-01 %RSD)	< 2.00E-02	(N/A %RSD)
238	4.74E+00	(5.25E-01 %RSD)	< 2.00E-02	(N/A %RSD)
239	1.16E+00	(1.38E-01 %RSD)	< 3.00E-02	(N/A %RSD)
240	8.52E-02	(4.02E-01 %RSD)	< 2.00E-02	(N/A %RSD)
241	2.91E-01	(4.98E-01 %RSD)	< 2.00E-02	(N/A %RSD)

IAEA Swipes Peroxide Fusion

Sample Preparation

Ramp up temperature then Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.4817 g in 100 mL (A) and 0.4850 g in 100 mL (H)

100x IDF in 2% nitric acid of quarter swipe with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

[69120_A] elemental Pb at 294 µg/swipe; 3.40 µg/swipe m/z 238; 1.06 µg/swipe m/z 239; [69120_H] no significant levels noted

Typical RL < 0.4 µg/swipe

	300313733 69120_A Peroxide Fusion Digestion IAEA Sample Swipe		300313734 69120_H Peroxide Fusion Digestion IAEA Blank Swipe	
	40 X		40 X	
m/z	ug/swipe	--	ug/swipe	--
204	3.95E+00	(1.58E-01 %RSD)	< 4.00E-01	(N/A %RSD)
206	7.34E+01	(6.58E-01 %RSD)	< 4.00E-01	(N/A %RSD)
207	6.15E+01	(3.77E-01 %RSD)	< 4.00E-01	(N/A %RSD)
208	1.54E+02	(2.76E-01 %RSD)	< 4.00E-01	(N/A %RSD)
238	3.40E+00	(1.15E+00 %RSD)	< 4.00E-01	(N/A %RSD)
239	1.06E+00	(2.22E-01 %RSD)	< 4.00E-01	(N/A %RSD)

ICP-MS: LANL Parent Drum Debris MA (LIMS 300313736) and PF (LIMS 300313741)

LANL Parent Drum Debris Mixed Acid

Sample Preparation

Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Let stand at room temp for 4 days. Solids removed by filtration and filtrate and wash solutions diluted to 50 mL; 0.6968 g in 50 mL

1000x IDF in 2% nitric acid with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 8.4 wt%; 224 ppm m/z 235 and 5370 ppm m/z 238; 338 ppm m/z 69; 293 ppm m/z 71; 21.0 ppm m/z 236; 25.2 ppm m/z 239; trace levels (< 10 ppm) at other masses

Typical RL < 0.7 ppm

	300313736 69120_B Mixed Acid Digestion LANL PARENT DRUM DEBRIS	
	71.7566 X	
m/z	ug/g	--
51	1.90E+00	(5.07E+00 %RSD)
59	5.97E+00	(7.51E+00 %RSD)
69	3.38E+02	(2.55E+00 %RSD)
71	2.93E+02	(3.57E+00 %RSD)
88	1.37E+00	(1.67E+00 %RSD)
91	2.33E+00	(7.58E+00 %RSD)
92	7.54E+00	(6.27E+00 %RSD)
94	6.62E+00	(1.04E+01 %RSD)
95	4.98E+00	(7.94E+00 %RSD)
96	5.88E+00	(3.98E+00 %RSD)
97	3.05E+00	(2.58E+00 %RSD)
98	7.75E+00	(1.33E+00 %RSD)
100	2.99E+00	(1.65E+00 %RSD)
103	3.73E+00	(3.85E+00 %RSD)
104	6.97E+00	(2.63E+00 %RSD)
112	1.33E+00	(3.39E+00 %RSD)
113	8.42E-01	(1.45E+01 %RSD)
114	1.61E+00	(2.95E+00 %RSD)
116	1.01E+00	(5.07E+00 %RSD)
118	1.71E+00	(1.05E+01 %RSD)
119	9.62E+01	(2.51E+00 %RSD)
120	1.67E+00	(2.53E+00 %RSD)
121	5.57E+00	(8.52E+00 %RSD)
123	4.20E+00	(4.54E+00 %RSD)
126	8.72E-01	(1.94E+01 %RSD)
128	1.14E+00	(4.81E+01 %RSD)
130	9.79E-01	(4.36E+00 %RSD)
140	2.06E+00	(1.34E+00 %RSD)
204	1.13E+03	(2.00E+00 %RSD)
206	2.10E+04	(3.00E-01 %RSD)
207	1.77E+04	(4.05E-01 %RSD)
208	4.40E+04	(4.77E-01 %RSD)
233	1.25E+00	(1.37E+00 %RSD)
234	4.37E+00	(1.28E+01 %RSD)
235	2.24E+02	(1.56E+00 %RSD)
236	2.10E+01	(2.27E+00 %RSD)
238	5.37E+03	(9.80E-01 %RSD)
239	2.52E+01	(1.82E+00 %RSD)
240	1.87E+00	(2.96E+00 %RSD)
241	6.88E+00	(1.66E+00 %RSD)

LANL Parent Drum Debris Peroxide Fusion

Sample Preparation

Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.4317 g in 100 mL

1000x IDF in 2% nitric acid with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 11 wt%; elemental Hf at 554 ppm; 134 ppm m/z 235 and 3490 ppm m/z 238; 553 ppm m/z 239; 494 ppm m/z 69, 424 ppm m/z 71; 148 ppm m/z 241; 53.6 ppm m/z 89; 41.8 ppm m/z 240; 28.6 ppm m/z 106; 24.8 ppm m/z 138; 16.1 ppm m/z 59; 14.7 ppm m/z 236; 14.1 ppm m/z 110; 10.7 ppm m/z 107; trace levels (< 10 ppm) at other masses

Typical RL < 2.3 ppm

	300313741 69120_B Peroxide Fusion Digestion LANL PARENT DRUM DEBRIS	
	231.6423 X	
m/z	ug/g	--
51	2.49E+00	(1.95E+01 %RSD)
59	1.61E+01	(7.19E+00 %RSD)
69	4.94E+02	(2.63E+00 %RSD)
71	4.24E+02	(2.25E+00 %RSD)
88	9.42E+00	(9.20E+00 %RSD)
89	5.36E+01	(8.74E-01 %RSD)
103	4.75E+00	(1.35E+01 %RSD)
104	8.20E+00	(3.23E+00 %RSD)
106	2.86E+01	(8.48E-01 %RSD)
107	1.07E+01	(4.49E+00 %RSD)
108	9.47E+00	(2.02E+00 %RSD)
110	1.41E+01	(7.15E-03 %RSD)
111	2.64E+00	(1.39E+01 %RSD)
112	4.98E+00	(1.52E+00 %RSD)
119	5.89E+01	(3.63E+00 %RSD)
128	2.36E+00	(2.27E+01 %RSD)
138	2.48E+01	(9.12E-01 %RSD)
140	9.25E+00	(8.26E+00 %RSD)
176	2.81E+01	(6.16E-03 %RSD)
177	1.03E+02	(3.11E-01 %RSD)
178	1.51E+02	(1.41E+00 %RSD)
179	7.43E+01	(2.23E-01 %RSD)
180	1.94E+02	(2.33E-01 %RSD)
196	2.39E+00	(2.73E-01 %RSD)
204	1.47E+03	(1.13E+00 %RSD)
206	2.70E+04	(1.07E+00 %RSD)
207	2.30E+04	(1.43E+00 %RSD)
208	5.54E+04	(1.91E-01 %RSD)
234	2.99E+00	(2.10E+01 %RSD)
235	1.34E+02	(1.58E+00 %RSD)
236	1.47E+01	(1.32E+00 %RSD)
237	3.98E+00	(3.26E+00 %RSD)
238	3.49E+03	(4.77E-02 %RSD)
239	5.53E+02	(1.46E-01 %RSD)
240	4.18E+01	(9.71E-01 %RSD)
241	1.48E+02	(4.34E-01 %RSD)

ICP-MS: CAM Filter 4 Mixed Acid (LIMS 300313598) and Peroxide Fusion (LIMS 300313602)

CAM Filter 4 Mixed Acid

Sample Preparation

Hot Mixed Acid-overnight heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF; 0.0284 g in 50 mL

100x IDF in 2% nitric acid of quarter filter with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 971 µg/filter; 0.260 µg/filter m/z 235 and 4.92 µg/filter m/z 238; 9.59 µg/filter m/z 239; 6.04 µg/filter m/z 181; 3.10 µg/filter m/z 71; trace levels (< 3 µg/filter) at other masses

Typical RL < 0.2 µg/filter

CAM Filter 4 Peroxide Fusion

Sample Preparation

Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.0247 g in 100 mL

100x IDF in 2% nitric acid of quarter filter with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 1770 µg/filter; elemental Sb at 14.5 µg/filter; 0.561 µg/filter m/z 235 and 10.3 µg/filter m/z 238; 6.26 µg/filter m/z 239; 6.13 µg/filter m/z 71; trace levels (< 3 µg/filter) at other masses

Typical RL < 0.4 µg/filter

	300313598 CAM FILTER 4, SECTION MA	
	20 X	
m/z	ug/filter	--
59	1.14E+00	(5.62E+00 %RSD)
71	3.10E+00	(4.64E+00 %RSD)
85	2.19E-01	(1.44E+01 %RSD)
88	4.70E-01	(1.35E+01 %RSD)
90	1.25E+00	(1.07E+00 %RSD)
91	2.90E-01	(1.32E+01 %RSD)
93	2.04E+00	(6.83E+00 %RSD)
110	4.54E-01	(1.88E+00 %RSD)
111	7.24E-01	(4.14E+00 %RSD)
112	1.50E+00	(5.40E-03 %RSD)
113	7.25E-01	(3.96E-01 %RSD)
114	1.64E+00	(3.41E-02 %RSD)
181	6.04E+00	(8.61E+00 %RSD)
204	1.32E+01	(2.87E-01 %RSD)
206	2.46E+02	(1.17E-01 %RSD)
207	2.12E+02	(4.44E-01 %RSD)
208	5.09E+02	(5.52E-01 %RSD)
235	2.60E-01	(1.17E+00 %RSD)
238	4.92E+00	(2.29E+00 %RSD)
239	9.59E+00	(1.13E+01 %RSD)
240	6.53E-01	(9.03E+00 %RSD)
241	7.97E-01	(2.44E-01 %RSD)

	300313602 CAM FILTER 4, SECTION PF	
	40 X	
m/z	ug/filter	--
51	5.29E-01	(1.14E+00 %RSD)
59	1.99E+00	(7.32E+00 %RSD)
71	6.13E+00	(1.80E+00 %RSD)
89	4.75E-01	(1.26E+01 %RSD)
95	5.37E-01	(5.23E+00 %RSD)
98	6.69E-01	(1.96E+00 %RSD)
113	1.45E+00	(1.10E+01 %RSD)
114	2.53E+00	(1.62E+00 %RSD)
121	8.31E+00	(1.10E-01 %RSD)
123	6.11E+00	(6.32E-01 %RSD)
181	5.23E-01	(9.16E-01 %RSD)
204	2.43E+01	(1.18E+00 %RSD)
206	4.52E+02	(1.14E-02 %RSD)
207	3.86E+02	(6.40E-02 %RSD)
208	9.25E+02	(9.42E-01 %RSD)
235	5.61E-01	(2.49E+00 %RSD)
238	1.03E+01	(1.13E+00 %RSD)
239	6.26E+00	(2.39E-01 %RSD)
240	4.62E-01	(4.16E-01 %RSD)
241	1.43E+00	(1.77E+00 %RSD)

ICP-MS: CAM Filter 6 Mixed Acid (LIMS 300313599) and Peroxide Fusion (LIMS 300313603)

CAM Filter 6 Mixed Acid

Sample Preparation

Hot Mixed Acid-overnight heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF; 0.0249 g in 50 mL

100x IDF in 2% nitric acid of quarter filter with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 775 µg/filter; 0.204 µg/filter m/z 235 and 4.06 µg/filter m/z 238; 3.85 µg/filter m/z 239; trace levels (< 3 µg/filter) at other masses

Typical RL < 0.2 µg/filter

	300313599 CAM FILTER 6, SECTION MA	
	20 X	
m/z	ug/filter	--
59	1.23E+00	(8.87E-01 %RSD)
71	2.47E+00	(1.27E+00 %RSD)
85	2.06E-01	(1.60E+01 %RSD)
88	4.65E-01	(9.97E-01 %RSD)
93	3.98E-01	(1.56E+01 %RSD)
110	3.60E-01	(5.85E-02 %RSD)
111	5.36E-01	(2.37E+00 %RSD)
112	1.18E+00	(2.91E+00 %RSD)
113	6.46E-01	(7.26E+00 %RSD)
114	1.35E+00	(5.18E-01 %RSD)
181	1.35E+00	(3.19E+00 %RSD)
204	1.06E+01	(3.04E-01 %RSD)
206	1.97E+02	(6.09E-01 %RSD)
207	1.70E+02	(8.36E-01 %RSD)
208	4.06E+02	(1.03E+00 %RSD)
235	2.04E-01	(4.47E+00 %RSD)
238	4.06E+00	(5.93E-02 %RSD)
239	3.85E+00	(1.28E+01 %RSD)
240	2.77E-01	(1.50E+01 %RSD)
241	6.23E-01	(4.81E+00 %RSD)

CAM Filter 6 Peroxide Fusion

Sample Preparation

Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.0365 g in 100 mL

100x IDF in 2% nitric acid of quarter filter with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 2400 µg/filter; elemental Sb at 24.8 µg/filter; 0.753 µg/filter m/z 235 and 14.5 µg/filter m/z 238; 9.14 µg/filter m/z 239; 8.28 µg/filter m/z 71; 3.46 µg/filter m/z 114; 3.14 µg/filter m/z 59; trace levels (< 3 µg/filter) at other masses

Typical RL < 0.4 µg/filter

	300313603 CAM FILTER 6, SECTION PF	
	40 X	
m/z	ug/filter	--
59	3.14E+00	(2.95E+00 %RSD)
71	8.28E+00	(4.88E+00 %RSD)
89	5.28E-01	(1.53E+00 %RSD)
95	4.03E-01	(6.54E+00 %RSD)
98	4.98E-01	(8.31E+00 %RSD)
113	2.01E+00	(1.04E+01 %RSD)
114	3.46E+00	(1.10E+00 %RSD)
121	1.42E+01	(2.29E+00 %RSD)
123	1.05E+01	(1.47E+00 %RSD)
181	8.02E-01	(2.02E+00 %RSD)
204	3.30E+01	(1.96E-01 %RSD)
206	6.07E+02	(8.96E-01 %RSD)
207	5.18E+02	(7.38E-02 %RSD)
208	1.26E+03	(4.72E-01 %RSD)
235	7.53E-01	(1.77E+00 %RSD)
238	1.45E+01	(1.67E+00 %RSD)
239	9.14E+00	(8.18E-01 %RSD)
240	6.96E-01	(2.90E+00 %RSD)
241	2.08E+00	(4.14E-02 %RSD)

ICP-MS: CAM Filter 9 Mixed Acid (LIMS 300313600) and Peroxide Fusion (LIMS 300313604)

CAM Filter 9 Mixed Acid

Sample Preparation

Hot Mixed Acid-overnight heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF; 0.0328 g in 50 mL

100x IDF in 2% nitric acid of quarter filter with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 1030 µg/filter; 0.296 µg/filter m/z 235 and 5.58 µg/filter m/z 238; 5.62 µg/filter m/z 239; 3.35 µg/filter m/z 71; trace levels (< 3 µg/filter) at other masses

Typical RL < 0.2 µg/filter

	300313600 CAM FILTER 9, SECTION MA	
	20 X	
m/z	ug/filter	--
59	2.00E+00	(1.93E+00 %RSD)
71	3.35E+00	(1.35E+00 %RSD)
85	2.48E-01	(9.26E+00 %RSD)
88	6.88E-01	(5.39E+00 %RSD)
93	7.18E-01	(4.34E+00 %RSD)
110	5.05E-01	(3.36E+00 %RSD)
111	7.96E-01	(5.32E+00 %RSD)
112	1.73E+00	(7.31E-01 %RSD)
113	8.98E-01	(7.51E+00 %RSD)
114	1.86E+00	(2.35E+00 %RSD)
181	2.35E+00	(6.41E+00 %RSD)
204	1.39E+01	(1.14E+00 %RSD)
206	2.59E+02	(1.03E-01 %RSD)
207	2.24E+02	(3.15E-01 %RSD)
208	5.40E+02	(1.42E-01 %RSD)
235	2.96E-01	(2.74E+00 %RSD)
238	5.58E+00	(7.37E-01 %RSD)
239	5.62E+00	(5.73E+00 %RSD)
240	4.16E-01	(6.54E+00 %RSD)
241	8.20E-01	(2.31E+00 %RSD)

CAM Filter 9 Peroxide Fusion

Sample Preparation

Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.0379 g in 100 mL

100x IDF in 2% nitric acid of quarter filter with In and Bi internal standard

Instrument and Analysis Conditions

The instrument used was an Agilent 7700x ICPMS with He Collision Cell

Blank, 0.5, 5, 10, 25 ppb calibration curve with In and Bi internal standard in 2% nitric acid

Data Interpretation

Elemental Pb at 2270 µg/filter; elemental Sb at 23.4 µg/filter; 0.739 µg/filter m/z 235 and 13.9 µg/filter m/z 238; 9.53 µg/filter m/z 239; 7.87 µg/filter m/z 71; 3.23 µg/filter m/z 59; 3.20 µg/filter m/z 114; trace levels (< 3 µg/filter) at other masses

Typical RL < 0.4 µg/filter

	300313604 CAM FILTER 9, SECTION PF	
	40 X	
m/z	ug/filter	--
51	< 4.00E-01	(N/A %RSD)
59	3.23E+00	(4.82E+00 %RSD)
71	7.87E+00	(1.18E+00 %RSD)
89	6.09E-01	(6.95E+00 %RSD)
95	4.04E-01	(9.44E+00 %RSD)
98	5.08E-01	(1.22E+00 %RSD)
113	1.94E+00	(1.22E+01 %RSD)
114	3.20E+00	(1.34E+00 %RSD)
121	1.34E+01	(1.37E+00 %RSD)
123	1.03E+01	(1.04E+00 %RSD)
181	8.42E-01	(4.00E-01 %RSD)
204	3.11E+01	(8.18E-01 %RSD)
206	5.79E+02	(1.98E-01 %RSD)
207	4.92E+02	(2.53E-01 %RSD)
208	1.19E+03	(1.29E-01 %RSD)
235	7.39E-01	(2.17E-01 %RSD)
238	1.39E+01	(1.82E-01 %RSD)
239	9.53E+00	(1.25E+00 %RSD)
240	7.10E-01	(1.01E+00 %RSD)
241	1.87E+00	(7.17E-01 %RSD)

Wet Chemistry Data

TOTAL BASE DETERMINATION – WIPP R15C5 Sample #2 (LIMS 300314454)

- Sample Preparations
  - Room temperature leach of sample (0.1366g) in DI water (20 mL) for 4 days; filter. The solution was used for both IC and titration methods.
- Sample Measurements
  - pH measurement was made at 10.8 in the liquid sample as received from the water leach .
  - Titrated 1.0 mL of liquid sample as received from the water leach with 0.1N HCl and the total base was determined at 0.022N
  - Approximately 30% of the total base was something other than hydroxide, probably carbonate.

ACID DETERMINATION - LANL Parent Drum Debris LIMS ID# 300313743

- Sample Preparations
  - Room temperature leach of sample (0.6901g) in DI water (20 mL) for 4 days; filter. The solution was used for both IC and titration methods
- Sample Measurements
  - pH measurement was made at 3.78 in the liquid sample as received from the water leach
  - Titrated 0.5 mL of liquid sample as received from the water leach 0.1N NaOH and the total acid determined at less than 0.005 N (method reporting limit)

Wet Chemistry Data – Total Organic Carbon (TOC), Total Inorganic Carbon (TIC)

sample	TIC (ug/mL)	TOC (ug/mL)	tc (ug/mL)
300313742	1.10	3.36	4.46
300314453 *	1.71	1.00	2.71
300314454**	298	159	457
300313744	1.6	800	802
Swheat	10.0	1200	1210

*(blank, 0.1000g / 20 mL)

**(R15C5, 0.1366g / 20mL)

TOC was measured at 159 mg/L in R15c5 and 1200 mg/L in undeployed Swheat

ICP-ES Data

WIPP 16-4 Sample #1 by Digestion and ICP-ES (LIMS 300313910)

- Mixed Acid - Sample Preparation
  - Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL; 0.1855g in 50mL
  - 2-100x dilution in 2% nitric acid with Sc internal standard

- Instrument and Analysis Conditions
  - The instrument used was a Leeman Prodigy-XP Eschelle Grating simultaneous ICP-ES
  - Blank, blank, 5 and 10 mg/L calibration curve with Sc internal standard in 2% nitric acid

- Peroxide Fusion – Sample Preparation
  - Sample was insufficient mass for both MA and PF (Ran MA only)

- Data Interpretation
  - Ca at 3890 µg/g
  - Na at 1290 µg/g
  - Mg at 1060 µg/g
  - Other metals all < 1000,
  - Noteworthy that Pb < 32

WIPP 16-4 Sample #1 by Digestion and ICP-ES (LIMS 300313910)

- 16-4 ICP-ES / Mixed Acid

	USER_SAMPLEID:	WIPP 16-4
	SAMPLE_ID:	300313910
	UNITS:	ug/g
Element		
Ca		3890
Na		1290
Mg		1060
Pb		< 32
Others		<1000

WIPP 16-4 Sample #1 by Digestion and ICP-ES (LIMS 300313910)

- ICP-ES: 16-4 Sample MA

ICP-OES Results

Description:	WIPP UG 8/15, MIXED ACID DISS.
Travel Copy:	66301
Instrument:	Leeman Prodigy ICP-ES
Reviewer:	Mark Jones
Comments:	

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	SAMPLE_1_MA	
SAMPLE_ID:	300313910	
UNITS:	ug/g	
Element		
Ag	< 7.92	(N/A %RSD)
Al	68.6	(12.2 %RSD)
Ba	39.4	(10.1 %RSD)
Be	< 0.755	(N/A %RSD)
Ca	3890	(10 %RSD)
Cd	< 5.61	(N/A %RSD)
Ce	< 22.5	(N/A %RSD)
Co	< 6.58	(N/A %RSD)
Cr	< 5.01	(N/A %RSD)
Cu	< 4.31	(N/A %RSD)
Fe	22.9	(10.4 %RSD)
Gd	< 3.77	(N/A %RSD)
K	< 170	(N/A %RSD)
La	< 2.05	(N/A %RSD)
Li	< 9.16	(N/A %RSD)
Mg	1060	(10.1 %RSD)
Mn	20.4	(10.8 %RSD)
Mo	< 18.5	(N/A %RSD)
Na	1290	(10 %RSD)
Ni	< 27.5	(N/A %RSD)
P	< 64.4	(N/A %RSD)
Pb	< 31.8	(N/A %RSD)
S	< 4850	(N/A %RSD)
Sb	< 154	(N/A %RSD)
Sn	< 49.2	(N/A %RSD)
Sr	10.4	(10.1 %RSD)
Th	< 19.6	(N/A %RSD)
Ti	26.9	(10.1 %RSD)
U	< 193	(N/A %RSD)
V	19.6	(11 %RSD)
Zn	9.76	(10.5 %RSD)
Zr	< 2.59	(N/A %RSD)

WIPP 15-5 Samples #2 and #3 by Digestion and ICP-ES (LIMS 300313911, 24 and 300314375, 77)

- Mixed Acid - Sample Preparation
  - Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away (Sample #2 solids sent for XRD analysis) and filtrate diluted to 50 mL; 0.1863g in 50mL (Sample #2); 0.1264g in 50mL (Sample #3)
  - 2-100x dilution in 2% nitric acid with Sc internal standard

- Peroxide Fusion - Sample Preparation
  - Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.2168g in 100mL (Sample #2); 0.0779g in 100mL (Sample #3)
  - 10-20x dilution in 2% nitric acid with Sc internal standard

- Instrument and Analysis Conditions
  - The instrument used was a Leeman Prodigy-XP Eschelle Grating simultaneous ICP-ES
  - Blank, blank, 5 and 10 mg/L calibration curve with Sc internal standard in 2% nitric acid

- Data Interpretation
  - Mg at 13 wt% in #2 and 21 wt% in #3
  - Na at 22 wt% in #2 and 6.6 wt% in #3
  - K poorly recovered in MA, present in PF blank, but at poor detection limit due to high sodium content (reported both ways)
  - Pb, Ca, Al, Si and Fe comparable (within 35%) between the different samples

WIPP 15-5 Samples #2 and #3 by Digestion and ICP-ES (LIMS 300313911, 24 and 300314375, 77)

- ICP-ES / Sodium Peroxide fusion (PF) except where noted as reported from Mixed (nitric+HF) acid dissolution (MA)

	USER_SAMPLEID:	#3 15_5	#2 15_5	
	SAMPLE_ID:	300314375, -377	300313911, -924	
	UNITS:	ug/g	ug/g	
Element				
Na		66400	217000	from MA [1]
Mg		210000	130000	
Pb		5680	7740	
K		1810 , 8090 (14400-6310)	6660 , 9140 (12300-3160)	MA, PF-bkg [2]
Ca		5120 , 5400 (11300-5900)	3260 , 3370 (5900-2530)	MA, PF-bkg [2]
Al		3130	2170	
Si		2470	1870	
Fe		1100	1160	

[1]Na is not obtainable in PF due to the sodium content of the reagent
[2]Mixed acid (MA) and Peroxide Fusion (PF) are reported for each method on K and Ca respectively. The PF analyses are substantially higher for K but comparable for Ca. PF analyses for both K and Ca are corrected for a significant contribution of these elements from the sodium peroxide reagent as shown above. K corrections are based on minimum detection levels and are not necessarily conservative.

WIPP 15-5 Samples #2 and #3 by Digestion and ICP-ES (LIMS 300313911, 24 and 300314375, 77)

- 15-5 #2 MA

ICP-OES Results

Description: WIPP UG 8/15, MIXED ACID DISS.
Travel Copy: 66301
Instrument: Leeman Prodigy ICP-ES
Reviewer: John Young
Comments:
Na from 100x all else 10x on dissolution

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		SAMPLE_2_MA	
SAMPLE_ID:	300313909		300313911	
UNITS:	ug/g		ug/g	
Element				
Ag	< 48.5	(N/A %RSD)	< 44.7	(N/A %RSD)
Al	< 154	(N/A %RSD)	217	(12.1 %RSD)
B	< 544	(N/A %RSD)	< 501	(N/A %RSD)
Ba	35	(11.4 %RSD)	38.5	(12.9 %RSD)
Be	< 3.5	(N/A %RSD)	< 3.23	(N/A %RSD)
Ca	< 18	(N/A %RSD)	3260	(10 %RSD)
Cd	< 31.8	(N/A %RSD)	< 29.3	(N/A %RSD)
Ce	< 280	(N/A %RSD)	< 259	(N/A %RSD)
Co	< 43.8	(N/A %RSD)	< 40.4	(N/A %RSD)
Cr	< 38.8	(N/A %RSD)	< 35.7	(N/A %RSD)
Cu	< 88.5	(N/A %RSD)	< 81.6	(N/A %RSD)
Fe	< 34.8	(N/A %RSD)	133	(10.3 %RSD)
Gd	< 111	(N/A %RSD)	< 102	(N/A %RSD)
K	< 977	(N/A %RSD)	6660	(10.6 %RSD)
La	< 49.3	(N/A %RSD)	< 45.4	(N/A %RSD)
Li	< 317	(N/A %RSD)	< 292	(N/A %RSD)
Mg	< 215	(N/A %RSD)	9150	(10.1 %RSD)
Mn	< 20	(N/A %RSD)	69.5	(10.9 %RSD)
Mo	< 448	(N/A %RSD)	< 414	(N/A %RSD)
Na	< 781	(N/A %RSD)	217000	(10.1 %RSD)
Ni	< 114	(N/A %RSD)	< 105	(N/A %RSD)
P	< 1180	(N/A %RSD)	4870	(11.6 %RSD)
Pb	< 3250	(N/A %RSD)	5090	(16.8 %RSD)
S	< 30000	(N/A %RSD)	< 27700	(N/A %RSD)
Sb	< 1030	(N/A %RSD)	< 948	(N/A %RSD)
Si	63500	(87.6 %RSD)	63500	(48.8 %RSD)
Sn	< 2330	(N/A %RSD)	< 2150	(N/A %RSD)
Sr	< 319	(N/A %RSD)	< 294	(N/A %RSD)
Th	< 290	(N/A %RSD)	< 268	(N/A %RSD)
Ti	< 23.3	(N/A %RSD)	94.1	(10.1 %RSD)
U	< 1750	(N/A %RSD)	< 1610	(N/A %RSD)
V	< 17.3	(N/A %RSD)	< 15.9	(N/A %RSD)
Zn	< 26.5	(N/A %RSD)	87	(10.7 %RSD)
Zr	< 15.5	(N/A %RSD)	< 14.3	(N/A %RSD)

WIPP 15-5 Samples #2 and #3 by Digestion and ICP-ES (LIMS 300313911, 24 and 300314375, 77)

- 15-5 #2 PF

ICP-OES Results

Description: WIPP UG 8/15, PEROXIDE FUSION DISS.
Travel Copy: 66302
Instrument: Leeman Prodigy ICP-ES
Reviewer: Mark Jones
Comments:

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		SAMPLE_2_PF	
SAMPLE_ID:	300313922		300313924	
UNITS:	ug/g		ug/g	
Element				
Ag	< 194	(N/A %RSD)	< 179	(N/A %RSD)
Al	< 435	(N/A %RSD)	2170	(10.1 %RSD)
B	< 156	(N/A %RSD)	< 144	(N/A %RSD)
Ba	< 66	(N/A %RSD)	89.5	(20.4 %RSD)
Be	< 14	(N/A %RSD)	< 12.9	(N/A %RSD)
Ca	2530	(10 %RSD)	5900	(10 %RSD)
Cd	< 104	(N/A %RSD)	< 95.9	(N/A %RSD)
Ce	< 418	(N/A %RSD)	< 386	(N/A %RSD)
Co	< 122	(N/A %RSD)	< 113	(N/A %RSD)
Cr	< 93	(N/A %RSD)	< 85.8	(N/A %RSD)
Cu	< 100	(N/A %RSD)	< 92.3	(N/A %RSD)
Fe	< 116	(N/A %RSD)	1160	(10 %RSD)
Gd	< 70	(N/A %RSD)	< 64.6	(N/A %RSD)
K	< 3160	(N/A %RSD)	12300	(13.2 %RSD)
La	< 38	(N/A %RSD)	< 35.1	(N/A %RSD)
Li	< 170	(N/A %RSD)	< 157	(N/A %RSD)
Mg	< 12	(N/A %RSD)	130000	(10 %RSD)
Mn	< 24	(N/A %RSD)	93.2	(14.3 %RSD)
Mo	< 344	(N/A %RSD)	< 317	(N/A %RSD)
Ni	< 455	(N/A %RSD)	< 420	(N/A %RSD)
P	< 1190	(N/A %RSD)	4380	(10.7 %RSD)
Pb	< 589	(N/A %RSD)	7740	(10.3 %RSD)
S	< 90000	(N/A %RSD)	< 83000	(N/A %RSD)
Sb	< 2860	(N/A %RSD)	< 2630	(N/A %RSD)
Si	< 327	(N/A %RSD)	1870	(10.2 %RSD)
Sn	< 912	(N/A %RSD)	< 841	(N/A %RSD)
Sr	20	(10 %RSD)	30.4	(10 %RSD)
Th	< 661	(N/A %RSD)	< 610	(N/A %RSD)
Ti	< 15	(N/A %RSD)	369	(10.1 %RSD)
U	< 3250	(N/A %RSD)	< 2990	(N/A %RSD)
V	< 69	(N/A %RSD)	< 63.7	(N/A %RSD)
Zn	< 106	(N/A %RSD)	115	(13.5 %RSD)

WIPP 15-5 Samples #2 and #3 by Digestion and ICP-ES (LIMS 300313911, 24 and 300314375, 77)

- 15-5 #3 MA

ICP-OES Results

Description: WIPP SAMPLE #3 (300313812) "15-5 SPARE"
Travel Copy: 66424
Instrument: Leeman Prodigy ICP-ES
Reviewer: John Young
Comments:
All from 2x, xc Na at 10x

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		WIPP #3 (SPARE 15_5)_MA	
SAMPLE_ID:	300314374		300314375	
UNITS:	ug/g		ug/g	
Element				
Ag	< 97	(N/A %RSD)	< 15.3	(N/A %RSD)
Al	< 309	(N/A %RSD)	68.7	(15.7 %RSD)
Ba	73.7	(20.2 %RSD)	46.1	(10.3 %RSD)
Be	< 7	(N/A %RSD)	< 1.11	(N/A %RSD)
Ca	79.5	(11.4 %RSD)	5120	(10 %RSD)
Cd	< 63.5	(N/A %RSD)	< 10	(N/A %RSD)
Ce	< 561	(N/A %RSD)	< 88.7	(N/A %RSD)
Co	< 87.5	(N/A %RSD)	< 13.8	(N/A %RSD)
Cr	< 77.5	(N/A %RSD)	< 12.3	(N/A %RSD)
Cu	< 177	(N/A %RSD)	< 28	(N/A %RSD)
Fe	< 69.5	(N/A %RSD)	16.7	(10.4 %RSD)
Gd	< 222	(N/A %RSD)	< 35.1	(N/A %RSD)
K	< 1950	(N/A %RSD)	1810	(11.4 %RSD)
La	< 98.5	(N/A %RSD)	< 15.6	(N/A %RSD)
Li	< 634	(N/A %RSD)	< 100	(N/A %RSD)
Mg	< 431	(N/A %RSD)	918	(10.3 %RSD)
Mn	< 40	(N/A %RSD)	65.6	(10.3 %RSD)
Mo	< 897	(N/A %RSD)	< 142	(N/A %RSD)
Na	< 1560	(N/A %RSD)	66400	(10 %RSD)
Ni	< 228	(N/A %RSD)	36.5	(17.5 %RSD)
P	< 2370	(N/A %RSD)	1230	(11.2 %RSD)
Pb	< 6500	(N/A %RSD)	4050	(12.3 %RSD)
S	< 60000	(N/A %RSD)	< 9490	(N/A %RSD)
Sb	< 2050	(N/A %RSD)	< 325	(N/A %RSD)
Sn	< 4650	(N/A %RSD)	< 736	(N/A %RSD)
Sr	< 638	(N/A %RSD)	< 101	(N/A %RSD)
Th	< 581	(N/A %RSD)	95.9	(10.4 %RSD)
Ti	< 46.5	(N/A %RSD)	340	(10 %RSD)
U	< 3500	(N/A %RSD)	< 553	(N/A %RSD)
V	< 34.5	(N/A %RSD)	6.17	(10.8 %RSD)
Zn	< 53	(N/A %RSD)	75.2	(10.2 %RSD)
Zr	< 31	(N/A %RSD)	< 4.91	(N/A %RSD)

WIPP 15-5 Samples #2 and #3 by Digestion and ICP-ES (LIMS 300313911, 24 and 300314375, 77)

- 15-5 #3 PF

ICP-OES Results

Description:	WIPP SAMPLE #3 (300313812) "15-5 SPARE"
Travel Copy:	66425
Instrument:	Leeman Prodigy ICP-ES
Reviewer:	John Young
Comments:	
	20x dilution of fusion; 10x file corrupted and discarded

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		WIPP #3 (SPARE 15_5)_PF	
SAMPLE_ID:	300314376		300314377	
UNITS:	ug/g		ug/g	
Element				
Al	1370	(11.6 %RSD)	3130	(10.5 %RSD)
B	< 718	(N/A %RSD)	< 922	(N/A %RSD)
Ba	160	(25.3 %RSD)	172	(64.1 %RSD)
Be	< 28	(N/A %RSD)	< 35.9	(N/A %RSD)
Ca	5900	(10 %RSD)	11300	(10 %RSD)
Ce	< 1870	(N/A %RSD)	< 2400	(N/A %RSD)
Co	< 244	(N/A %RSD)	< 313	(N/A %RSD)
Cr	< 310	(N/A %RSD)	< 398	(N/A %RSD)
Cu	< 160	(N/A %RSD)	< 205	(N/A %RSD)
Fe	< 278	(N/A %RSD)	1100	(14.6 %RSD)
K	< 6310	(N/A %RSD)	14400	(26.1 %RSD)
La	< 164	(N/A %RSD)	< 211	(N/A %RSD)
Li	< 340	(N/A %RSD)	< 436	(N/A %RSD)
Mg	< 22	(N/A %RSD)	210000	(10 %RSD)
Mn	< 160	(N/A %RSD)	< 205	(N/A %RSD)
Mo	< 556	(N/A %RSD)	3560	(10.5 %RSD)
Ni	< 1020	(N/A %RSD)	< 1310	(N/A %RSD)
P	< 2390	(N/A %RSD)	< 3070	(N/A %RSD)
Pb	< 1180	(N/A %RSD)	5680	(10.6 %RSD)
S	< 2E+05	(N/A %RSD)	< 3E+05	(N/A %RSD)
Si	< 654	(N/A %RSD)	2470	(14.2 %RSD)
Sn	< 1820	(N/A %RSD)	< 2340	(N/A %RSD)
Sr	36	(10 %RSD)	53.9	(10 %RSD)
Th	< 1320	(N/A %RSD)	< 1700	(N/A %RSD)
Ti	< 186	(N/A %RSD)	298	(10.6 %RSD)
U	< 7200	(N/A %RSD)	< 9240	(N/A %RSD)
V	< 138	(N/A %RSD)	< 177	(N/A %RSD)
Zn	< 130	(N/A %RSD)	< 167	(N/A %RSD)

LANL Parent Drum Debris by Digestion and ICP-ES (LIMS 300313736, 741, 743)

- Mixed Acid - Sample Preparation
  - Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL; 0.6968g in 50mL
  - 2-100x dilution in 2% nitric acid with Sc internal standard

- Peroxide Fusion - Sample Preparation
  - Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.4317g in 100mL
  - 10-20x dilution in 2% nitric acid with Sc internal standard

- Water Leach - Sample Preparation
  - H_2O leach in closed vessel for four days, followed by filtration and removal of solids prior to analysis; 0.6901g in 20mL. The solution was also used for both IC and titration methods

- Instrument and Analysis Conditions
  - The instrument used was a Leeman Prodigy-XP Eschelle Grating simultaneous ICP-ES
  - Blank, blank, 5 and 10 mg/L calibration curve with Sc internal standard in 2% nitric acid

- Data Interpretation
  - Pb at 11.7 wt%
  - Na at 2.8 wt%, noteworthy that best recovery of Na was from water leach
  - Ca and K reeported from MA, present in PF blank, but at poor detection limit due to high sodium content (reported both ways)
  - Pb, Ca, Al, Si and Fe comparable (within 35%) between the different samples

LANL Parent Drum Debris by Digestion and ICP-ES (LIMS 300313736, 741, 743)

- ICP-ES / Peroxide fusion except where noted

	USER_SAMPLEID:	WIPP Parent Drum Debris	
	SAMPLE_ID:	300313736, -741	
	UNITS:	ug/g	
Element			
Pb		117000	
Na		28100	from water dissolution
Al		9050	
Fe		7300	
Mg		5010	
U		3540	
K		1270	from mixed acid (K elevated in PF blank)
Ca		573	from mixed acid (Ca elevated in PF blank)

LANL Parent Drum Debris by Digestion and ICP-ES (LIMS 300313736, 741, 743)

- LANL Parent Drum Debris MA

ICP-OES Results

Description: WIPP LANL PARENT DRUM SOLID DEBRIS
Travel Copy: 66285
Instrument: Leeman Prodigy ICP-ES
Reviewer: Mark Jones
Comments:

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		69120_B_MA	
SAMPLE_ID:	300313735		300313736	
UNITS:	ug/g		ug/g	
Element				
Ag	< 21	(N/A %RSD)	< 21.1	(N/A %RSD)
Al	< 88.1	(N/A %RSD)	2450	(10 %RSD)
Ba	< 14.3	(N/A %RSD)	< 14.4	(N/A %RSD)
Be	< 2	(N/A %RSD)	< 2.01	(N/A %RSD)
Ca	< 7	(N/A %RSD)	573	(10 %RSD)
Cd	< 14.9	(N/A %RSD)	< 14.9	(N/A %RSD)
Ce	< 59.7	(N/A %RSD)	< 60	(N/A %RSD)
Co	< 17.4	(N/A %RSD)	< 17.5	(N/A %RSD)
Cr	< 22.1	(N/A %RSD)	843	(10 %RSD)
Cu	< 31.3	(N/A %RSD)	290	(10.1 %RSD)
Fe	< 19.9	(N/A %RSD)	2880	(10 %RSD)
Gd	< 39	(N/A %RSD)	< 39.2	(N/A %RSD)
K	< 451	(N/A %RSD)	1270	(10.3 %RSD)
La	< 11.7	(N/A %RSD)	< 11.8	(N/A %RSD)
Li	< 24.3	(N/A %RSD)	< 24.4	(N/A %RSD)
Mg	< 1.57	(N/A %RSD)	112	(10 %RSD)
Mn	< 2.71	(N/A %RSD)	12.9	(11.2 %RSD)
Mo	< 39.7	(N/A %RSD)	< 39.9	(N/A %RSD)
Na	< 64.4	(N/A %RSD)	11200	(10 %RSD)
Ni	< 73	(N/A %RSD)	343	(11.1 %RSD)
P	< 200	(N/A %RSD)	< 201	(N/A %RSD)
Pb	< 84.1	(N/A %RSD)	86100	(10 %RSD)
S	< 12900	(N/A %RSD)	< 12900	(N/A %RSD)
Sb	< 408	(N/A %RSD)	< 410	(N/A %RSD)
Sn	< 130	(N/A %RSD)	< 131	(N/A %RSD)
Sr	< 1.43	(N/A %RSD)	1.58	(10 %RSD)
Th	< 79.9	(N/A %RSD)	< 80.2	(N/A %RSD)
Ti	< 2.14	(N/A %RSD)	14.1	(10.3 %RSD)
U	< 514	(N/A %RSD)	5650	(10.3 %RSD)
V	< 9.86	(N/A %RSD)	< 9.9	(N/A %RSD)
Zn	< 9.29	(N/A %RSD)	54.1	(10.6 %RSD)
Zr	< 6.86	(N/A %RSD)	< 6.89	(N/A %RSD)

LANL Parent Drum Debris by Digestion and ICP-ES (LIMS 300313736, 741, 743)

- LANL Parent Drum Debris PF

ICP-OES Results

Description: WIPP LANL PARENT DRUM SOLID DEBRIS
Travel Copy: 66287
Instrument: Leeman Prodigy ICP-ES
Reviewer: Mark Jones
Comments:

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		69120_B_PF	
SAMPLE_ID:	300313740		300313741	
UNITS:	ug/g		ug/g	
Element				
Ag	< 97	(N/A %RSD)	< 89.9	(N/A %RSD)
Al	< 309	(N/A %RSD)	9050	(10 %RSD)
B	< 78	(N/A %RSD)	265	(10 %RSD)
Ba	< 50	(N/A %RSD)	1610	(10 %RSD)
Be	< 7	(N/A %RSD)	< 6.49	(N/A %RSD)
Ca	1620	(10 %RSD)	3750	(10 %RSD)
Cd	< 52	(N/A %RSD)	< 48.2	(N/A %RSD)
Ce	< 468	(N/A %RSD)	< 434	(N/A %RSD)
Co	< 61	(N/A %RSD)	< 56.5	(N/A %RSD)
Cr	< 77.5	(N/A %RSD)	457	(10 %RSD)
Cu	< 40	(N/A %RSD)	216	(10 %RSD)
Fe	< 40	(N/A %RSD)	7300	(10 %RSD)
Gd	< 35	(N/A %RSD)	< 32.4	(N/A %RSD)
K	3480	(29.6 %RSD)	8210	(10.4 %RSD)
La	< 41	(N/A %RSD)	< 38	(N/A %RSD)
Li	< 85	(N/A %RSD)	< 78.8	(N/A %RSD)
Mg	< 10	(N/A %RSD)	5010	(10 %RSD)
Mn	< 12	(N/A %RSD)	23.2	(176 %RSD)
Mo	< 139	(N/A %RSD)	< 129	(N/A %RSD)
Ni	< 247	(N/A %RSD)	408	(11.1 %RSD)
P	< 597	(N/A %RSD)	< 553	(N/A %RSD)
Pb	< 295	(N/A %RSD)	117000	(10 %RSD)
S	< 45000	(N/A %RSD)	< 41700	(N/A %RSD)
Sb	< 1430	(N/A %RSD)	< 1320	(N/A %RSD)
Si	< 164	(N/A %RSD)	403	(13.7 %RSD)
Sn	< 456	(N/A %RSD)	< 423	(N/A %RSD)
Sr	11.5	(10 %RSD)	19	(10 %RSD)
Th	< 331	(N/A %RSD)	< 306	(N/A %RSD)
Ti	< 28	(N/A %RSD)	< 25.9	(N/A %RSD)
U	< 1800	(N/A %RSD)	3540	(11.1 %RSD)
V	< 34.5	(N/A %RSD)	< 32	(N/A %RSD)
Zn	< 30	(N/A %RSD)	643	(10 %RSD)

LANL Parent Drum Debris by Digestion and ICP-ES (LIMS 300313736, 741, 743)

- LANL Parent Drum Debris Water Leach

ICP-OES Results

Description: WIPP LANL PARENT DRUM SOLID DEBRIS
Travel Copy: 66288
Instrument: Leeman Prodigy ICP-ES
Reviewer: Mark Jones
Comments:

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		69120_B_IC	
SAMPLE_ID:	300313742		300313743	
UNITS:	ug/g		ug/g	
Element				
Ag	< 8.4	(N/A %RSD)	< 8.52	(N/A %RSD)
Al	< 35.3	(N/A %RSD)	1870	(10 %RSD)
B	< 8.91	(N/A %RSD)	53.3	(10 %RSD)
Ba	< 3.77	(N/A %RSD)	< 3.83	(N/A %RSD)
Be	< 1.6	(N/A %RSD)	< 1.62	(N/A %RSD)
Ca	< 4.11	(N/A %RSD)	2020	(10 %RSD)
Cd	< 7.26	(N/A %RSD)	< 7.36	(N/A %RSD)
Ce	< 23.9	(N/A %RSD)	< 24.2	(N/A %RSD)
Co	< 6.97	(N/A %RSD)	< 7.07	(N/A %RSD)
Cr	< 6.34	(N/A %RSD)	82.8	(10.1 %RSD)
Cu	< 4.57	(N/A %RSD)	60.2	(10 %RSD)
Fe	< 7.94	(N/A %RSD)	260	(10 %RSD)
Gd	< 4	(N/A %RSD)	< 4.06	(N/A %RSD)
K	< 180	(N/A %RSD)	2140	(10.4 %RSD)
La	< 4.69	(N/A %RSD)	< 4.75	(N/A %RSD)
Li	< 19.4	(N/A %RSD)	< 19.7	(N/A %RSD)
Mg	< 1.26	(N/A %RSD)	3930	(10 %RSD)
Mn	< 1.37	(N/A %RSD)	19.3	(10.5 %RSD)
Mo	< 19.7	(N/A %RSD)	< 19.9	(N/A %RSD)
Na	< 25.8	(N/A %RSD)	28100	(10 %RSD)
Ni	< 28.2	(N/A %RSD)	109	(10 %RSD)
P	< 68.2	(N/A %RSD)	< 69.2	(N/A %RSD)
Pb	< 33.7	(N/A %RSD)	36300	(10 %RSD)
S	< 5140	(N/A %RSD)	< 5220	(N/A %RSD)
Sb	< 27.3	(N/A %RSD)	< 27.6	(N/A %RSD)
Si	< 18.7	(N/A %RSD)	< 19	(N/A %RSD)
Sn	< 52.1	(N/A %RSD)	< 52.9	(N/A %RSD)
Sr	< 0.686	(N/A %RSD)	3.36	(10 %RSD)
Th	< 20.8	(N/A %RSD)	< 21.1	(N/A %RSD)
Ti	< 3.2	(N/A %RSD)	< 3.25	(N/A %RSD)
U	< 206	(N/A %RSD)	547	(10.4 %RSD)
V	< 3.94	(N/A %RSD)	< 4	(N/A %RSD)
Zn	< 3.71	(N/A %RSD)	23.1	(10.3 %RSD)
Zr	< 3.54	(N/A %RSD)	< 3.59	(N/A %RSD)

LANL Parent Drum IAEA Swipe by Digestion and ICP-ES (LIMS 300313726)

- Mixed Acid - Sample Preparation
  - Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL; 0.5218g in 50mL
  - 2-100x dilution in 2% nitric acid with Sc internal standard

- Instrument and Analysis Conditions
  - The instrument used was a Leeman Prodigy-XP Eschelle Grating simultaneous ICP-ES
  - Blank, blank, 5 and 10 mg/L calibration curve with Sc internal standard in 2% nitric acid

- Data Interpretation
  - All elements below minimum detection level except when also observed in the blank swipe (H column)
  - No valuable data
  - Results units were normalized to weight of filter, in order to discern if any non-filter elemental components were present

LANL Parent Drum IAEA Swipe by Digestion and ICP-ES (LIMS 300313726)

- ICP-ES: IAEA Swipe Sample MA

ICP-OES Results

Description: WIPP LANL PARENT DRUM IAEA SWIPE
Travel Copy: 66283
Instrument: Leeman Prodigy ICP-ES
Reviewer: John Young
Comments:
all from 2x dilution of dissolution, reported per mass of sample

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		69120_A_MA		69120_H_MA	
SAMPLE_ID:	300313725		300313726		300313729	
UNITS:	ug/g		ug/g		ug/g	
Element						
Ag	< 3.88	(N/A %RSD)	< 3.72	(N/A %RSD)	< 3.77	(N/A %RSD)
Al	22.1	(14.2 %RSD)	34.6	(11.6 %RSD)	31.3	(13 %RSD)
B	< 43.5	(N/A %RSD)	< 41.7	(N/A %RSD)	< 42.2	(N/A %RSD)
Ba	14.5	(10.2 %RSD)	14.8	(10.1 %RSD)	14.7	(10 %RSD)
Be	< 0.28	(N/A %RSD)	< 0.27	(N/A %RSD)	< 0.27	(N/A %RSD)
Ca	2.85	(11.2 %RSD)	195	(10 %RSD)	191	(10 %RSD)
Cd	< 2.54	(N/A %RSD)	< 2.43	(N/A %RSD)	< 2.47	(N/A %RSD)
Ce	< 22.4	(N/A %RSD)	< 21.5	(N/A %RSD)	< 21.8	(N/A %RSD)
Co	< 3.5	(N/A %RSD)	< 3.35	(N/A %RSD)	< 3.4	(N/A %RSD)
Cr	< 3.1	(N/A %RSD)	< 2.97	(N/A %RSD)	< 3.01	(N/A %RSD)
Cu	< 7.08	(N/A %RSD)	< 6.78	(N/A %RSD)	< 6.88	(N/A %RSD)
Fe	7.18	(11.4 %RSD)	29.1	(10.1 %RSD)	18.7	(10.5 %RSD)
Gd	< 8.88	(N/A %RSD)	< 8.51	(N/A %RSD)	< 8.62	(N/A %RSD)
K	< 78.2	(N/A %RSD)	< 74.9	(N/A %RSD)	< 75.9	(N/A %RSD)
La	< 3.94	(N/A %RSD)	< 3.78	(N/A %RSD)	< 3.83	(N/A %RSD)
Li	< 25.4	(N/A %RSD)	< 24.3	(N/A %RSD)	< 24.6	(N/A %RSD)
Mg	< 17.2	(N/A %RSD)	224	(10.3 %RSD)	211	(10.2 %RSD)
Mn	< 1.6	(N/A %RSD)	2.15	(19.6 %RSD)	2.09	(19.6 %RSD)
Mo	< 35.9	(N/A %RSD)	< 34.4	(N/A %RSD)	< 34.8	(N/A %RSD)
Na	71.7	(30.6 %RSD)	181	(18.4 %RSD)	131	(17.5 %RSD)
Ni	< 9.1	(N/A %RSD)	< 8.72	(N/A %RSD)	< 8.84	(N/A %RSD)
P	< 94.6	(N/A %RSD)	< 90.6	(N/A %RSD)	< 91.9	(N/A %RSD)
Pb	< 260	(N/A %RSD)	< 249	(N/A %RSD)	< 253	(N/A %RSD)
S	< 2400	(N/A %RSD)	< 2300	(N/A %RSD)	< 2330	(N/A %RSD)
Sb	< 82.2	(N/A %RSD)	< 78.7	(N/A %RSD)	< 79.8	(N/A %RSD)
Si	9290	(32.9 %RSD)	8150	(25.6 %RSD)	8370	(15.7 %RSD)
Sn	< 186	(N/A %RSD)	< 178	(N/A %RSD)	< 181	(N/A %RSD)
Sr	< 25.5	(N/A %RSD)	< 24.5	(N/A %RSD)	< 24.8	(N/A %RSD)
Th	< 23.2	(N/A %RSD)	< 22.2	(N/A %RSD)	< 22.6	(N/A %RSD)
Ti	1.91	(13 %RSD)	3.31	(11.5 %RSD)	2.4	(11 %RSD)
U	< 140	(N/A %RSD)	< 134	(N/A %RSD)	< 136	(N/A %RSD)
V	< 1.38	(N/A %RSD)	< 1.32	(N/A %RSD)	< 1.34	(N/A %RSD)
Zn	< 2.12	(N/A %RSD)	2.58	(13.6 %RSD)	2.34	(13.4 %RSD)
Zr	< 1.24	(N/A %RSD)	< 1.19	(N/A %RSD)	< 1.2	(N/A %RSD)

WIPP CAM Filters 4, 6 and 9 by Digestion and ICP-ES (LIMS 300313598-600)

- Mixed Acid - Sample Preparation
  - Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL; 0.0284g in 50 mL (CAM #4) ; 0.0249g in 50 mL (CAM #6) ; 0.0328g in 50 mL (CAM #9)
  - 20x dilution in 2% nitric acid with Sc internal standard

- Peroxide Fusion - Sample Preparation
  - Na2O2 fusion at 675 °C in Zr crucible; dissolve flux with HNO3; 0.0247g in 50 mL (CAM #4) ; 0.0365g in 50 mL (CAM #6) ; 0.0379g in 50 mL (CAM #9)
  - 20x dilution in 2% nitric acid with Sc internal standard

- Instrument and Analysis Conditions
  - The instrument used was a Leeman Prodigy-XP Eschelle Grating simultaneous ICP-ES
  - Blank, blank, 5 and 10 mg/L calibration curve with Sc internal standard in 2% nitric acid

- Data Interpretation
  - Contained approximately 10 mg of ICP-ES analytes. Predominant elements above 1000 µg were Na, Pb, Mg. Traces of Fe, Ca and Al were above 100 µg)
  - Nothing noteworthy over previous reports

WIPP CAM Filters 4, 6 and 9 by Digestion and ICP-ES (LIMS 300313598-600)

- Major elements: ICP-ES / Peroxide fusion except where noted

	SAMPLEID:	WIPP BLANK	CAM 4	CAM 6	CAM 9	
	AD#:	300313601	300313602	300313603	300313604	
	UNITS:	ug/filter	ug/filter	ug/filter	ug/filter	
Element						
Na		213	6390	5350	7490	from mixed acid
Pb		< 471	1830	2370	2020	
Mg		133	706	1000	1000	
Fe		144	241	154	195	
Ca		9.55	193	161	181	from mixed acid
Al		< 43.5	140	102	114	from mixed acid

WIPP CAM Filters 4, 6 and 9 by Digestion and ICP-ES (LIMS 300313598-600)

- **ICP-ES / Peroxide fusion**

ICP-OES Results

Description: WIPP CAM FILTERS 4, 6, 9 PF DISS
Travel Copy: 66248
Instrument: Leeman Prodigy ICP-ES
Reviewer: John Young
Comments:
units expressed as ug/filter (1/4 digested to 100 ml)

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	WIPP BLANK PF		CAM FILTER 4, SECTION PF		CAM FILTER 6, SECTION PF		CAM FILTER 9, SECTION PF	
SAMPLE_ID:	300313601		300313602		300313603		300313604	
UNITS:	ug/filter		ug/filter		ug/filter		ug/filter	
Element								
Ag	< 155	(N/A %RSD)	< 155	(N/A %RSD)	< 155	(N/A %RSD)	< 155	(N/A %RSD)
Al	661	(22.3 %RSD)	711	(13 %RSD)	483	(16.3 %RSD)	876	(14 %RSD)
B	< 1740	(N/A %RSD)	< 1740	(N/A %RSD)	< 1740	(N/A %RSD)	< 1740	(N/A %RSD)
Ba	74.4	(16.2 %RSD)	78.4	(12.8 %RSD)	88.8	(10.5 %RSD)	91.2	(16.2 %RSD)
Be	< 5.6	(N/A %RSD)	< 5.6	(N/A %RSD)	< 5.6	(N/A %RSD)	< 5.6	(N/A %RSD)
Ca	4640	(10 %RSD)	4680	(10 %RSD)	4630	(10 %RSD)	5050	(10 %RSD)
Cd	< 102	(N/A %RSD)	< 102	(N/A %RSD)	< 102	(N/A %RSD)	< 102	(N/A %RSD)
Ce	< 828	(N/A %RSD)	< 828	(N/A %RSD)	< 828	(N/A %RSD)	< 828	(N/A %RSD)
Co	< 114	(N/A %RSD)	< 114	(N/A %RSD)	< 114	(N/A %RSD)	< 114	(N/A %RSD)
Cr	< 74.4	(N/A %RSD)	< 74.4	(N/A %RSD)	< 74.4	(N/A %RSD)	< 74.4	(N/A %RSD)
Cu	< 64	(N/A %RSD)	< 64	(N/A %RSD)	< 64	(N/A %RSD)	< 64	(N/A %RSD)
Fe	144	(13.6 %RSD)	241	(11.7 %RSD)	154	(13.6 %RSD)	195	(11.9 %RSD)
Gd	< 56	(N/A %RSD)	< 56	(N/A %RSD)	< 56	(N/A %RSD)	< 56	(N/A %RSD)
K	8020	(27.5 %RSD)	9580	(21.6 %RSD)	10100	(23.8 %RSD)	11000	(15.5 %RSD)
La	< 158	(N/A %RSD)	< 158	(N/A %RSD)	< 158	(N/A %RSD)	< 158	(N/A %RSD)
Li	< 136	(N/A %RSD)	< 136	(N/A %RSD)	< 136	(N/A %RSD)	< 136	(N/A %RSD)
Mg	133	(10 %RSD)	706	(10 %RSD)	1000	(10 %RSD)	1000	(10.4 %RSD)
Mn	< 64	(N/A %RSD)	< 64	(N/A %RSD)	< 64	(N/A %RSD)	< 64	(N/A %RSD)
Mo	< 275	(N/A %RSD)	< 275	(N/A %RSD)	< 275	(N/A %RSD)	< 275	(N/A %RSD)
Ni	< 395	(N/A %RSD)	< 395	(N/A %RSD)	< 395	(N/A %RSD)	< 395	(N/A %RSD)
P	< 955	(N/A %RSD)	< 955	(N/A %RSD)	< 955	(N/A %RSD)	< 955	(N/A %RSD)
Pb	< 471	(N/A %RSD)	1830	(12.6 %RSD)	2370	(13 %RSD)	2020	(10.8 %RSD)
S	< 96000	(N/A %RSD)	< 96000	(N/A %RSD)	< 96000	(N/A %RSD)	< 96000	(N/A %RSD)
Sb	< 3290	(N/A %RSD)	< 3290	(N/A %RSD)	< 3290	(N/A %RSD)	< 3290	(N/A %RSD)
Si	< 262	(N/A %RSD)	< 262	(N/A %RSD)	294	(90.5 %RSD)	743	(41.9 %RSD)
Sn	< 730	(N/A %RSD)	< 730	(N/A %RSD)	< 730	(N/A %RSD)	< 730	(N/A %RSD)
Sr	28.4	(10.1 %RSD)	27.6	(10.1 %RSD)	27.2	(10 %RSD)	28.4	(10.2 %RSD)
Th	< 929	(N/A %RSD)	< 929	(N/A %RSD)	< 929	(N/A %RSD)	< 929	(N/A %RSD)
Ti	< 12	(N/A %RSD)	27.2	(10.1 %RSD)	52	(10.1 %RSD)	51.2	(10.5 %RSD)
U	< 2880	(N/A %RSD)	< 2880	(N/A %RSD)	< 2880	(N/A %RSD)	< 2880	(N/A %RSD)
V	< 55.2	(N/A %RSD)	< 55.2	(N/A %RSD)	< 55.2	(N/A %RSD)	< 55.2	(N/A %RSD)
Zn	< 52	(N/A %RSD)	< 52	(N/A %RSD)	< 52	(N/A %RSD)	< 52	(N/A %RSD)

Al, Ba, Ca and K in peroxide reagent

WIPP CAM Filters 4, 6 and 9 by Digestion and ICP-ES (LIMS 300313598-600)

- **ICP-ES / Mixed Acid**

ICP-OES Results

Description: WIPP CAM FILTERS 4, 6, 9 MA DISS
Travel Copy: 66247
Instrument: Leeman Prodigy ICP-ES
Reviewer: John Young
Comments:
results expressed as ug/filter (1/4 analyzed)

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	WIPP BLANK MA		CAM FILTER 4, SECTION MA		CAM FILTER 6, SECTION MA		CAM FILTER 9, SECTION MA	
SAMPLE_ID:	300313597		300313598		300313599		300313600	
UNITS:	ug/filter		ug/filter		ug/filter		ug/filter	
Element								
Ag	< 19.4	(N/A %RSD)	< 19.4	(N/A %RSD)	< 19.4	(N/A %RSD)	< 19.4	(N/A %RSD)
Al	< 43.5	(N/A %RSD)	140	(14.1 %RSD)	102	(10.5 %RSD)	114	(10.1 %RSD)
B	< 217	(N/A %RSD)	< 217	(N/A %RSD)	< 217	(N/A %RSD)	< 217	(N/A %RSD)
Ba	46.3	(10.1 %RSD)	29.1	(10.2 %RSD)	30.3	(10.2 %RSD)	30	(10.8 %RSD)
Be	< 0.7	(N/A %RSD)	< 0.7	(N/A %RSD)	< 0.7	(N/A %RSD)	< 0.7	(N/A %RSD)
Ca	9.55	(14.1 %RSD)	193	(10.1 %RSD)	161	(10.2 %RSD)	181	(10.1 %RSD)
Cd	< 12.7	(N/A %RSD)	< 12.7	(N/A %RSD)	< 12.7	(N/A %RSD)	< 12.7	(N/A %RSD)
Ce	< 104	(N/A %RSD)	< 104	(N/A %RSD)	< 104	(N/A %RSD)	< 104	(N/A %RSD)
Co	< 14.3	(N/A %RSD)	< 14.3	(N/A %RSD)	< 14.3	(N/A %RSD)	< 14.3	(N/A %RSD)
Cr	< 9.3	(N/A %RSD)	< 9.3	(N/A %RSD)	< 9.3	(N/A %RSD)	< 9.3	(N/A %RSD)
Cu	< 8	(N/A %RSD)	< 8	(N/A %RSD)	< 8	(N/A %RSD)	< 8	(N/A %RSD)
Fe	14.2	(10.1 %RSD)	56.4	(10.2 %RSD)	37.8	(10 %RSD)	33.7	(10.2 %RSD)
Gd	< 7	(N/A %RSD)	< 7	(N/A %RSD)	< 7	(N/A %RSD)	< 7	(N/A %RSD)
K	< 391	(N/A %RSD)	558	(13.1 %RSD)	< 391	(N/A %RSD)	< 391	(N/A %RSD)
La	< 19.7	(N/A %RSD)	< 19.7	(N/A %RSD)	< 19.7	(N/A %RSD)	< 19.7	(N/A %RSD)
Li	< 17	(N/A %RSD)	< 17	(N/A %RSD)	< 17	(N/A %RSD)	< 17	(N/A %RSD)
Mg	6.45	(10.2 %RSD)	384	(10 %RSD)	314	(10 %RSD)	386	(10 %RSD)
Mn	< 8	(N/A %RSD)	< 8	(N/A %RSD)	< 8	(N/A %RSD)	< 8	(N/A %RSD)
Mo	< 34.4	(N/A %RSD)	< 34.4	(N/A %RSD)	< 34.4	(N/A %RSD)	< 34.4	(N/A %RSD)
Na	213	(10 %RSD)	6390	(10 %RSD)	5350	(10 %RSD)	7490	(10 %RSD)
Ni	< 45.5	(N/A %RSD)	< 45.5	(N/A %RSD)	< 45.5	(N/A %RSD)	< 45.5	(N/A %RSD)
P	< 119	(N/A %RSD)	185	(17.9 %RSD)	176	(19.6 %RSD)	182	(13.6 %RSD)
Pb	< 503	(N/A %RSD)	935	(16.6 %RSD)	687	(12.9 %RSD)	1050	(12.1 %RSD)
S	< 12000	(N/A %RSD)	< 12000	(N/A %RSD)	< 12000	(N/A %RSD)	< 12000	(N/A %RSD)
Sb	< 411	(N/A %RSD)	< 411	(N/A %RSD)	< 411	(N/A %RSD)	< 411	(N/A %RSD)
Sn	< 91.2	(N/A %RSD)	< 91.2	(N/A %RSD)	< 91.2	(N/A %RSD)	< 91.2	(N/A %RSD)
Sr	< 1.2	(N/A %RSD)	< 1.2	(N/A %RSD)	< 1.2	(N/A %RSD)	< 1.2	(N/A %RSD)
Th	< 116	(N/A %RSD)	< 116	(N/A %RSD)	< 116	(N/A %RSD)	< 116	(N/A %RSD)
Ti	16	(14.9 %RSD)	35.7	(10.6 %RSD)	26.5	(10 %RSD)	26.2	(10.1 %RSD)
U	< 360	(N/A %RSD)	< 360	(N/A %RSD)	< 360	(N/A %RSD)	< 360	(N/A %RSD)
V	< 6.9	(N/A %RSD)	< 6.9	(N/A %RSD)	< 6.9	(N/A %RSD)	< 6.9	(N/A %RSD)
Zn	< 6.5	(N/A %RSD)	26	(10.2 %RSD)	12	(10.1 %RSD)	17.8	(10.4 %RSD)

WIPP Non Deployed Swheat by Digestion and ICP-ES (LIMS 30031433, 335)

- Mixed Acid - Sample Preparation
  - Hot Mixed Acid-6 hr. heating at 115 °C in sealed Teflon vessel with 4 mL HNO_3/2 mL HCl/2mL HF. Solids filtered away and filtrate diluted to 50 mL; 0.3930g in 100mL
  - 20x dilution in 2% nitric acid with Sc internal standard

- Peroxide Fusion - Sample Preparation
  - Na_2O_2 fusion at 675 °C in Zr crucible; dissolve flux with HNO_3; 0.3012g in 100mL
  - 20x dilution in 2% nitric acid with Sc internal standard

- Instrument and Analysis Conditions
  - The instrument used was a Leeman Prodigy-XP Eschelle Grating simultaneous ICP-ES
  - Blank, blank, 5 and 10 mg/L calibration curve with Sc internal standard in 2% nitric acid

- Data Interpretation
  - Contained 16.4 wt% ICP-ES analytes (B, Na, Al, K, P, Mg and Ca all above 1000 µg/g)
  - Nothing noteworthy unless significant for salt ratios

WIPP Non Deployed Swheat by Digestion and ICP-ES (LIMS 30031433, 335)

- ICP-ES / Peroxide Fusion vs Mixed Acid selected for best recovery

	USER_SAMPLEID:	UNUSED SWHEAT	Dissolution
	SAMPLE_ID:	300314333,-335	Selected
	UNITS:	ug/g	
Element			
B		69600	MA
Na		56400	MA
Al		22200	MA
K		8870	PF
P		4590	PF
Mg		1700	PF
Ca		1310	MA

WIPP Non Deployed Swheat by Digestion and ICP-ES (LIMS 30031433, 335)

- Unused Swheat MA

ICP-OES Results

Description: UNUSED SWHEAT, MIXED ACID DISS
Travel Copy: 66418
Instrument: Leeman Prodigy ICP-ES
Reviewer: Mark Jones
Comments:

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		UNUSED SWHEAT _MA	
SAMPLE_ID:	300314334		300314335	
UNITS:	ug/g		ug/g	
Element				
Ag	< 36.8	(N/A %RSD)	< 37.4	(N/A %RSD)
Al	< 154	(N/A %RSD)	22200	(10 %RSD)
B	< 89.8	(N/A %RSD)	69600	(10 %RSD)
Ba	< 25	(N/A %RSD)	< 25.4	(N/A %RSD)
Be	< 1.75	(N/A %RSD)	< 1.78	(N/A %RSD)
Ca	< 12.3	(N/A %RSD)	1310	(10 %RSD)
Cd	< 26	(N/A %RSD)	< 26.5	(N/A %RSD)
Ce	< 105	(N/A %RSD)	< 106	(N/A %RSD)
Co	< 30.5	(N/A %RSD)	< 31	(N/A %RSD)
Cr	< 23.3	(N/A %RSD)	< 23.7	(N/A %RSD)
Cu	< 20	(N/A %RSD)	< 20.4	(N/A %RSD)
Fe	< 34.8	(N/A %RSD)	396	(10.2 %RSD)
Gd	< 17.5	(N/A %RSD)	< 17.8	(N/A %RSD)
K	< 789	(N/A %RSD)	4480	(10 %RSD)
La	< 20.5	(N/A %RSD)	< 20.9	(N/A %RSD)
Li	< 42.5	(N/A %RSD)	< 43.3	(N/A %RSD)
Mg	< 4	(N/A %RSD)	515	(10 %RSD)
Mn	< 6	(N/A %RSD)	44.3	(11.6 %RSD)
Mo	< 69.5	(N/A %RSD)	< 70.7	(N/A %RSD)
Na	< 500	(N/A %RSD)	56400	(10 %RSD)
Ni	< 114	(N/A %RSD)	< 116	(N/A %RSD)
P	< 299	(N/A %RSD)	4210	(10.1 %RSD)
Pb	< 147	(N/A %RSD)	< 150	(N/A %RSD)
S	< 22500	(N/A %RSD)	< 22900	(N/A %RSD)
Sb	< 714	(N/A %RSD)	< 727	(N/A %RSD)
Sn	< 228	(N/A %RSD)	< 232	(N/A %RSD)
Sr	< 3	(N/A %RSD)	6.62	(10 %RSD)
Th	< 91	(N/A %RSD)	< 92.6	(N/A %RSD)
Ti	< 14	(N/A %RSD)	117	(10.2 %RSD)
U	< 812	(N/A %RSD)	< 826	(N/A %RSD)
V	< 17.3	(N/A %RSD)	< 17.6	(N/A %RSD)
Zn	< 16.3	(N/A %RSD)	39.2	(11.8 %RSD)
Zr	< 15.5	(N/A %RSD)	744	(10.1 %RSD)

WIPP Non Deployed Swheat by Digestion and ICP-ES (LIMS 30031433, 335)

- Unused Swheat PF

ICP-OES Results

Description: UNUSED SWHEAT, PEROXIDE FUSION DISS
Travel Copy: 66417
Instrument: Leeman Prodigy ICP-ES
Reviewer: Mark Jones
Comments:

Method Detection Limit (MDL) = Instrument Detection Limit (IDL) x Dilution Factor.
Uncertainty is the RMS of the method uncertainty and the sample uncertainty.

USER_SAMPLEID:	ADS GENERATED BLANK		UNUSED SWHEAT - PF	
SAMPLE_ID:	300314332		300314333	
UNITS:	ug/g		ug/g	
Element				
Ag	< 129	(N/A %RSD)	< 129	(N/A %RSD)
Al	< 290	(N/A %RSD)	< 289	(N/A %RSD)
B	< 104	(N/A %RSD)	< 104	(N/A %RSD)
Ba	< 44	(N/A %RSD)	< 43.8	(N/A %RSD)
Be	< 4.67	(N/A %RSD)	< 4.65	(N/A %RSD)
Ca	1890	(10 %RSD)	2470	(10 %RSD)
Cd	< 69.3	(N/A %RSD)	< 69.1	(N/A %RSD)
Ce	< 558	(N/A %RSD)	< 556	(N/A %RSD)
Co	< 81.3	(N/A %RSD)	< 81	(N/A %RSD)
Cr	< 62	(N/A %RSD)	< 61.8	(N/A %RSD)
Cu	< 53.3	(N/A %RSD)	< 53.1	(N/A %RSD)
Fe	< 232	(N/A %RSD)	< 231	(N/A %RSD)
Gd	< 46.7	(N/A %RSD)	< 46.5	(N/A %RSD)
K	< 2100	(N/A %RSD)	8870	(13.1 %RSD)
La	< 54.7	(N/A %RSD)	< 54.4	(N/A %RSD)
Li	< 113	(N/A %RSD)	< 113	(N/A %RSD)
Mg	< 50	(N/A %RSD)	1700	(10 %RSD)
Mn	< 53.3	(N/A %RSD)	< 53.1	(N/A %RSD)
Mo	< 185	(N/A %RSD)	< 185	(N/A %RSD)
Ni	< 329	(N/A %RSD)	< 328	(N/A %RSD)
P	< 796	(N/A %RSD)	4590	(10.5 %RSD)
Pb	< 393	(N/A %RSD)	< 391	(N/A %RSD)
S	< 60000	(N/A %RSD)	< 59800	(N/A %RSD)
Sb	< 318	(N/A %RSD)	< 317	(N/A %RSD)
Si	< 218	(N/A %RSD)	516	(16 %RSD)
Sn	< 608	(N/A %RSD)	< 606	(N/A %RSD)
Sr	17.3	(10 %RSD)	17.9	(10 %RSD)
Th	< 441	(N/A %RSD)	< 439	(N/A %RSD)
Ti	< 37.3	(N/A %RSD)	< 37.2	(N/A %RSD)
U	< 2160	(N/A %RSD)	< 2160	(N/A %RSD)
V	< 46	(N/A %RSD)	< 45.8	(N/A %RSD)
Zn	< 43.3	(N/A %RSD)	< 43.2	(N/A %RSD)

XRD/XRF Data

Solids from Mixed Acid Digestion of WIPP R16C4 Sample #1 by XRD (LIMS 300313910)

- Mixed Acid Sample Preparation
  - Approximately 0.2g of sample was placed in a Teflon vessel with 4 mL HNO_3, 2 mL HCl and 2mL HF. The vessel was sealed and heated at 115 °C for 6 hours. Solids were noted in the solution and filtered away and the filtrate diluted to 50 mL. The solids were sent to XRD and XRF for analysis.

- XRD Sample Preparation
  - The filtered solids were ground in an agate mortar and pestle to reduce the particle size and to homogenize the samples. The ground powder(s) was attached to a plate glass slide using a Collodion/Amyl Acetate solution.

- XRD Instrument and Data Collection
  - X-ray diffraction data were collected on a Bruker D8 X-ray Diffractometer by step scanning over the 2Θ range of 5-70º with a step size of 0.02º and a dwell time of 1s. All the instrument parameters are listed in Table 1. Search-match identification was performed with Jade software from Materials Data Inc. and the International Centre of Diffraction Database.

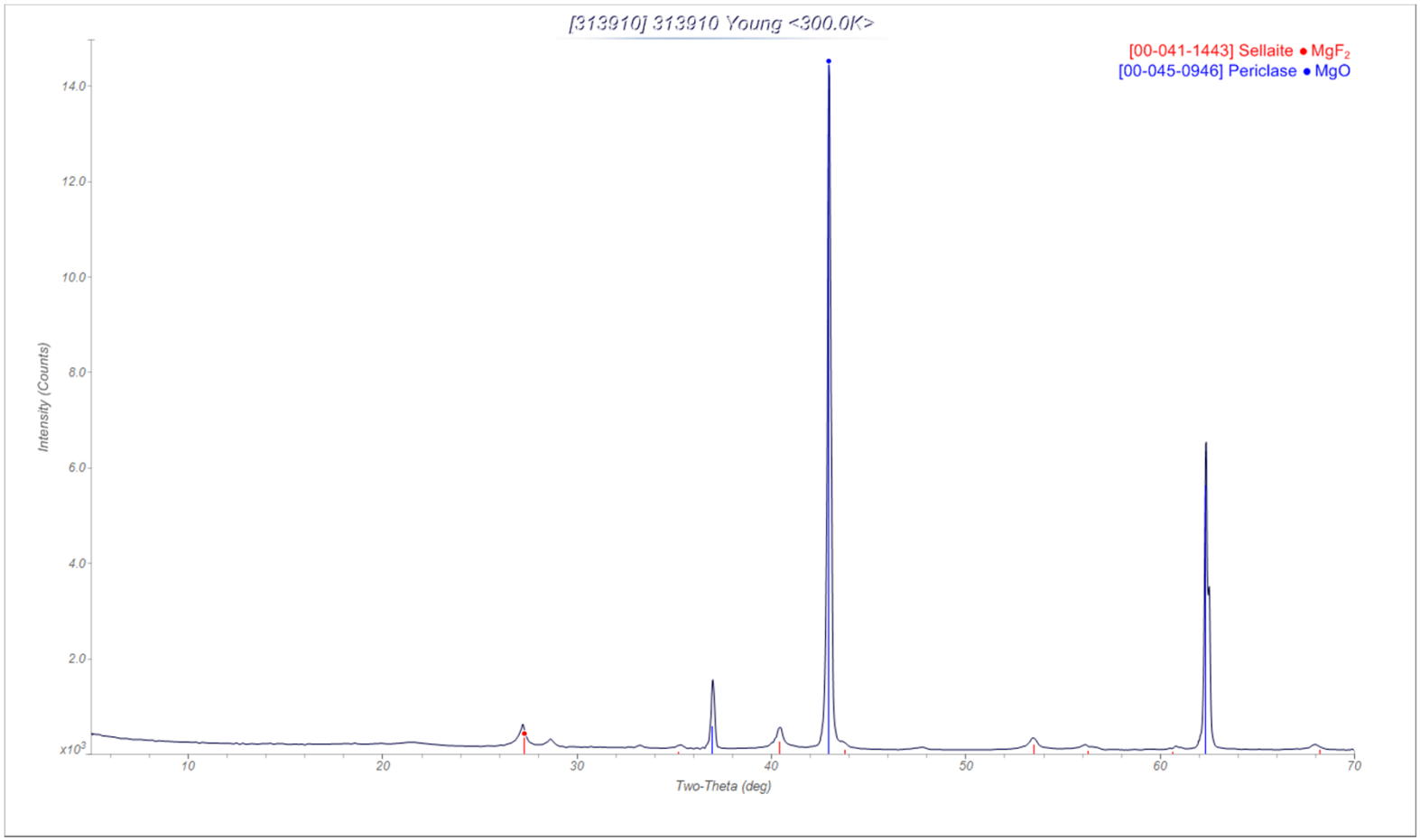
Solids from Mixed Acid Digestion of WIPP R16C4 Sample #1 by XRD (LIMS 300313910)

- TABLE I. Instrument Parameters.

– Radiation Source	CuKα X-ray
– Source Power	45 kV, 40 mA
– Wavelength	1.5405982 Å
– Goniometer	Bruker D8
– Divergence Slit	1º
– Divergence Soller Slit	None
– Divergence Anti-scatter	1º
– Specimen Rotation	No
– Diffracted Beam Anti-scatter	1º
– Diffracted Beam Soller Slit	2º
– Secondary Monochromator	Curved pyrolytic graphite
– Receiving Slit	0.18º
– Detector Slit	0.6º
– Detector	NaI Scintillation
– 2Θ Range	5º - 70º
– Step Interval	0.02º (2Θ)
– Fixed counting Time	1 s/step

Solids from Mixed Acid Digestion of WIPP R16C4 Sample #1 by XRD (LIMS 300313910)

- Sample spectrum indicates that not all MgO dissolved and that some MgF_2 formed in the mixed acid sample preparations



Solids from Mixed Acid Digestion of WIPP R16C4 Sample #1 by XRF (LIMS 300313910)

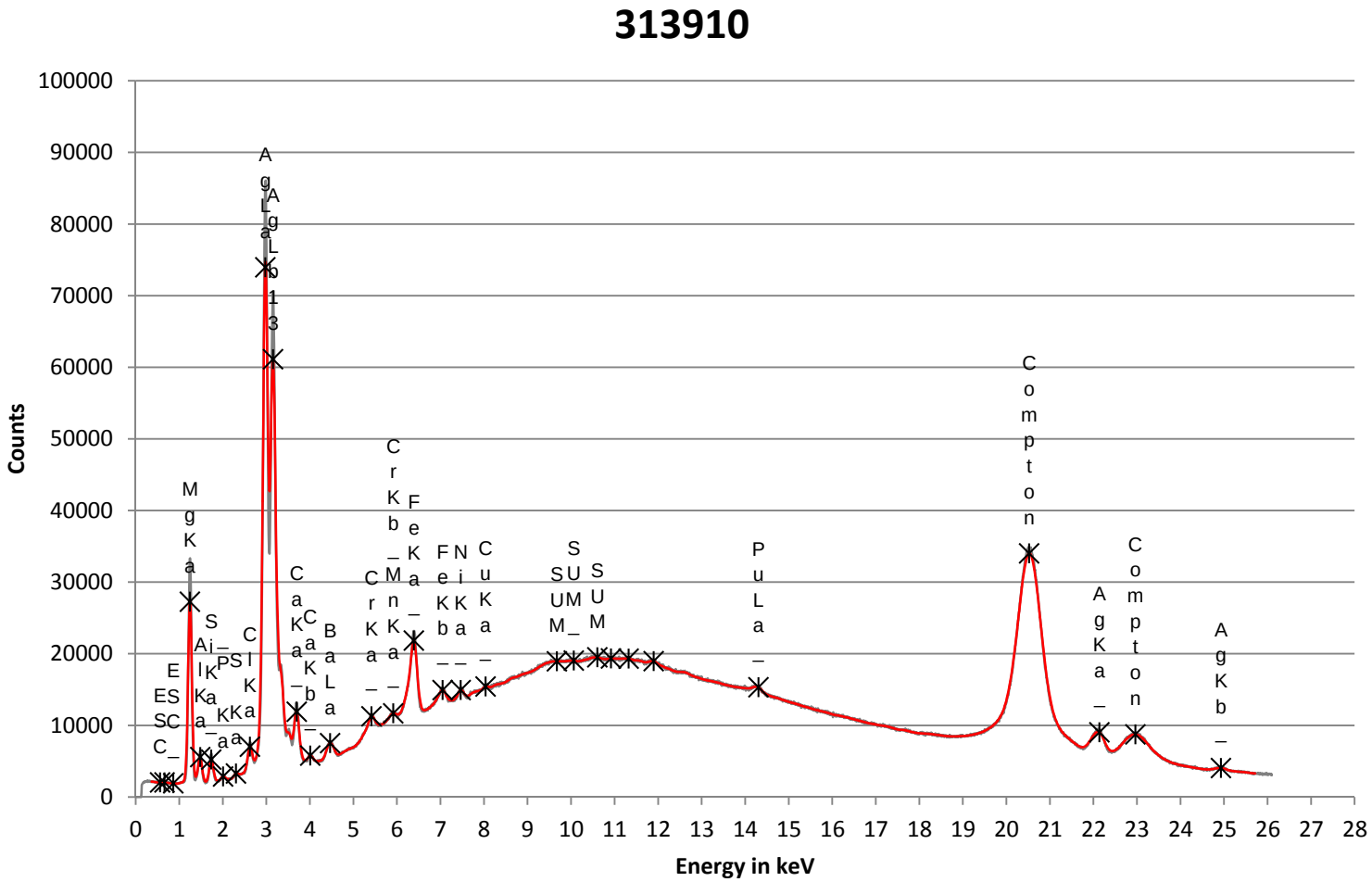
- Mixed Acid Sample Preparation
  - Approximately 0.2g of sample was placed in a Teflon vessel with 4 mL HNO3, 2 mL HCl and 2mL HF. The vessel was sealed and heated at 115 °C for 6 hours. Solids were noted in the solution and filtered away and the filtrate diluted to 50 mL. The solids were sent to XRD and XRF for analysis.

- XRF Sample Preparation
  - The WIPP samples were ground in an agate mortar and pestle to reduce the particle size and to homogenize the samples. The ground WIPP powder(s) was poured into a Plexiglas slide with an Ultralene film.

- XRF Instrument and Data Collection
  - X-ray fluorescence data were collected on an Amptek X123-SDD X-ray Spectrometer with an Ag x-ray tube. The instrument was ran at 50kV 80µA for 1800 seconds.

Solids from Mixed Acid Digestion of WIPP R16C4 Sample #1 by XRF (LIMS 300313910)

- Sample Spectrum…



WIPP R15C5 Sample #2 by XRD (LIMS 300313811)

- Sample Preparation
  - The samples were ground in an agate mortar and pestle to reduce the particle size and to homogenize the samples. The ground powder(s) was attached to a plate glass slide using a Collodion/Amyl Acetate solution.

- Instrument and Data Collection
  - X-ray diffraction data were collected on a Bruker D8 X-ray Diffractometer by step scanning over the 2Θ range of 5-70º with a step size of 0.02º and a dwell time of 1s. All the instrument parameters are listed in Table 1. Search-match identification was performed with Jade software from Materials Data Inc. and the International Centre of Diffraction Database.

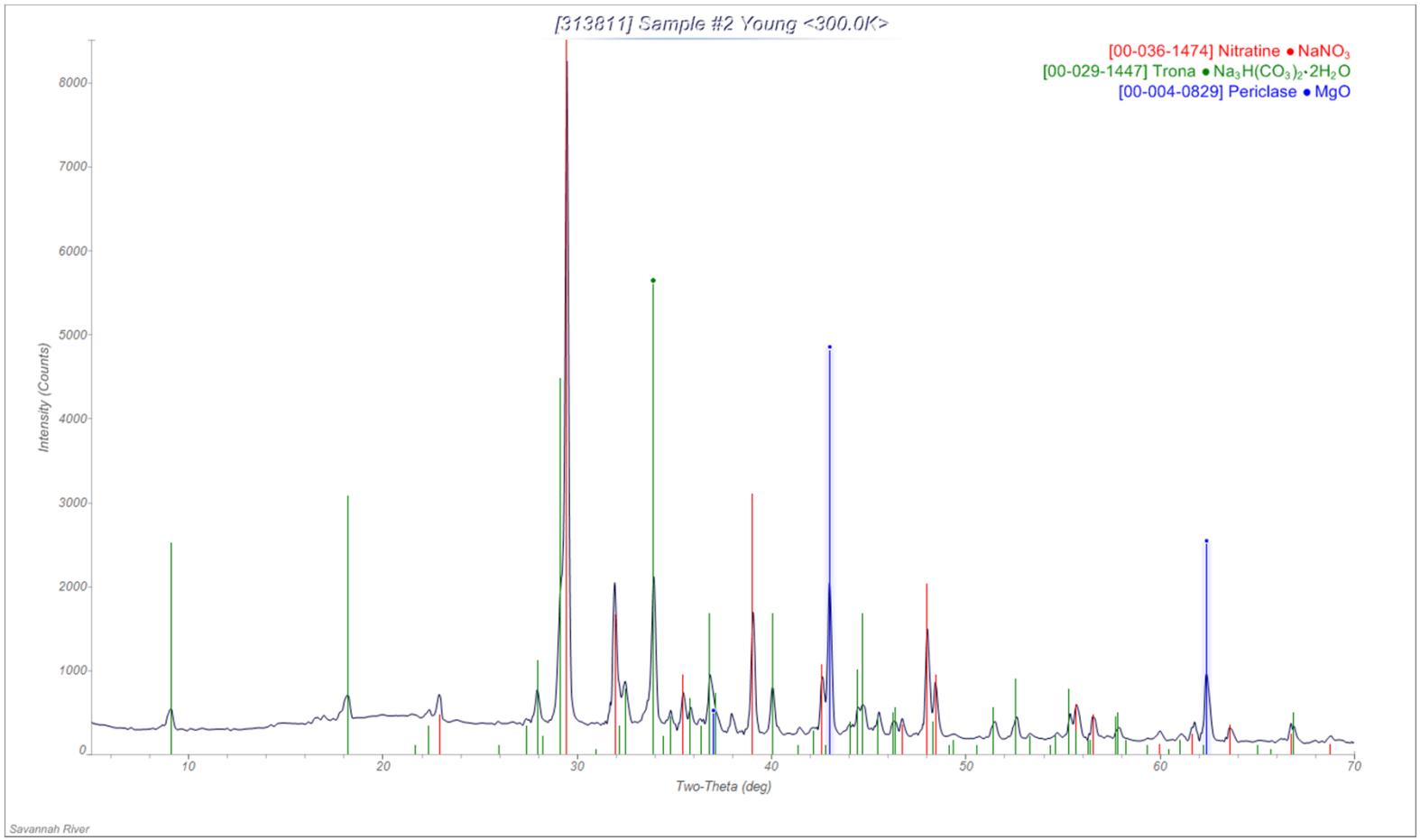
WIPP R15C5 Sample #2 by XRD (LIMS 300313811)

- TABLE I. Instrument Parameters.

– Radiation Source	CuKα X-ray
– Source Power	45 kV, 40 mA
– Wavelength	1.5405982 Å
– Goniometer	Bruker D8
– Divergence Slit	1º
– Divergence Soller Slit	None
– Divergence Anti-scatter	1º
– Specimen Rotation	No
– Diffracted Beam Anti-scatter	1º
– Diffracted Beam Soller Slit	2º
– Secondary Monochromator	Curved pyrolytic graphite
– Receiving Slit	0.18º
– Detector Slit	0.6º
– Detector	NaI Scintillation
– 2Θ Range	5º - 70º
– Step Interval	0.02º (2Θ)
– Fixed counting Time	1 s/step

WIPP R15C5 Sample #2 by XRD (LIMS 300313811)

- Spectra indicates presence of MgO, $NaNO_2$ and $Na_3H(CO_3)_2 \cdot 2H_2O$



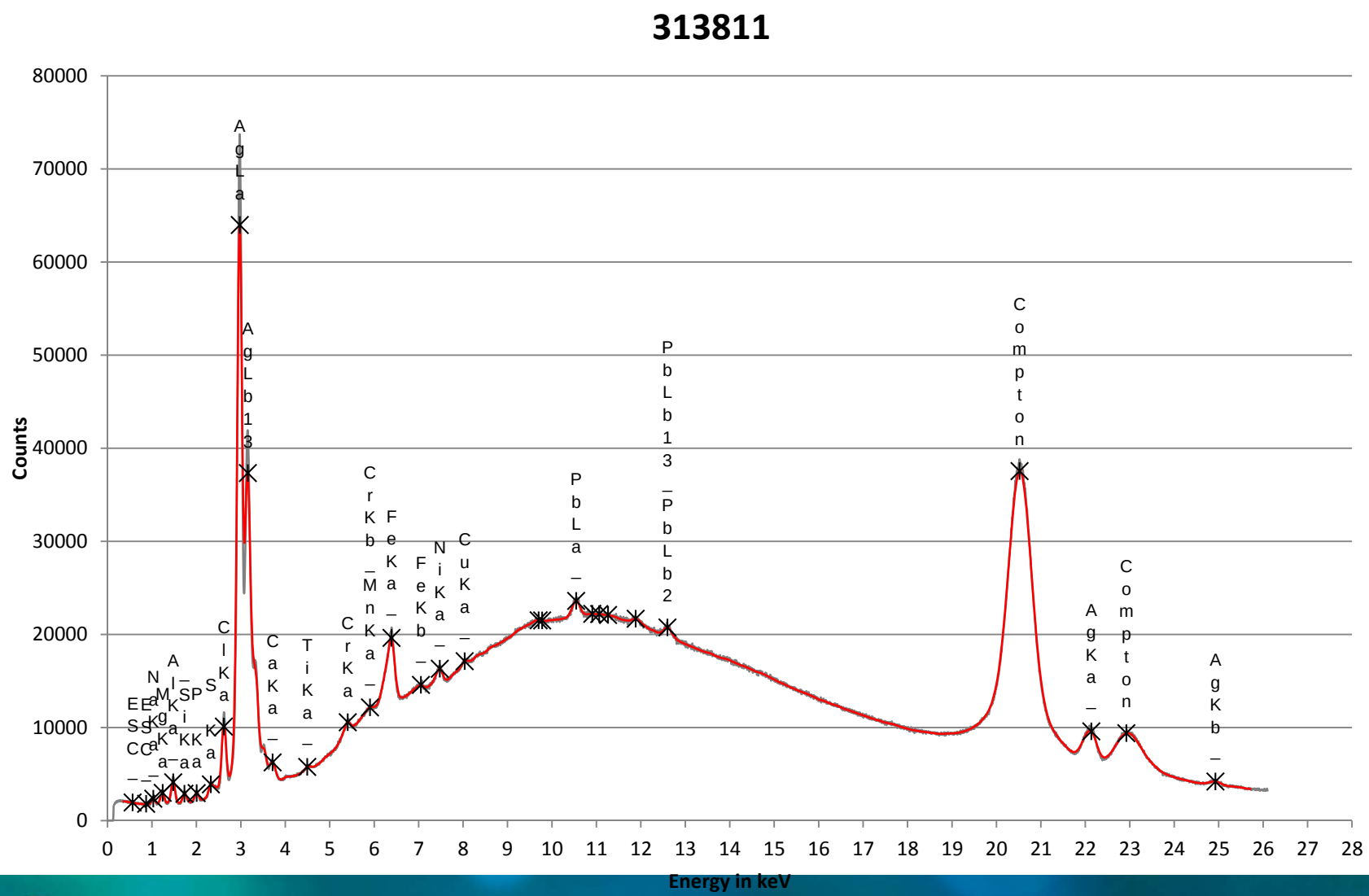
WIPP R15C5 Sample #2 by XRF (LIMS 300313811)

- Sample Preparation
  - The WIPP samples were ground in an agate mortar and pestle to reduce the particle size and to homogenize the samples. The ground WIPP powder(s) was poured into a Plexiglas slide with an Ultralene film.

- Instrument and Data Collection
  - X-ray fluorescence data were collected on an Amptek X123-SDD X-ray Spectrometer with an Ag x-ray tube. The instrument was ran at 50kV 80µA for 1800 seconds.

WIPP R15C5 Sample #2 by XRD (LIMS 300313811)

- Sample Spectrum…



WIPP Super Sack MgO Surface Samples #6 and #7 by XRD (LIMS 300313920/921)

- Sample Preparation
  - The samples were ground in an agate mortar and pestle to reduce the particle size and to homogenize the samples. The ground powder(s) was attached to a plate glass slide using a Collodion/Amyl Acetate solution.

- Instrument and Data Collection
  - X-ray diffraction data were collected on a Bruker D8 X-ray Diffractometer by step scanning over the 2Θ range of 5-70º with a step size of 0.02º and a dwell time of 1s. All the instrument parameters are listed in Table 1. Search-match identification was performed with Jade software from Materials Data Inc. and the International Centre of Diffraction Database.

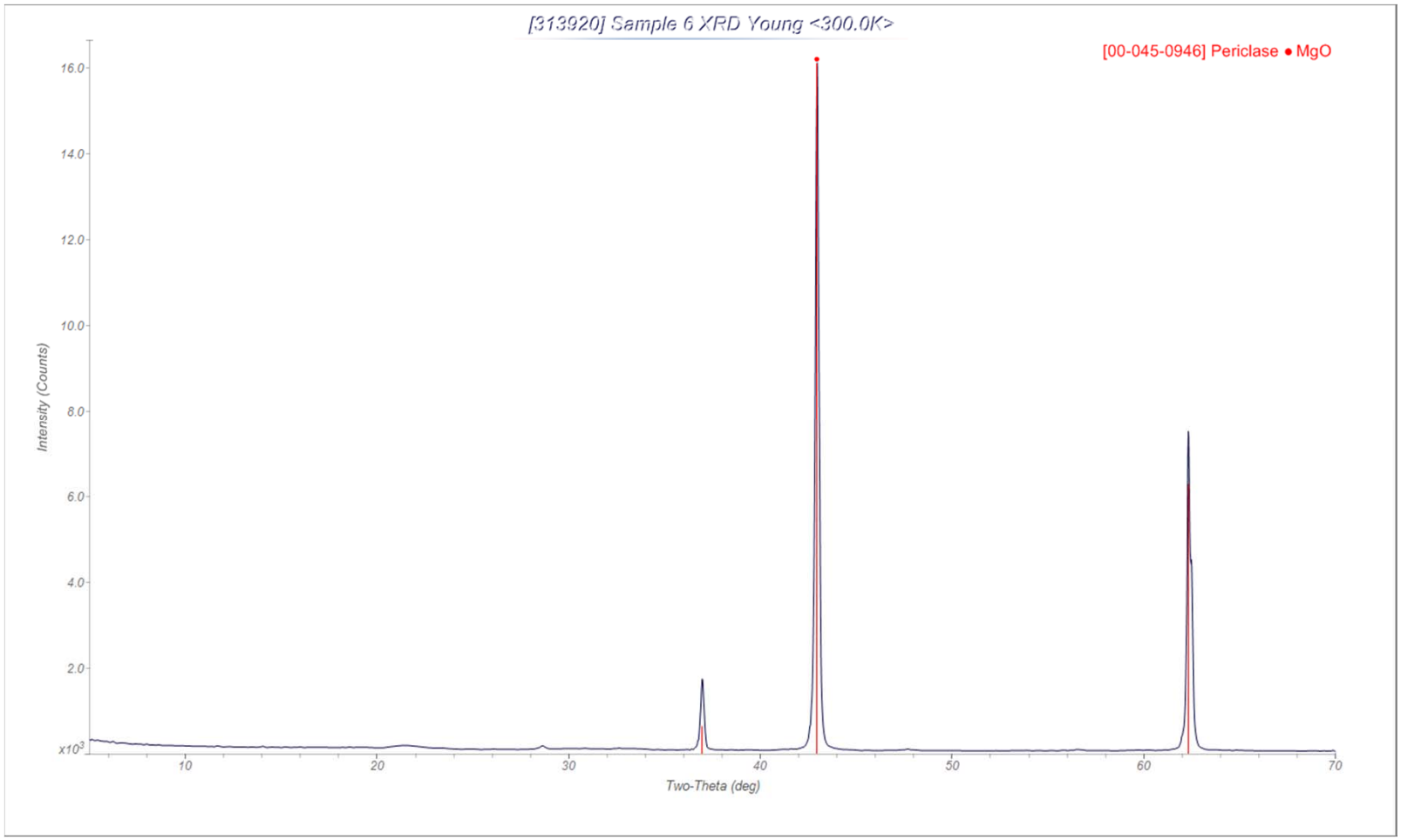
- Data Interpretation
  - Samples #6 and #7 spectra were very similar and consistent with magnesium oxide (no Mg(OH)2, MgCO3 or other reaction intermediates)

WIPP Super Sack MgO Surface Sample #6 by XRD (LIMS 300313920)

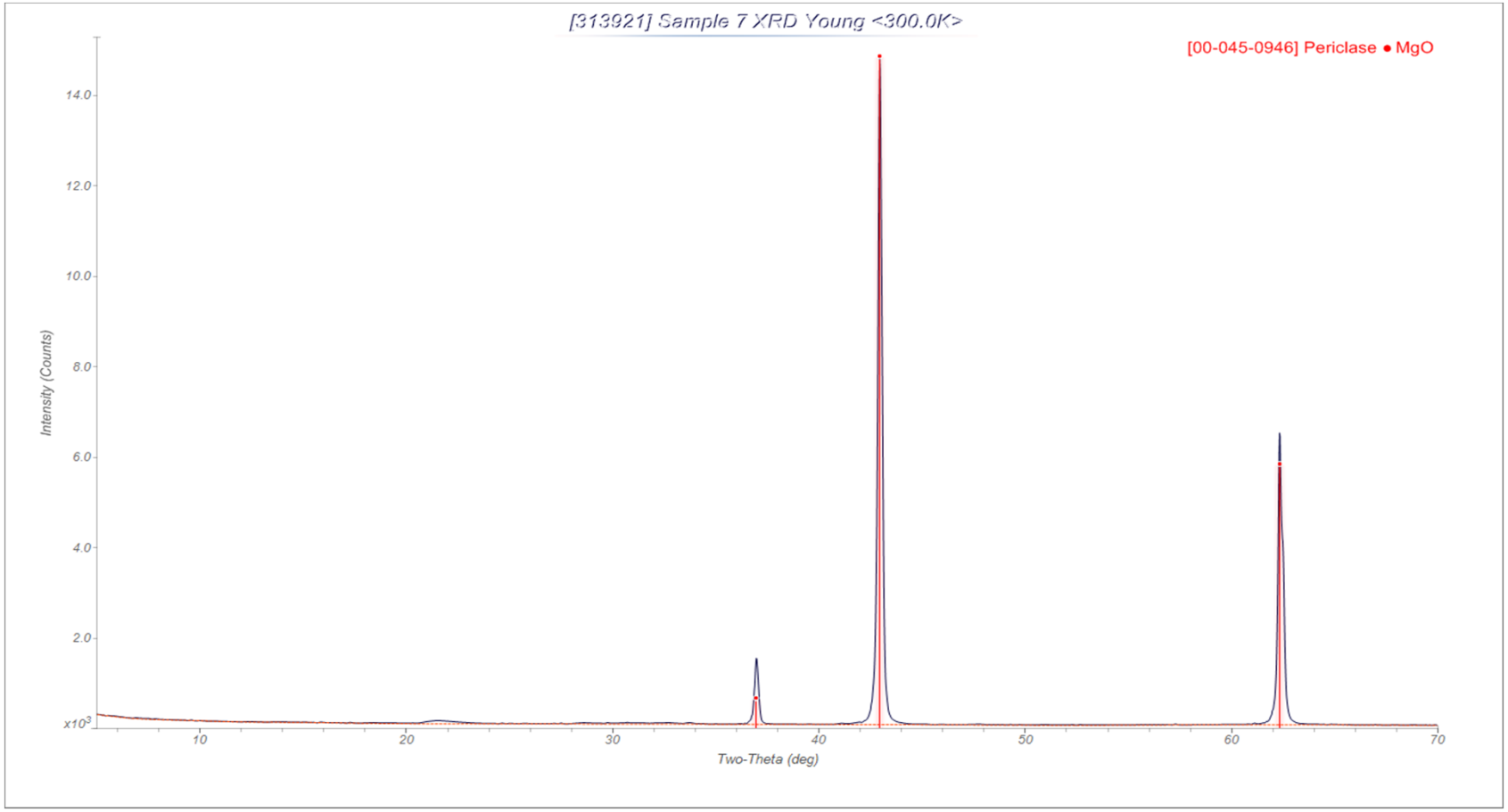
- TABLE I. Instrument Parameters.

– Radiation Source	CuKα X-ray
– Source Power	45 kV, 40 mA
– Wavelength	1.5405982 Å
– Goniometer	Bruker D8
– Divergence Slit	1º
– Divergence Soller Slit	None
– Divergence Anti-scatter	1º
– Specimen Rotation	No
– Diffracted Beam Anti-scatter	1º
– Diffracted Beam Soller Slit	2º
– Secondary Monochromator	Curved pyrolytic graphite
– Receiving Slit	0.18º
– Detector Slit	0.6º
– Detector	NaI Scintillation
– 2Θ Range	5º - 70º
– Step Interval	0.02º (2Θ)
– Fixed counting Time	1 s/step

WIPP Super Sack MgO Surface Sample #6 by XRD (LIMS 300313920)



WIPP Super Sack MgO Sub Surface (6-inch) Sample #7 by XRD (LIMS 300313921)



SEM Data

WIPP R16C4 Sample #1 by SEM/EDS (LIMS 300313918)

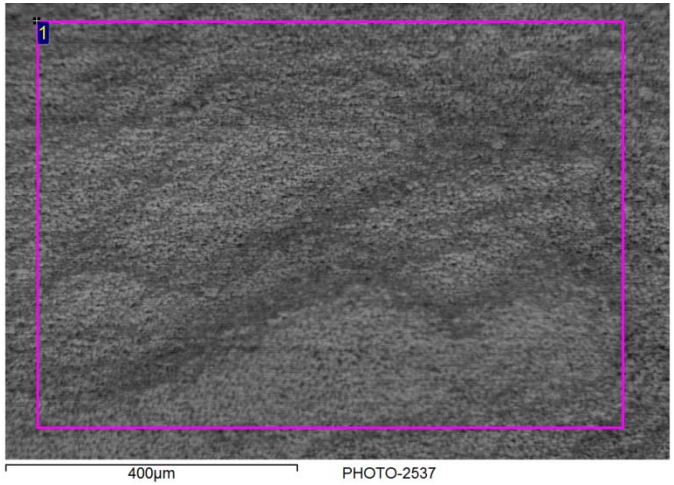
WIPP SAMPLE-1 R16C4 LIMS ID# 300313918

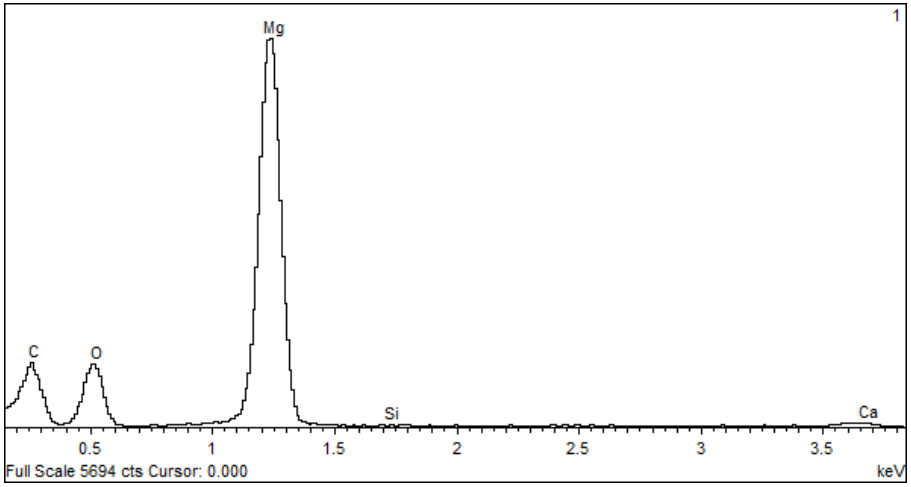
- This particle appears to be MgO with traces of Si and Ca, as is typical for MgO particles.

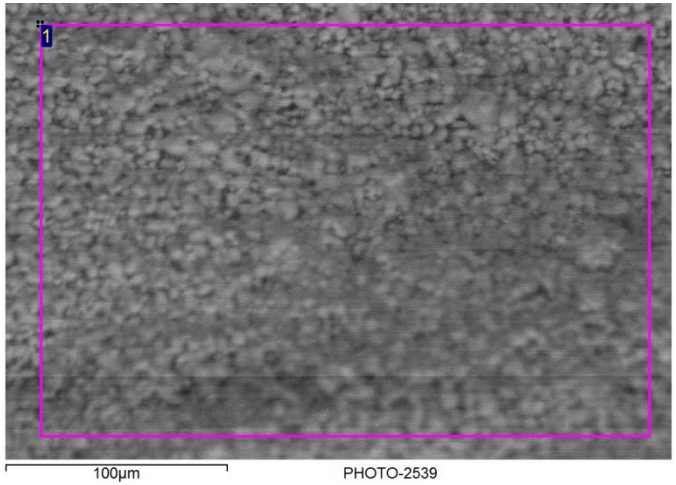
EXPERIMENTAL DETAILS:

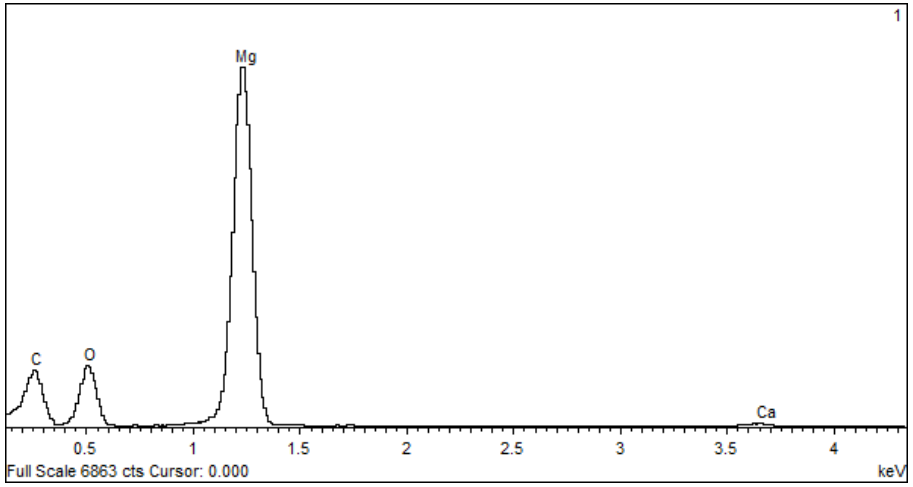
- All particles were mounted on carbon sticky tape on aluminum stubs and evaporatively carbon coated. The LEO S440 SEM enclosed in a glovebox was used as was a liquid nitrogen cooled Oxford Instruments SiLi EDS detector. 30 kV was the accelerating voltage.

WIPP R16C4 Sample #1 by SEM/EDS (photos 2537 & 2539)









WIPP R15C5 Sample #2 by SEM/EDS (LIMS 300313919)

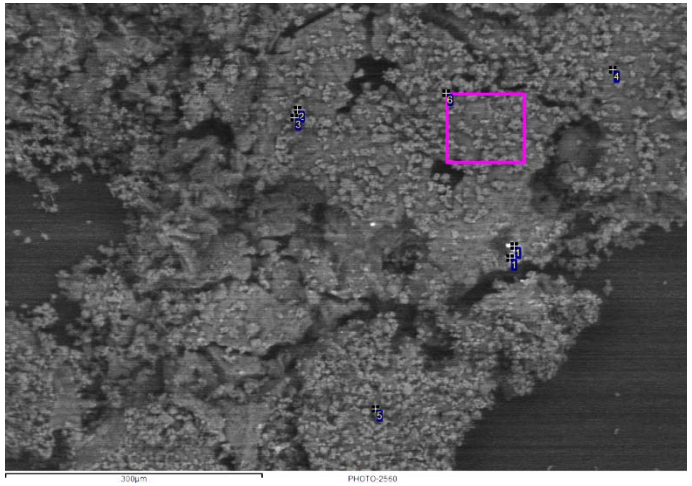
WIPP SAMPLE-2 R15C5

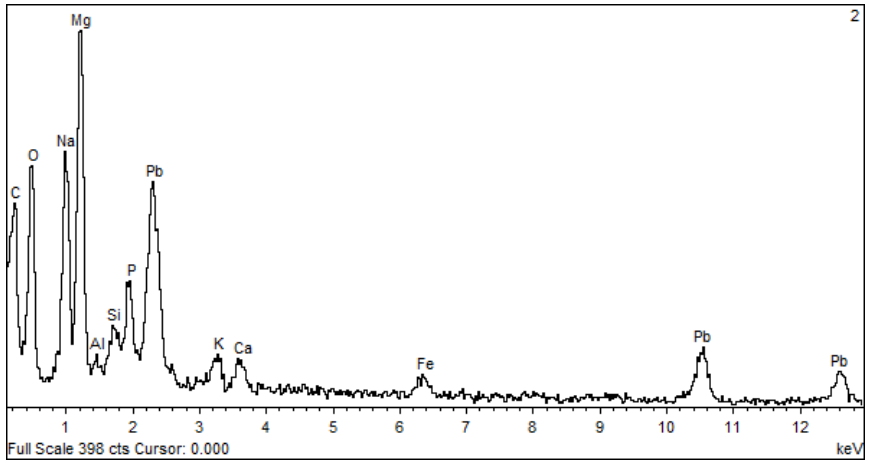
- This material is fairly heterogeneous. The background particles contain mostly Mg, O, and Na with small amounts of K, Ca and Cl. In backscatter imaging, bright particles that show up contain mostly Pb, with small amounts of Al, P, Fe and occasionally Si.

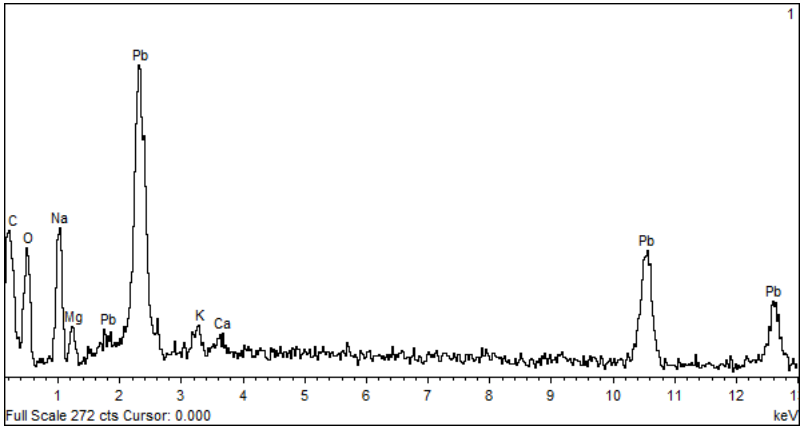
EXPERIMENTAL DETAILS:

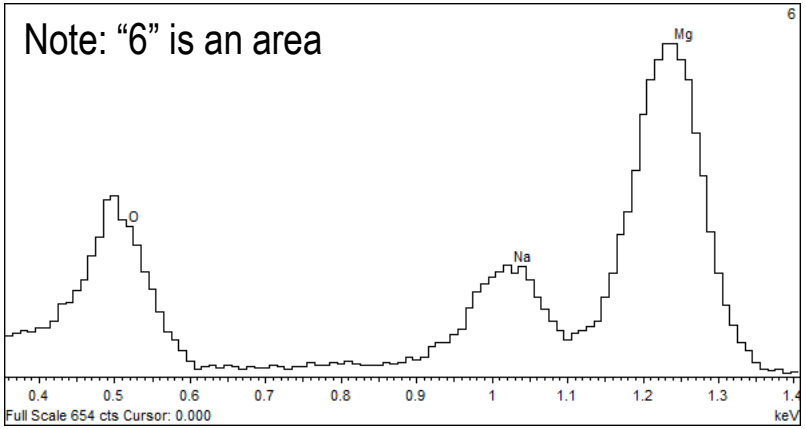
- All particles were mounted on carbon sticky tape on aluminum stubs and evaporatively carbon coated. The LEO S440 SEM enclosed in a glovebox was used as was a liquid nitrogen cooled Oxford Instruments SiLi EDS detector. 30 kV was the accelerating voltage.

WIPP R15C5 Sample #2 by SEM/EDS (photo 2560)

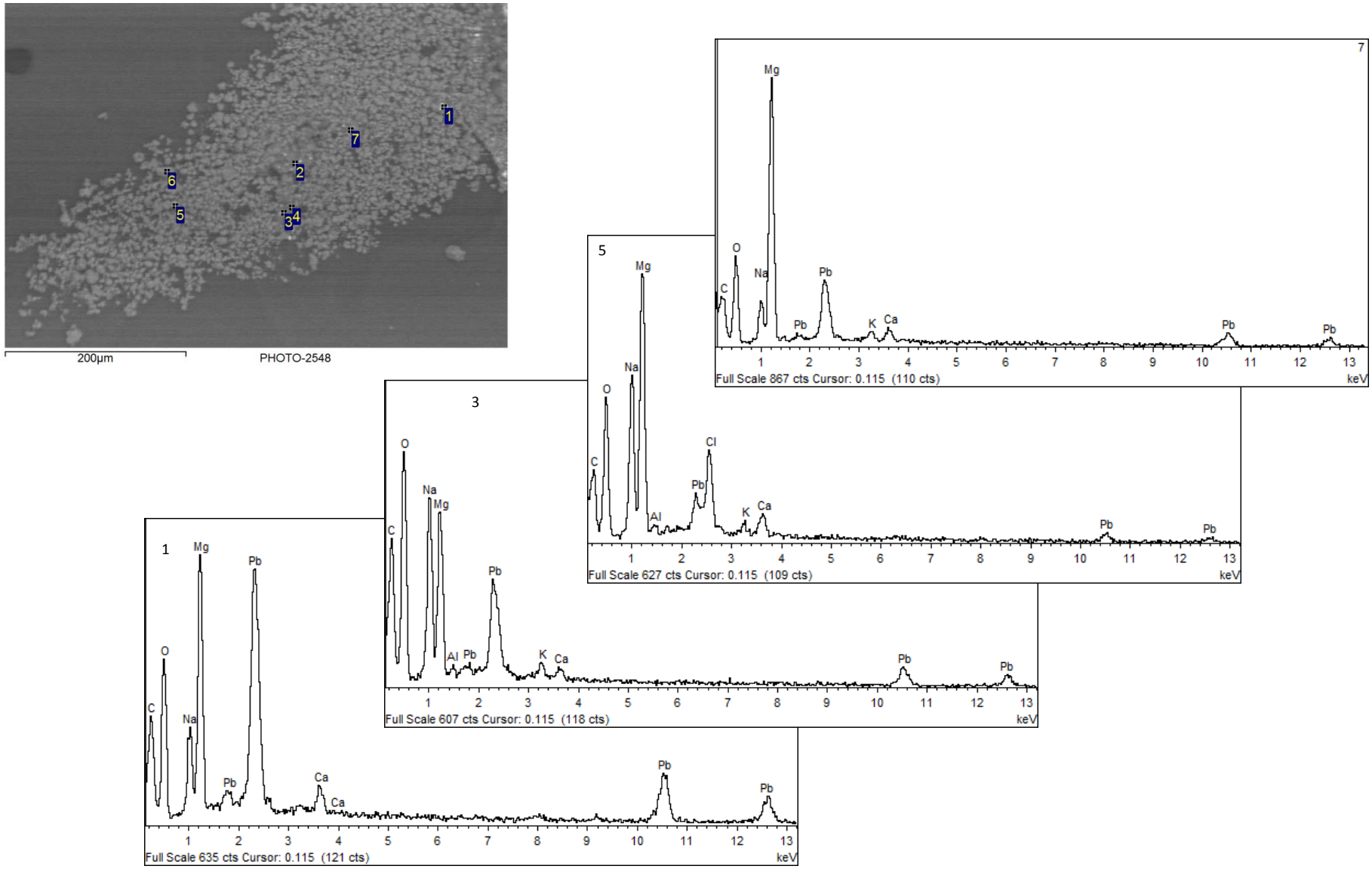








WIPP R15C5 Sample #2 by SEM/EDS (photo 2548)



LANL Parent Drum Debris by SEM/EDS (LIMS 300313607)

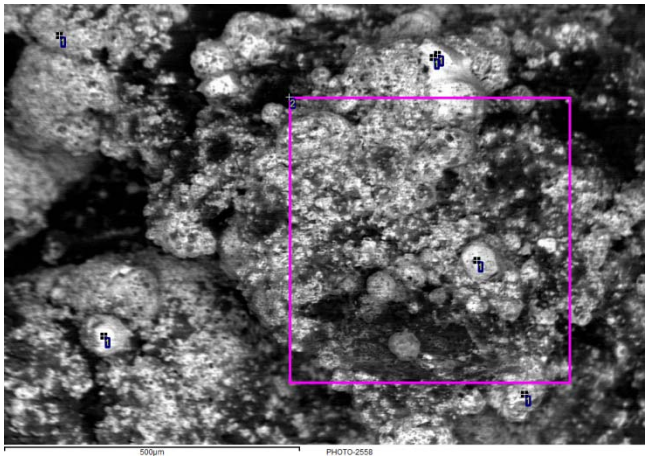
LANL PARENT DRUM DEBRIS

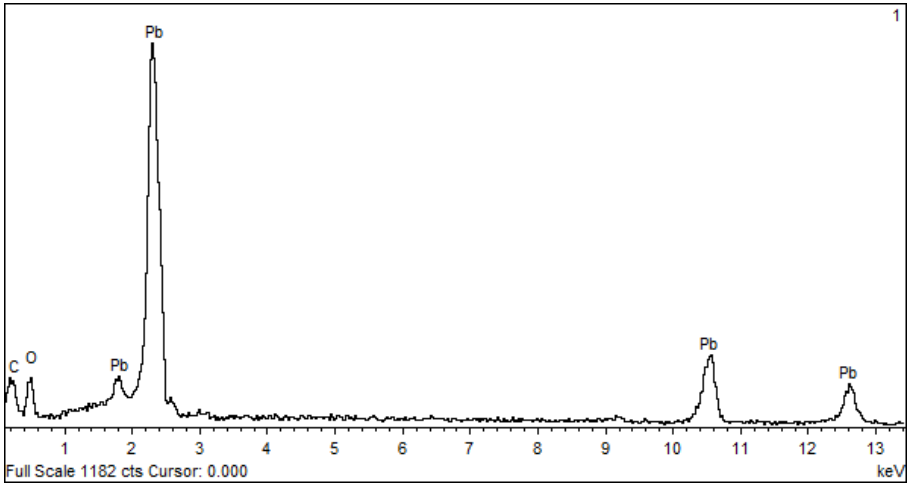
- This chunk of debris contains predominantly Pb, with small amounts of Na, Mg, Al, K, Ca, and Fe.

EXPERIMENTAL DETAILS:

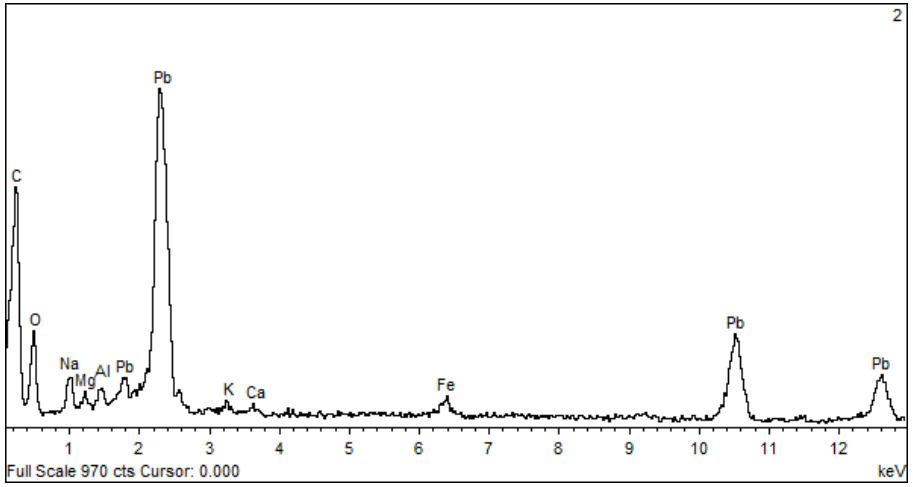
- All particles were mounted on carbon sticky tape on aluminum stubs and evaporatively carbon coated. The LEO S440 SEM enclosed in a glovebox was used as was a liquid nitrogen cooled Oxford Instruments SiLi EDS detector. 30 kV was the accelerating voltage.

LANL Parent Drum Debris by SEM/EDS (photo 2558)

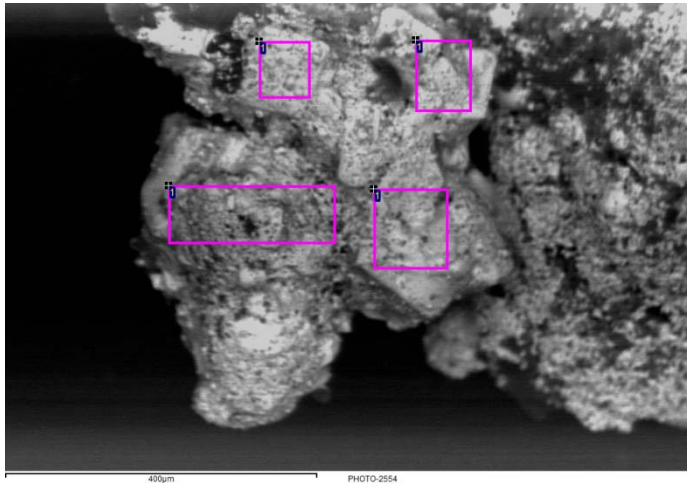


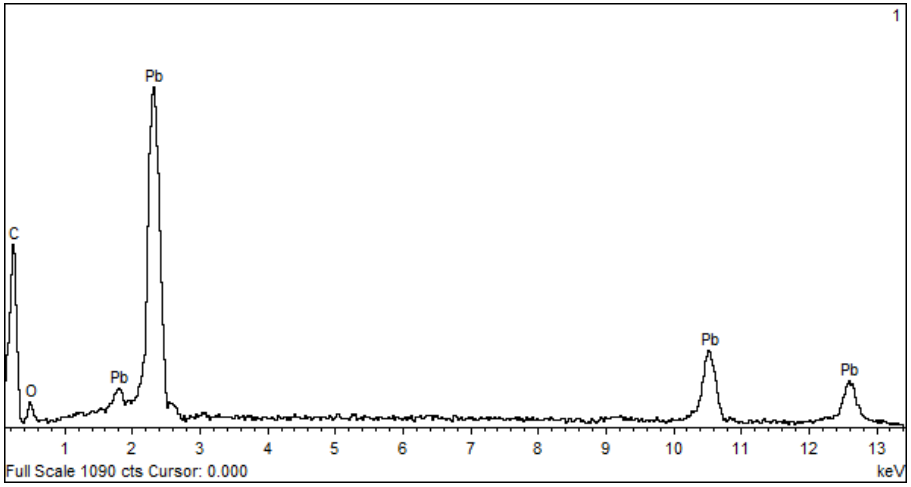


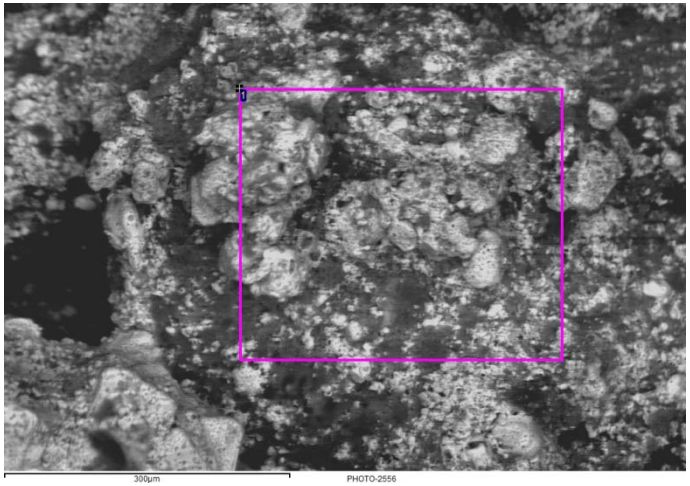
Note: “2” is an area

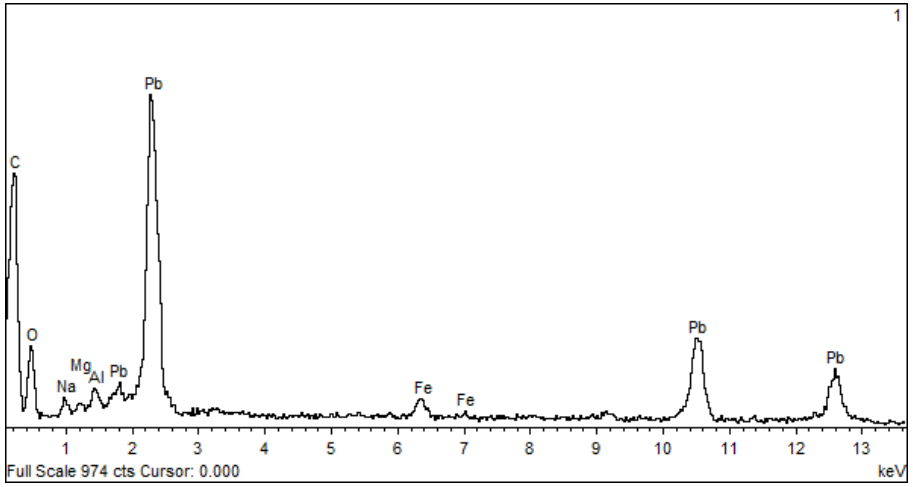


LANL Parent Drum Debris by SEM/EDS (photo 2554 & 2556)

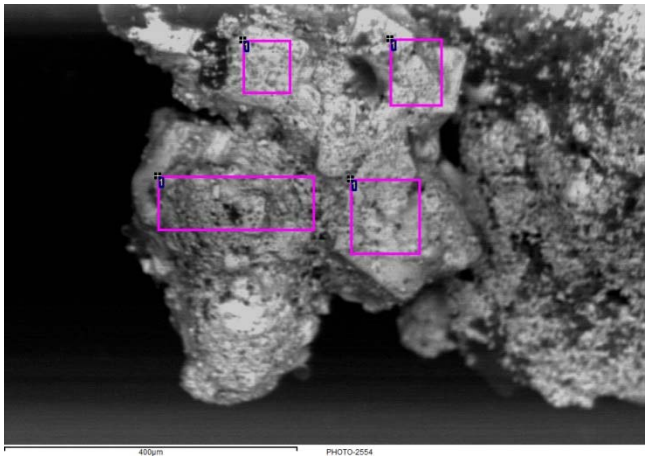


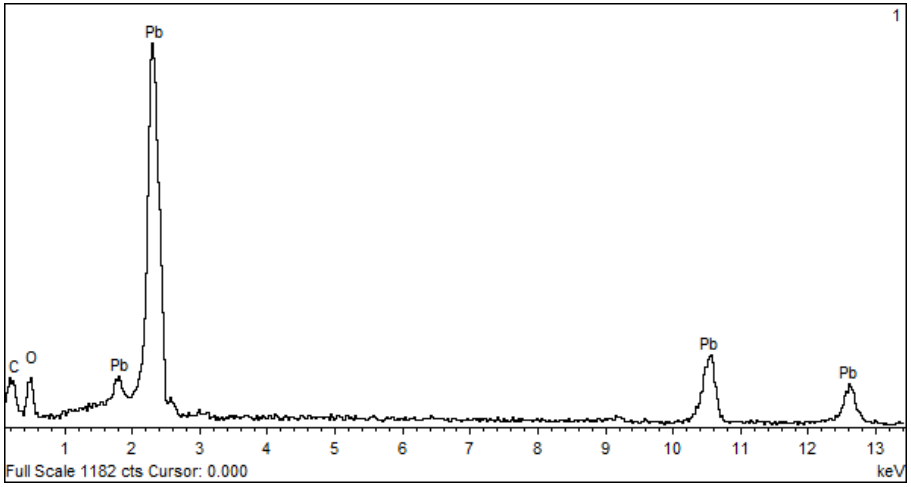




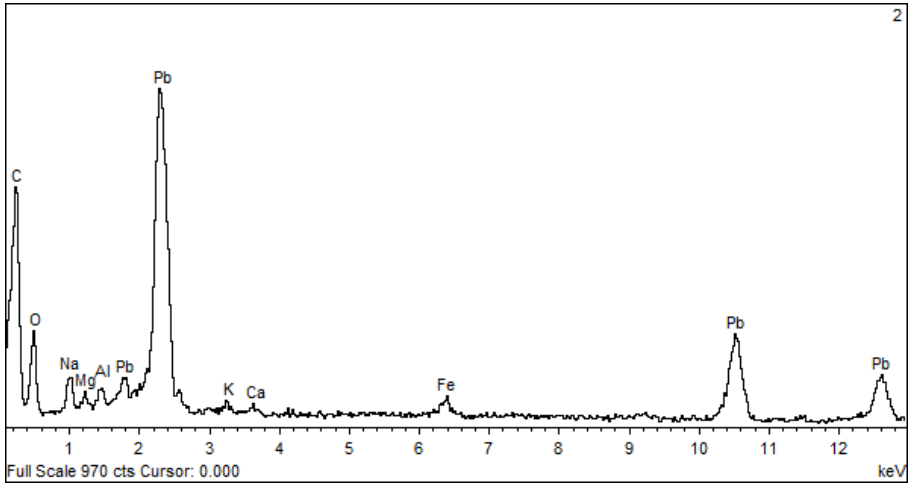


LANL Parent Drum Debris by SEM/EDS (photo 2554)





Note: “2” is an area

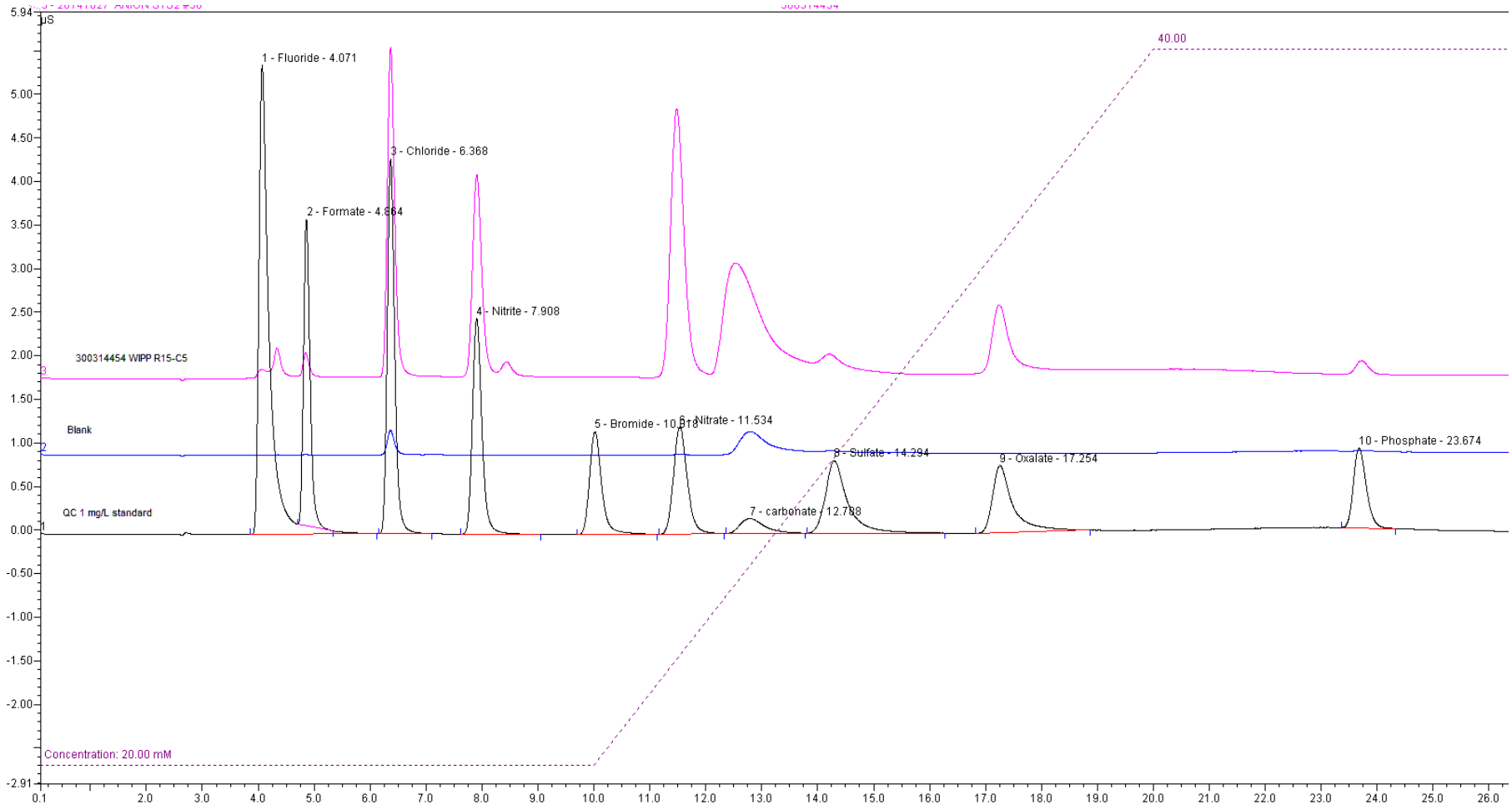


IC Data

Ion Chromatography – WIPP R15C5 Sample #2 (LIMS 300314454)

- Sample Preparation
  - Weighed amounts
    - *WIPP sample #2 (R15-C5) for water leach (TC 66439 – 300314454) 0.1366 g*
  - 20 mL of DI water
  - Leached for 4 days
  - Filtered prior to analysis
- Instrument
  - Contained Dionex RFIC 3000 Ion Chromatography system
  - AG-19 4x50 mm guard column and AS-19 4x250 mm analytical column
  - 50 μL injection
  - 1.0 mL/min flow rate @ 30 ^{o}C
  - Mobile phase 20-40 mMol KOH gradient
  - Calibration Curve 1 mg/L to 50 mg/L, $r^2 > 0.994$
  - Standard contains Fluoride, Formate, Chloride, Nitrite, Bromide, Nitrate, Sulfate, Oxalate, Phosphate

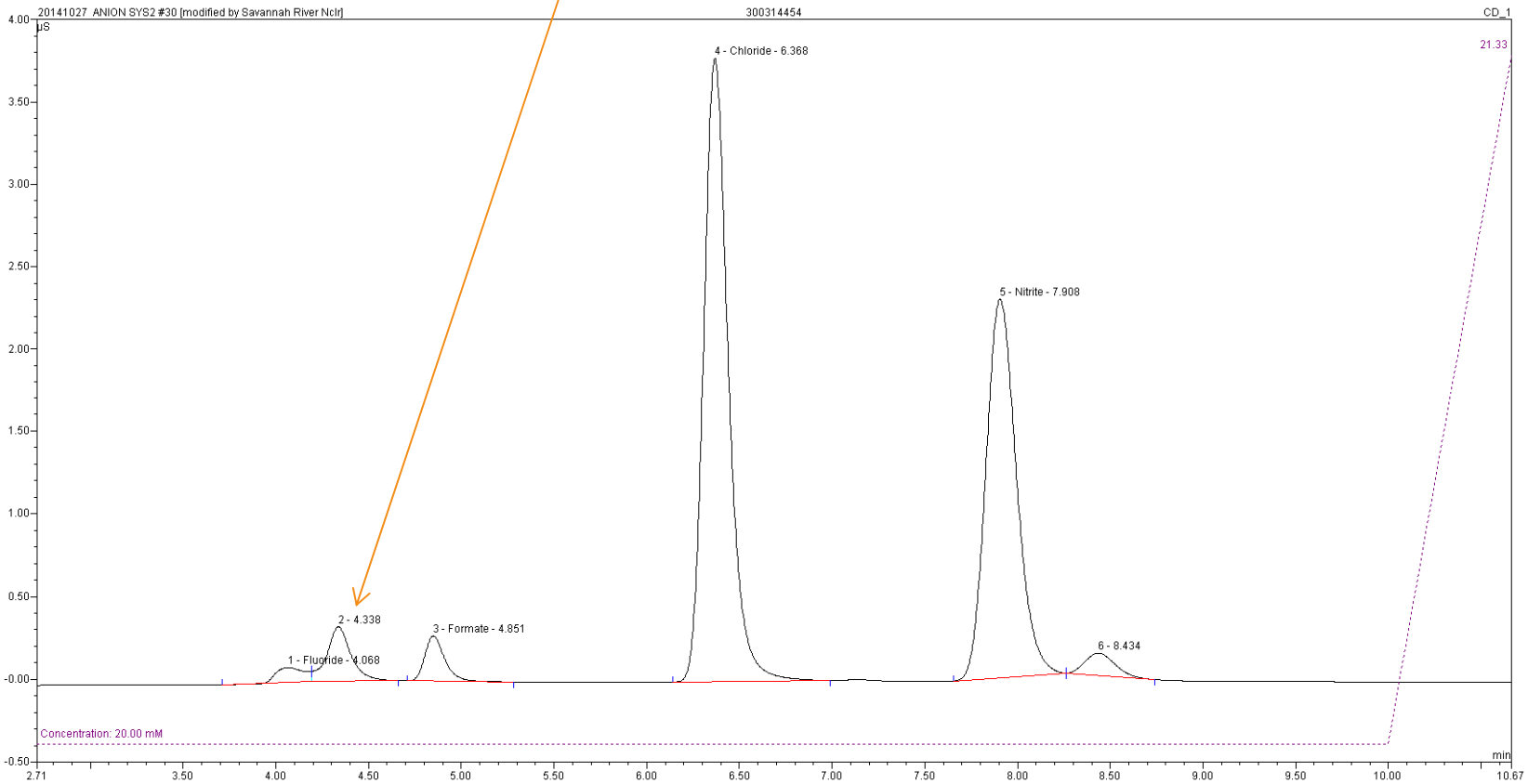
Ion Chromatography – WIPP R15C5 Sample #2 (LIMS 300314454)



Cust ID	Fluoride	Formate	Chloride	Nitrite	Bromide	Nitrate	Phosphate	Sulfate	Oxalate
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
TC 66439-Blank	<1460	<1460	<1460	<1460	<7300	<1460	<1460	<1460	<1460
TC 66439-WIPP #2(R15-C5)water leach	<1460	<1460	13100	13100	<7300	34600	1610	3070	13100

Ion Chromatography – WIPP R15C5 Sample #2 (LIMS 300314454)

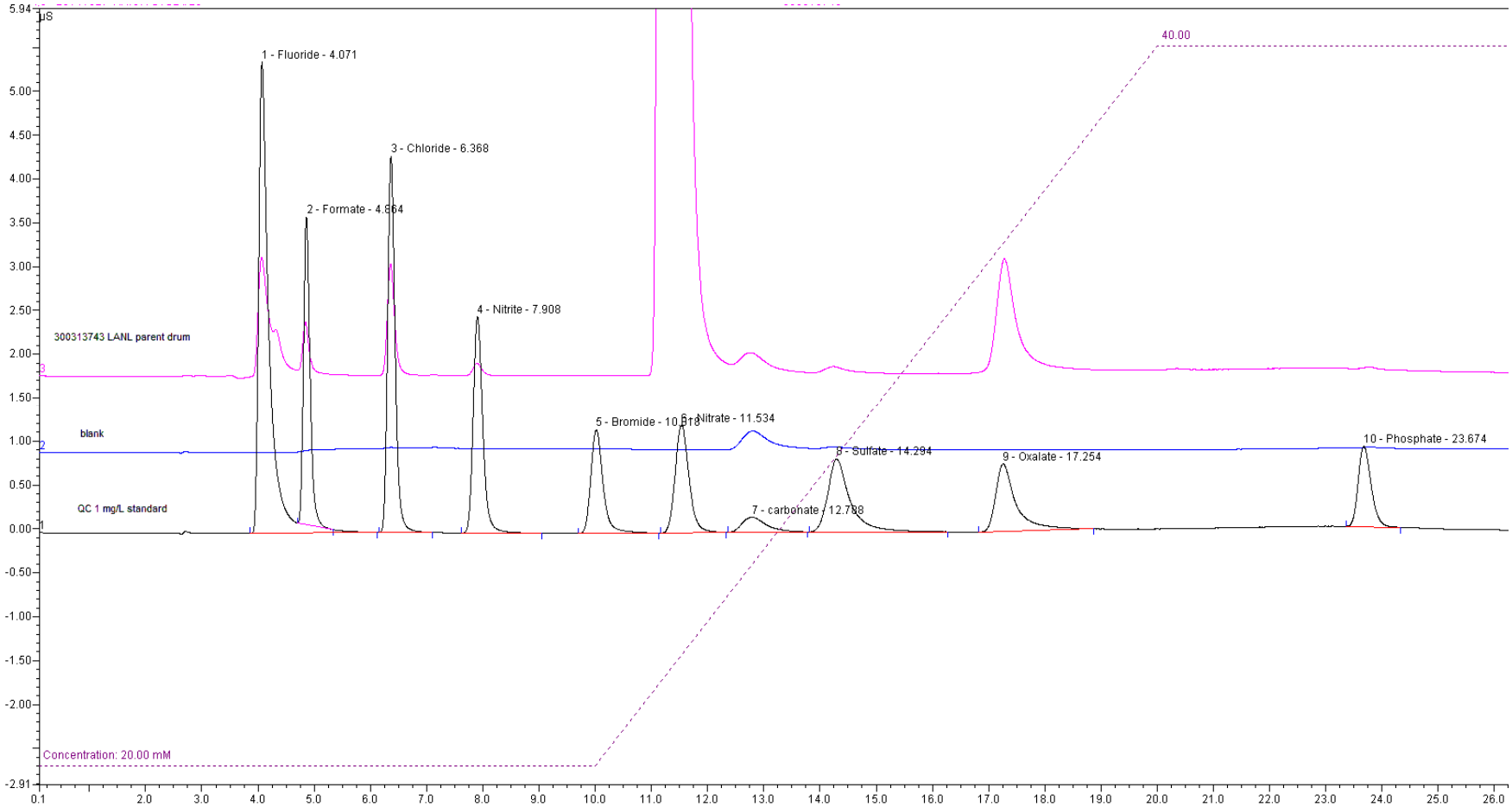
- Major analytes
  - Mainly nitrate, nitrite, chloride
  - Peak co eluting with fluoride is likely acetate
  - Oxalate and Formate present
  - Increase in carbonate present (previous slide)
  - Small unknown peak at 8.4 min



Ion Chromatography – LANL Parent Drum Debris (LIMS 300313743)

- Sample Preparation
  - Weighed amounts
    - *WIPP LANL Parent Drum solid debris (TC 66288 – 300313743) 0.6901 g*
  - 20 mL of DI water
  - Leached for 4 days
  - Filtered prior to analysis
- Instrument Conditions
  - Contained Dionex RFIC 3000 Ion Chromatography system
  - AG-19 4x50 mm guard column and AS-19 4x250 mm analytical column
  - 50 μL injection
  - 1.0 mL/min flow rate @ 30 ^{o}C
  - Mobile phase 20-40 mMol KOH gradient
  - Calibration Curve 1 mg/L to 50 mg/L, $r^2 > 0.994$
  - Standard contains Fluoride, Formate, Chloride, Nitrite, Bromide, Nitrate, Sulfate, Oxalate, Phosphate

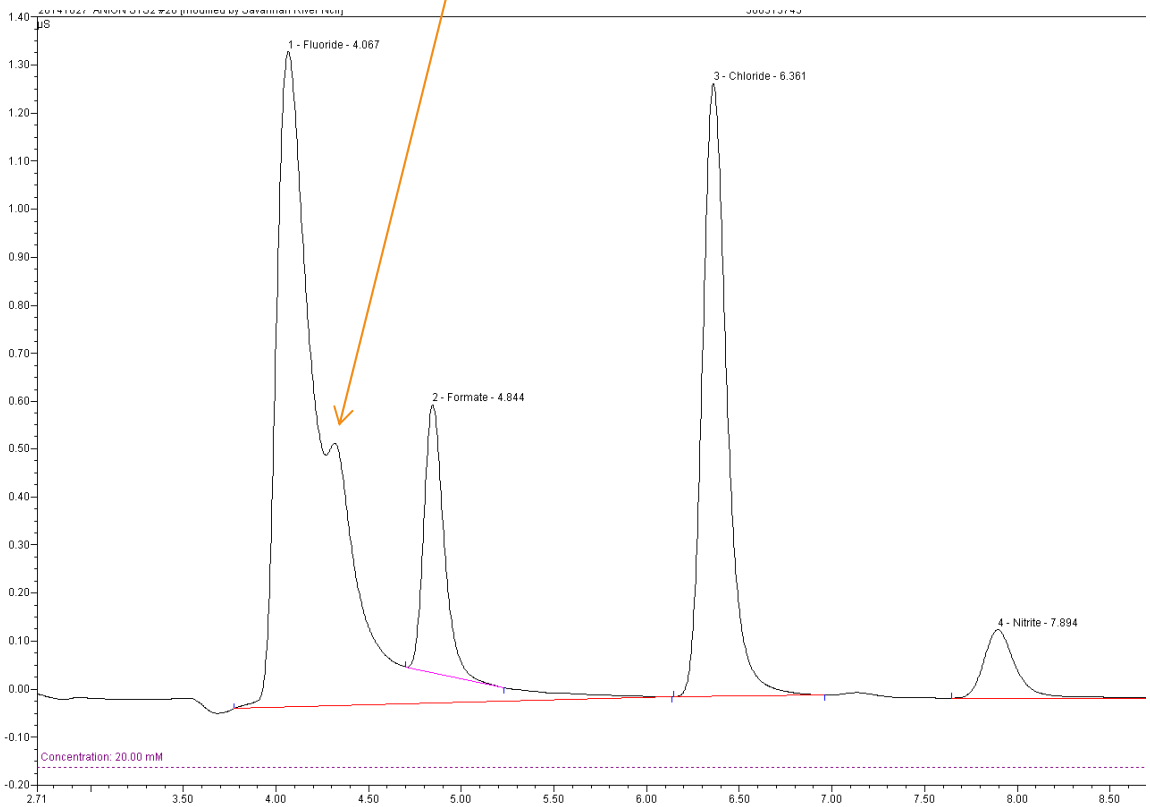
Ion Chromatography – LANL Parent Drum Debris (LIMS 300313743)



Cust ID	Fluoride	Formate	Chloride	Nitrite	Bromide	Nitrate	Phosphate	Sulfate	Oxalate
	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg	mg/Kg
TC 66288-Blank	<290	<290	<290	<290	<1500	<290	<290	<290	<290
TC 66288-WIPP LANL Parent Drum	667	638	899	<290	<1500	126000	<290	290	2640

Ion Chromatography – LANL Parent Drum Debris (LIMS 300313743)

- Major analytes
  - Mainly nitrate
    - *trace nitrite*
  - Peak co eluting with fluoride is likely acetate
  - Oxalate and Formate present
  - Some carbonate present (previous slide)



GC-MS Data

FAS and CAM Filter Air Sample Analysis by GC/MS

- Gas Chromatography / Mass Spectrometry (GC/MS) analysis was employed to identify organic compounds in the samples. Analytical separations were carried out on an Agilent 6890 gas chromatograph, equipped with a 25 m J&W DB-5MS column, with 0.20 mm diameter and 0.33 um film thickness. Characterization and quantification was performed using an Agilent 5973 mass selective detector.

- Each sample was weighed then extracted three times with methylene chloride at room temperature. The samples were then dried and weighed to quantify the amount of material extracted. Each extract was concentrated to one mL, spiked with internal standard (see Table 1 below), then analyzed by GC/MS.

- Blanks were analyzed before and after each set of samples. Internal standard analytes were used for quantification of sample analytes detected.

Table 1. Internal Standard Analytes:

Retention Time	Analyte	Melting Point, °C	Boiling Point, °C
9.022	1,4-Dichlorobenzene-d4	54	173
10.437	Naphthalene-d8	82	218
12.248	Acenaphthene-d10	95	278
13.773	Anthracene-d10	218	342
16.818	Chrysene-d12	252	448
18.739	Perylene-d12	278	475

Table 2. Sample Information

Customer ID	Lab No.	TC	Sample Mass, mg	Extractable Mass, mg
FAS Filter (311038)–E	300311110	65771	19.7	2.1
CAM Filter 2 (311040)-E	300311165	65791	13.5	0.79
CAM Filter 7 (311045)-E	300311171	65791	17.0	0.97
CAM Filter 11 (311049)-E	300311189	65792	16.6	0.84

FAS and CAM Filter Air Sample Analysis by GC/MS

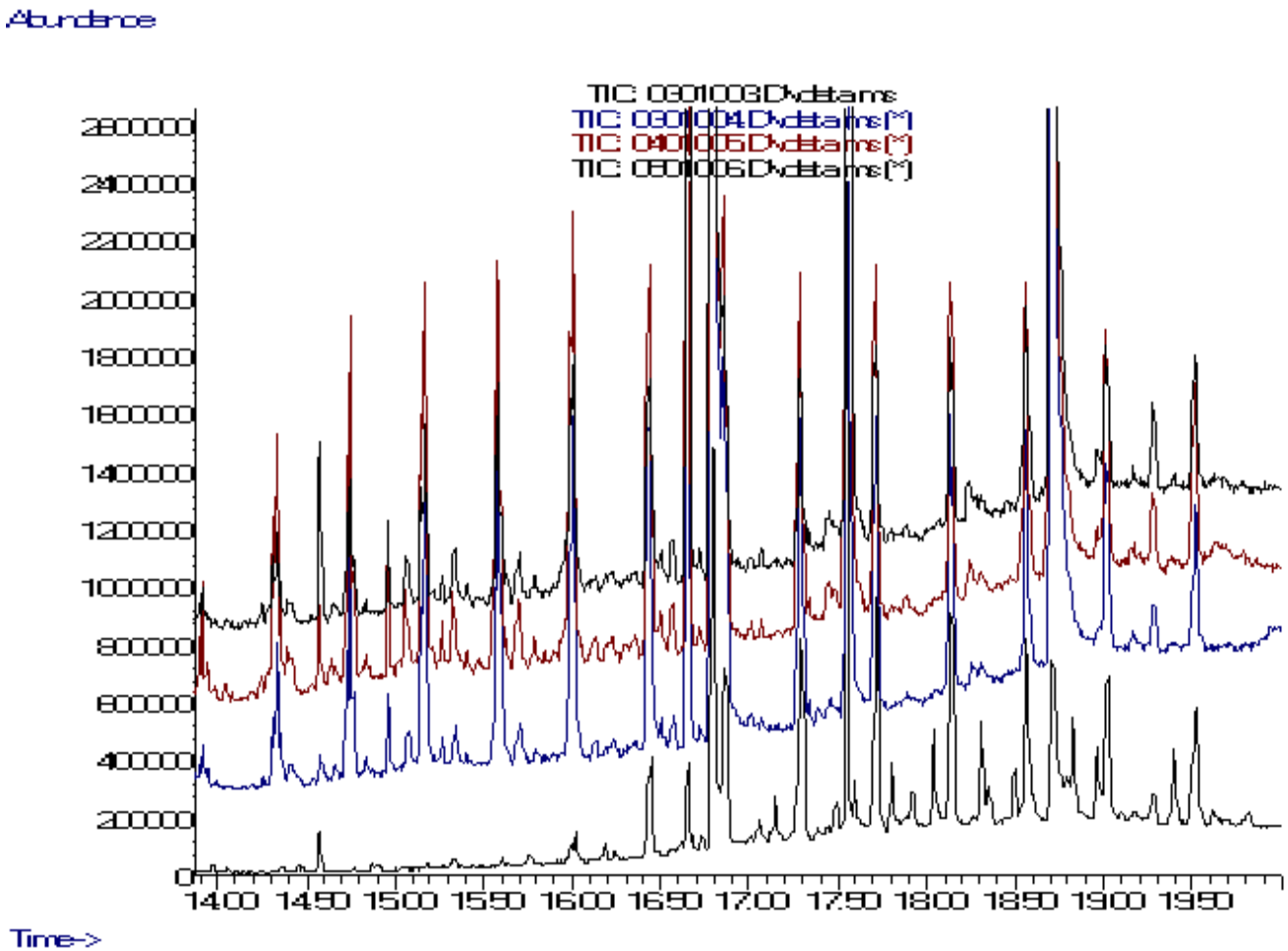
Discussion of Results

- Four samples were submitted for semivolatile organic compound (SVOC) analysis. The samples contained long chain hydrocarbons with a boiling point range of 330 to 490+ degrees Celsius. Extracted masses are tabulated in Table 2 above.

Notes

- Organic nitrates were not present in any samples, as determined by lack of characteristic [O- N-O]+ ion fragments.
- The analytes identified may be artifacts of reactive gas interactions with the Rados (R) filter material.
- The Rados (R) filter material is not adsorptive to organics, and the organic analytes captured may not be representative of analytes present in the air samples. The Rados filter medium is composed of a mixed cellulose ester material.
- A blank filter from the WIPP site could not be obtained to determine if these materials were present in the filter.
- The sample chromatograms appear to be different in comparing the CAM samples to the FAS. Refer to the overlay of chromatograms presented in Figure 1. The bottom trace represents the FAS sample, and the top three are from the CAM samples.

FAS and CAM Filter Air Sample Analysis by GC/MS



<u>Figure 1</u>. Overlay of the Chromatograms. Bottom is from FAS and top three are from CAM.

WIPP R15 C5 Sample-2 Concentrated Extract by GC/MS (LIMS 300313811)

- Sample Preparation
  - 0.24g of sample was extracted in 6.8 mL of CDCl3 for 24 hours
  - Extract was concentrated to 0.5 mL under N2 at ambient temperature
  - Extract was filtered and spiked with internal standard prior to analysis
  - Method Reporting Limit is 2 mg/kg
  - Internal Standard contains:

Retention Time	Analyte	Melting Point, ºC	Boiling Point, ºC
9.022	1,4-Dichlorobenzene-d4	54	173
10.437	Naphthalene-d8	82	218
12.248	Acenaphthene-d10	95	278
13.773	Anthracene-d10-	218	342
16.818	Chrysene-d12	252	448
18.739	Perylene-d12	278	475

- Instrument and Conditions of Analysis
  - Contained Agilent 6890/5973A GC/MS
  - Column: J&W DB-5MS, 25 m length, 0.200 mm ID, 0.33 µm film thickness
  - 0.5 µL injection
  - 0.7 mL/min flow rate of helium
  - Oven temperature program:
  - 50 ºC for 5 min, ramp 25 ºC/min to 275 ºC, ramp 15 ºC/min to 340 ºC, hold 1.67 min

WIPP R15 C5 Sample-2 Concentrated Extract by GC/MS (LIMS 300313811)

- Data

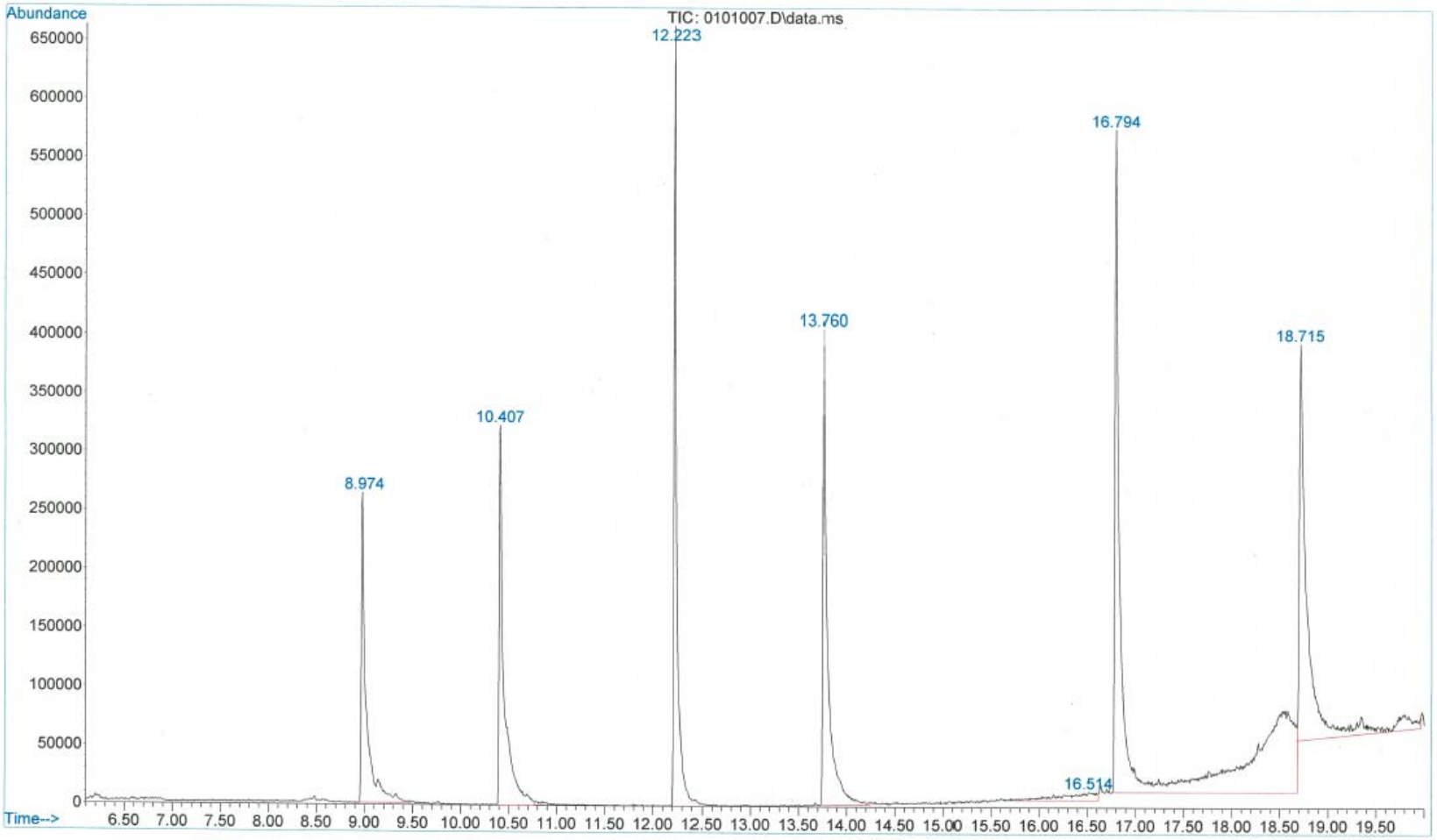
Analyte	RT, min	BP, ºC	mg/kg (ppm)
Diisooctyl Phthalate	16.660	370	22
Butyl Caprylate	11.280	242	19
Hexadecane	12.596	287	3.2
Heptadecane	13.068	302	2.8

- Chromatograms
  - Semi Volatiles Blank
  - WIPP Sample-2 R15 C5 – Concentrated Extract with Internal Standard
  - WIPP Sample-2 R15 C5 – Concentrated Extract without Internal Standard

(Note that the peaks at RT 14.035 and 14.600 are laboratory impurities)

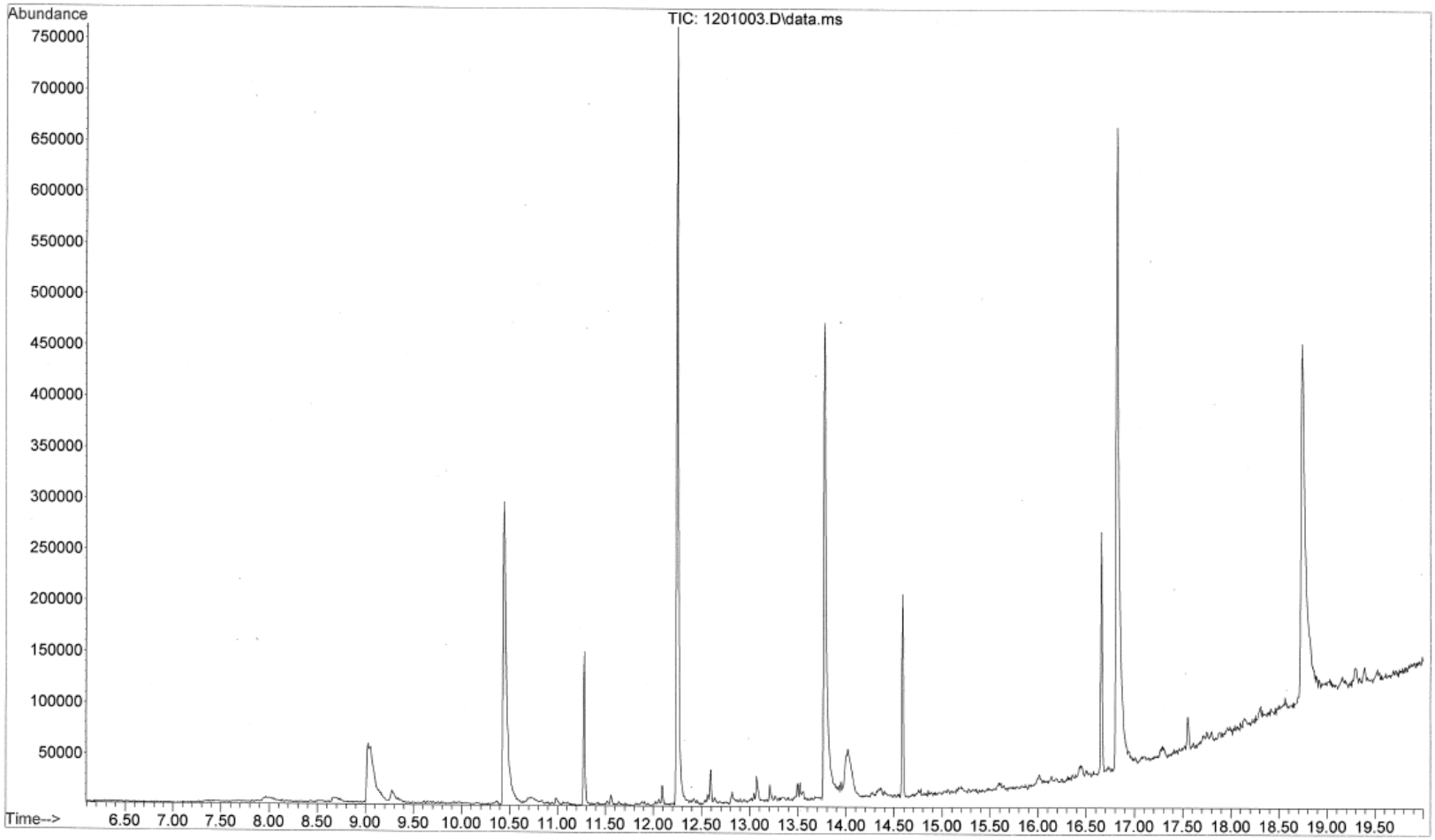
WIPP R15 C5 Sample-2 Concentrated Extract by GC/MS (LIMS 300313811)

- Semi Volatile Blank



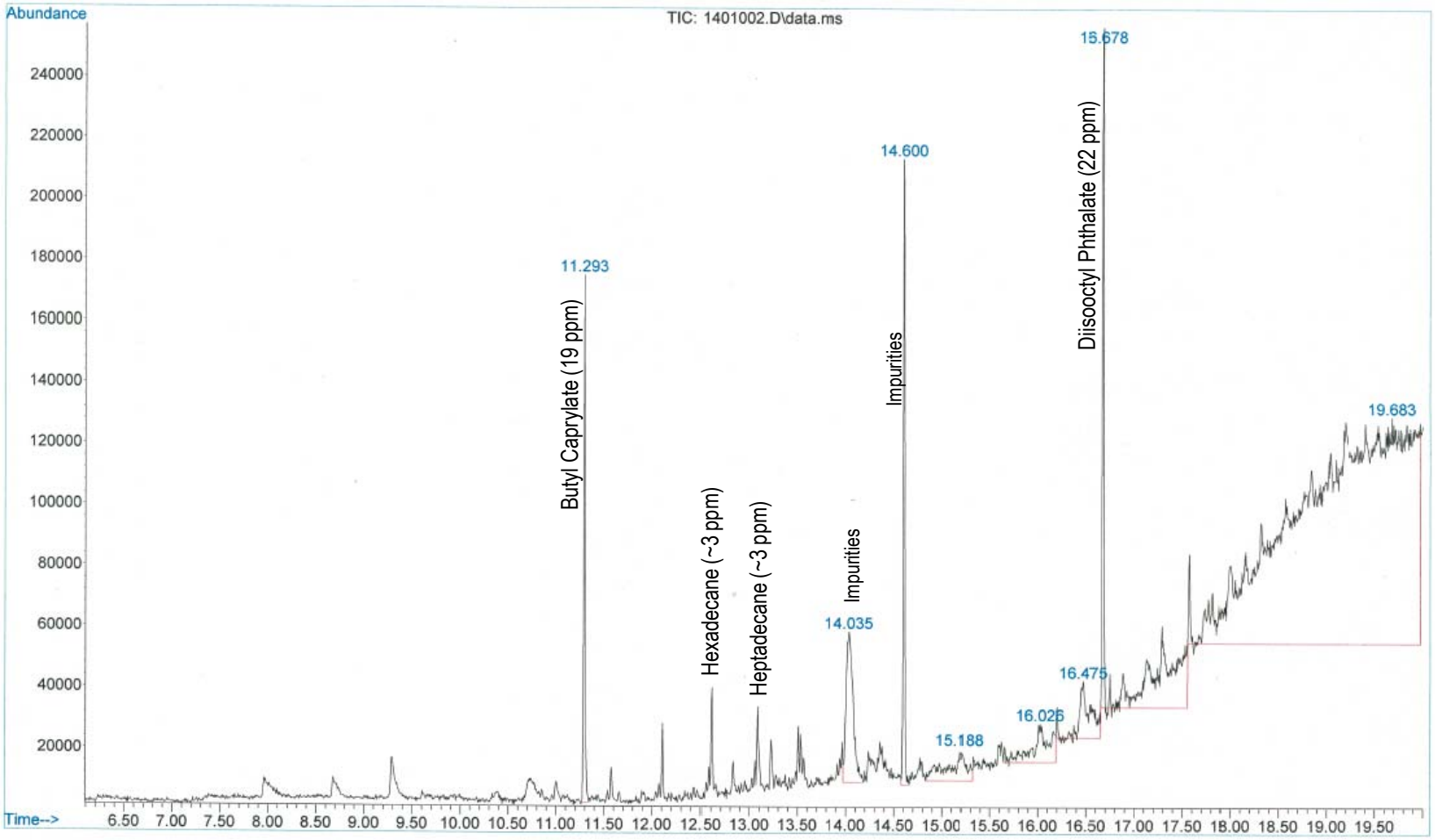
WIPP R15 C5 Sample-2 Concentrated Extract by GC/MS (LIMS 300313811)

- WIPP Sample-2 R15 C5 – Concentrated Extract with Internal Standard



WIPP R15 C5 Sample-2 Concentrated Extract by GC/MS (LIMS 300313811)

- WIPP Sample-2 R15 C5 – Concentrated Extract without Internal Standard



LANL Parent Drum Debris Concentrated Extract by GC/MS (LIMS 300313607)

- Sample Preparation
  - 1.0g of sample was extracted in 6.8 mL of CDCl3 for 24 hours
  - Extract was concentrated to 0.5 mL under N2 at ambient temperature
  - Extract was filtered and spiked with internal standard prior to analysis
  - Method Reporting Limit is 0.5 mg/kg
  - Internal Standard contains:

Retention Time	Analyte	Melting Point, ºC	Boiling Point, ºC
9.022	1,4-Dichlorobenzene-d4	54	173
10.437	Naphthalene-d8	82	218
12.248	Acenaphthene-d10	95	278
13.773	Anthracene-d10-	218	342
16.818	Chrysene-d12	252	448
18.739	Perylene-d12	278	475

- Instrument and Conditions of Analysis
  - Contained Agilent 6890/5973A GC/MS
  - Column: J&W DB-5MS, 25 m length, 0.200 mm ID, 0.33 µm film thickness
  - 0.5 µL injection
  - 0.7 mL/min flow rate of helium
  - Oven temperature program:
  - 50 ºC for 5 min, ramp 25 ºC/min to 275 ºC, ramp 15 ºC/min to 340 ºC, hold 1.67 min

LANL Parent Drum Debris Concentrated Extract by GC/MS (LIMS 300313607)

- Data

Analyte	Retention Time, min	Boiling Point, ^{o}C	Concentration, mg/kg
Hexadecanoic Acid	14.216	352	15
Nonanal	9.72	195	7.3
2-Ethyl-1-hexanoic Acid	9.79	228	6.0
Hexachloroethane	9.59	187	1.7
9-Hexadecenoic Acid	14.903	360	0.66

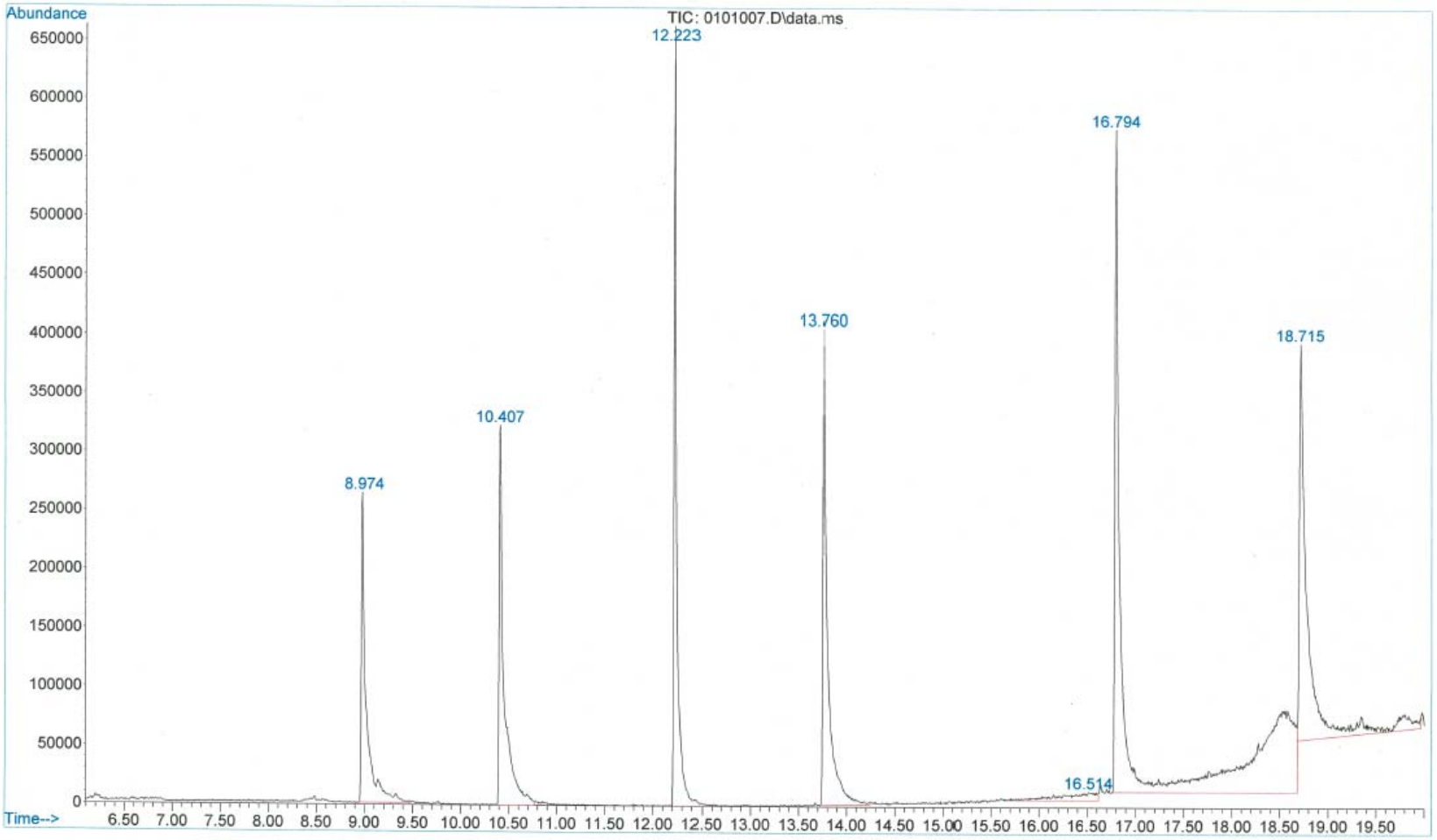
- Analytical Notes
  - Hexadecanoic Acid is Palmitic Acid
  - 9-Hexadecenoic Acid is Oleic Acid

- Chromatograms
  - Semi Volatiles Blank
  - LANL Parent Drum Debris Archive B Sample – Concentrated Extract with Internal Standard
  - LANL Parent Drum Debris Archive B Sample – Concentrated Extract without Internal Standard

(Note that the peaks at RT 14.035 and 14.600 are laboratory impurities)

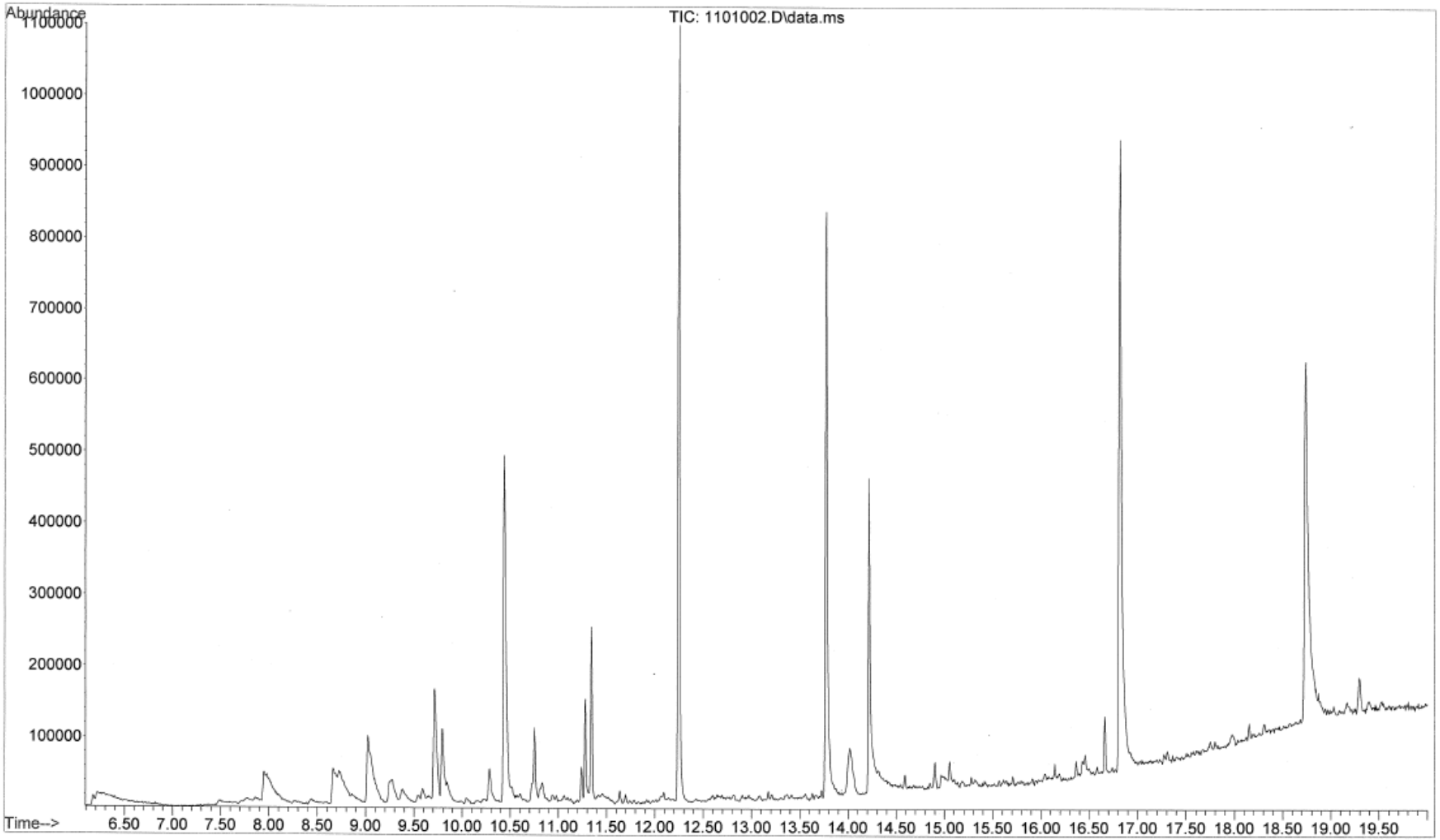
LANL Parent Drum Debris Concentrated Extract by GC/MS (LIMS 300313607)

- Semi Volatile Blank



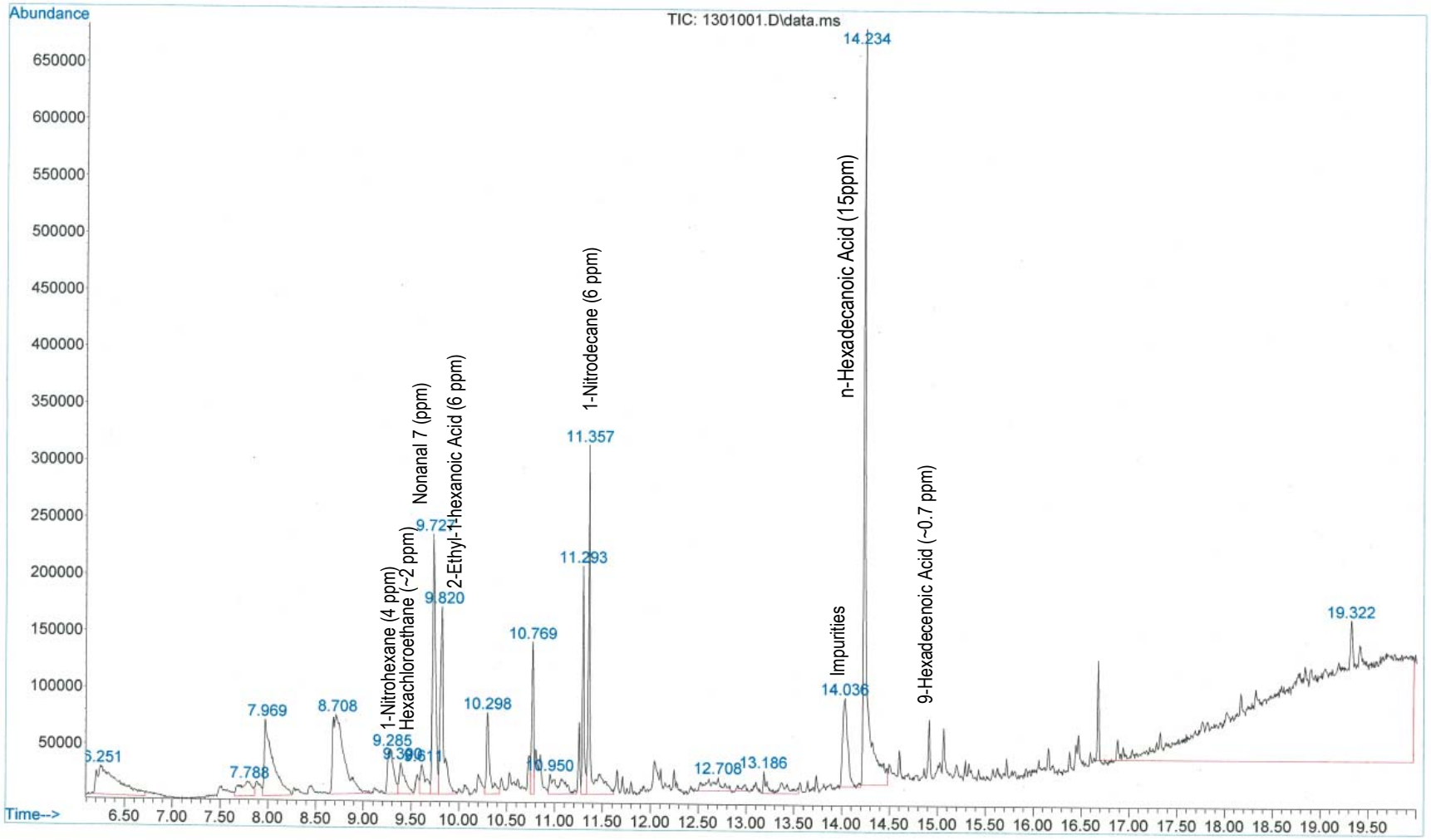
LANL Parent Drum Debris Concentrated Extract by GC/MS (LIMS 300313607)

- LANL Parent Drum Debris Archive B Sample – Concentrated Extract with Internal Standard



LANL Parent Drum Debris Concentrated Extract by GC/MS (LIMS 300313607)

- LANL Parent Drum Debris Archive B Sample – Concentrated Extract without Internal Standard



WIPP Non Deployed Swheat Analysis by GC/MS

- Sample Preparation
  - 2.5g of sample was extracted 3x with 5 mL of CH_2Cl_2
  - Extract was concentrated to 1.0 mL under N2 at ambient temperature
  - Extract was filtered and spiked with internal standard prior to analysis
  - Method Reporting Limit is 0.5 mg/kg
  - Internal Standard contains:

Retention Time	Analyte	Melting Point, ºC	Boiling Point, ºC
9.022	1,4-Dichlorobenzene-d4	54	173
10.437	Naphthalene-d8	82	218
12.248	Acenaphthene-d10	95	278
13.773	Anthracene-d10-	218	342
16.818	Chrysene-d12	252	448
18.739	Perylene-d12	278	475

- Instrument and Conditions of Analysis
  - Contained Agilent 6890/5973A GC/MS
  - Column: J&W DB-5MS, 25 m length, 0.200 mm ID, 0.33 µm film thickness
  - 0.5 µL injection
  - 0.7 mL/min flow rate of helium
  - Oven temperature program:
  - 50 ºC for 5 min, ramp 25 ºC/min to 275 ºC, ramp 15 ºC/min to 340 ºC, hold 1.67 min

WIPP Non Deployed Swheat Analysis by GC/MS

- Data

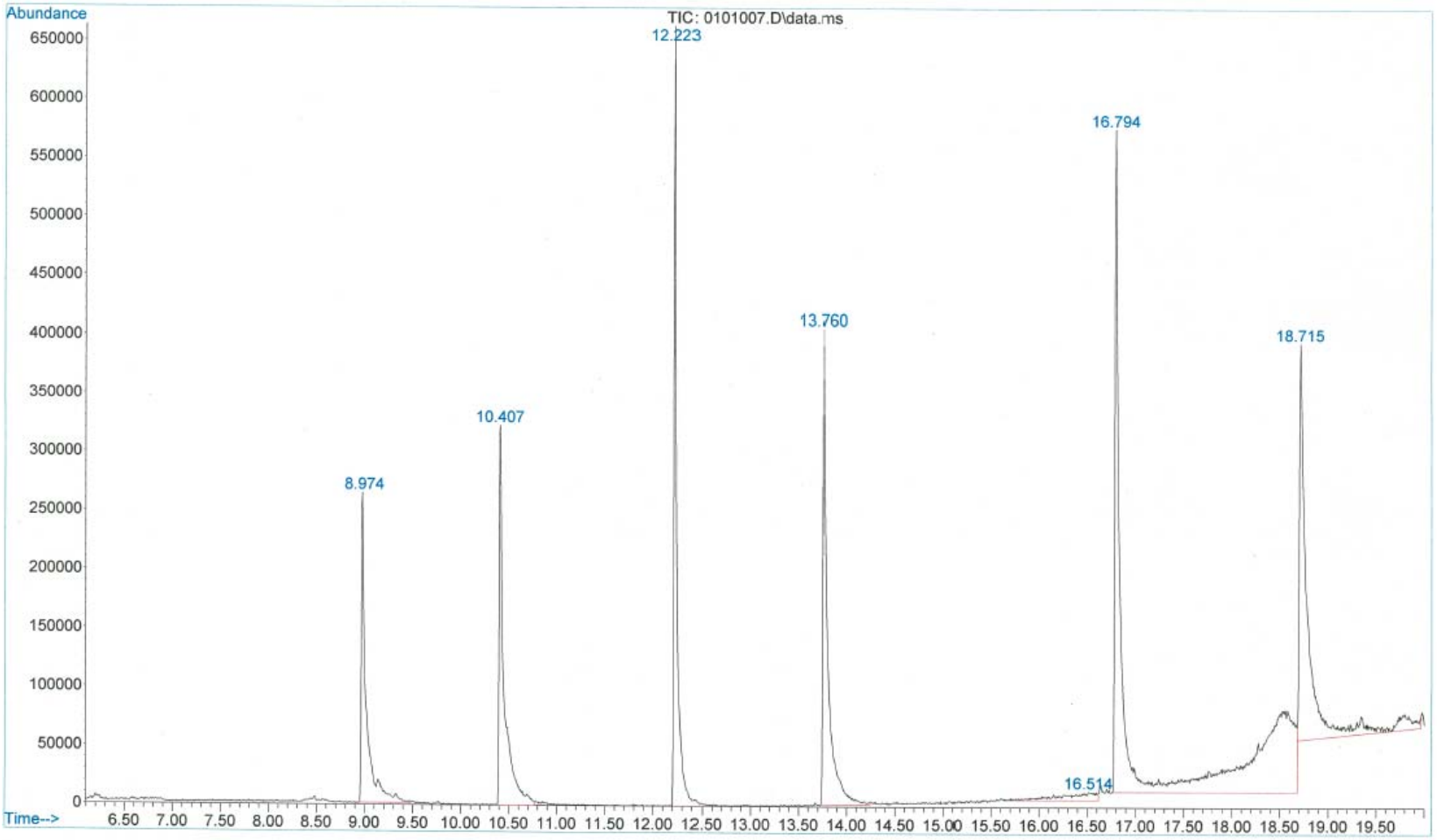
Analyte	RT, min	BP, ºC	Concentration, mg/kg (ppm)
(Z,Z)-9,12-Octadecadienoic Acid	15.165	407	1200
n-Hexadecanoic Acid	14.344	352	240
Tricosane	16.528	453	6.4

- Analytical Notes
  - (Z,Z)-9,12 Octadecadienoic Acid is Linoleic Acid
  - n-Hexadecanoic Acid is Palmitic Acid

- Chromatograms
  - Semi Volatiles Blank
  - WIPP Non Deployed Swheat

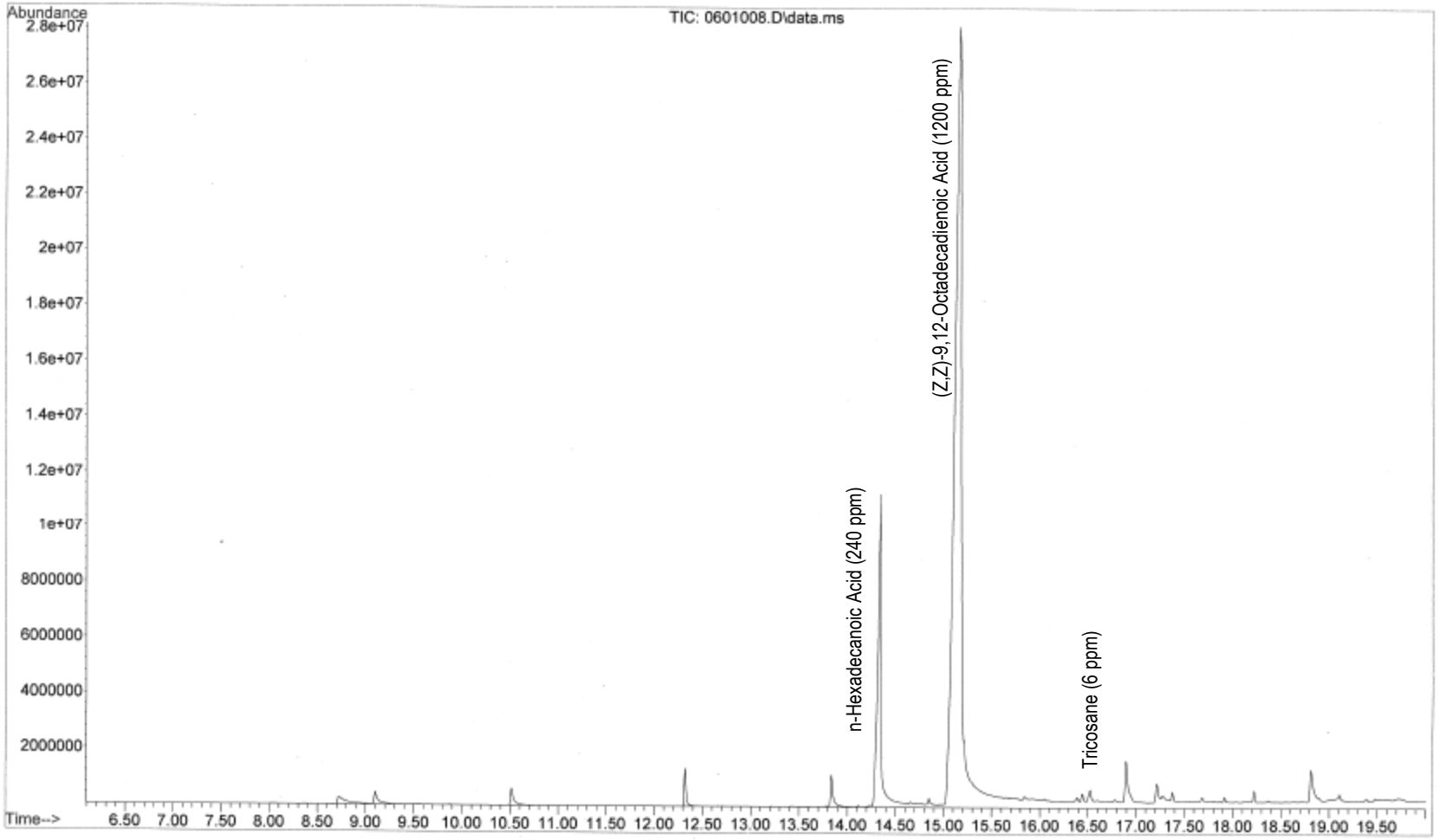
WIPP Non Deployed Swheat Analysisby GC/MS (LIMS 300313607)

- Semi Volatile Blank



WIPP Non Deployed Swheat Analysis by GC/MS

- WIPP Non Deployed Swheat – Concentrated Extract



GC/MS Analysis of Derivatized Extracts

Sample ID

Customer ID	ADS No.	Extract Mass, mg
Debris Extract	300313607	1.1
R15C5 Sample_2	300313811	0.1

Discussion of Results

- Two samples were submitted for semivolatile organic compound (SVOC) analysis. The method detection limit (mdl) for this study was one mg/L for sample extracts derived from samples 300313607 and 300313811.

- Derivatizations were carried out on each extract in two ways. Half of each extract was treated with N-Methylbis-(trifluoroacetamide) (MBTFA), and half was treated with BF3-Butanol (BF3). The MBTFA reagent is best for trifluoroacylation of carbohydrates and sugars, and the BF3 reagent is best for esterifying carboxylic acids to their butyl esters. The chromatograms resulting from the analysis of these derivatized extracts will then show any carbohydrates, sugars, or carboxylic acids in derivatized form which was originally contained in the sample extracts.

- Data files were searched for presence of NO2+ (nitronium, m/z = 46) and H4PO4+ (m/z = 99), and neither ion was found. This indicates the absence of nitrated organics, as well as organic phosphates (such as tributyl or trimethyl phosphate).

GC/MS Analysis of Derivatized Extracts

Internal Standard contains:

Retention Time	Analyte	Melting Point, ºC	Boiling Point, ºC
9.022	1,4-Dichlorobenzene-d4	54	173
10.437	Naphthalene-d8	82	218
12.248	Acenaphthene-d10	95	278
13.773	Anthracene-d10-	218	342
16.818	Chrysene-d12	252	448
18.739	Perylene-d12	278	475

Instrument and Conditions of Analysis

- Contained Agilent 6890/5973A GC/MS
- Column: J&W DB-5MS, 25 m length, 0.200 mm ID, 0.33 µm film thickness
- 0.5 µL injection
- 0.7 mL/min flow rate of helium
- Oven temperature program:
- 50 ºC for 5 min, ramp 25 ºC/min to 275 ºC, ramp 15 ºC/min to 340 ºC, hold 1.67 min

GC/MS Analysis of Derivatized Extracts

Notable analytes on Page 99 of the chromatogram for BF_3-Butanol derivatization of the R15C5 extract are as follows

Analyte	RT, minutes
Dibutyl ether	7.758
Dibutyldimethylsiloxane	9.703
Siloxane	9.779
Butyl formate	9.866
1,1-Dibutoxybutane	10.716
Tributyl borate	11.249
Siloxane	13.391
Siloxane	15.080
Siloxane	15.545
Plasticizer	15.854

Notable analytes seen on Page 101 of the chromatogram for MBTFA derivatization of the R15C5 Extract are as follows

Analyte	RT, minutes
Pyridine	8.250
1,2,4-Trimethylbenzene	8.704
Siloxane	9.106
N-Cyclopentyl-trifluoroacetamide	9.222
N-Methyl-m-hemipimide	10.189
Unidentified	13.47
Plasticizer	16.401

Notable analytes seen on Page 100 of the chromatogram for BF_3-Butanol derivatization of the Debris Extract are as follows

Analyte	RT, minutes
Dibutyl ether	7.740
Dibutyldimethylsiloxane	9.702
Siloxane	9.772
Butyl formate	9.866
1,1-Dibutoxybutane	10.715
Tributyl borate	11.001
Internal Standard	14.626
Dibutyl azelate	14.696
Butyl palmitate	15.121
Plasticizer	15.849

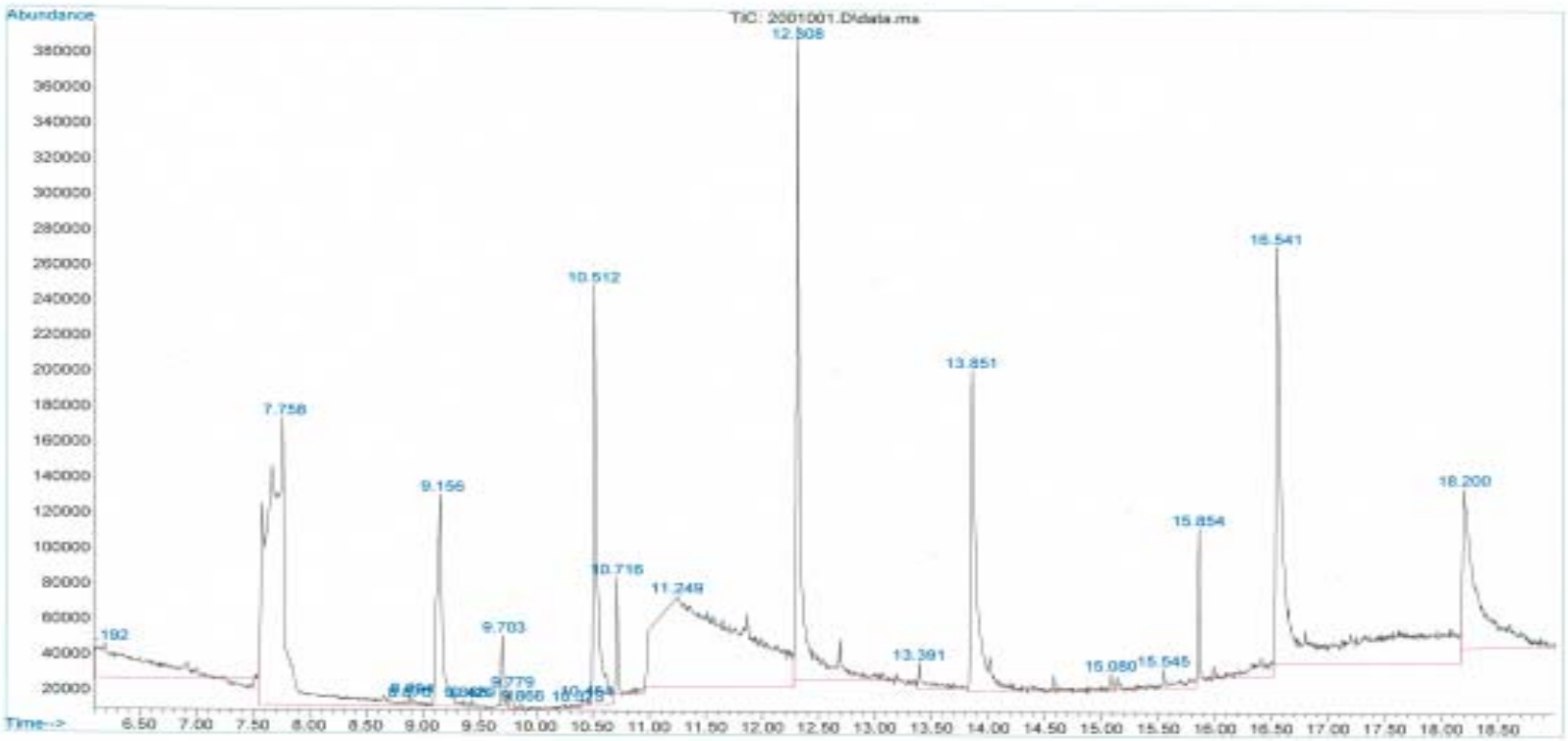
Notable analytes seen on Page 102 of the chromatogram for MBTFA derivatization of the Debris Extract are as follows

Analyte	RT, minutes
Perfluroracetic Acid	7.5
Pyridine	8.214
1,2,4-Trimethylbenzene	8.685
N-Cyclopentyl-trifluoroacetamide	9.221
Siloxane	9.6
Unidentified	9.733
2-Ethyl-1-dodecanol	13.017
Unidentified	13.459
Siloxane	16.411

GC/MS Analysis of Derivatized Extracts

BF_3 Butanol Derivative of WIPP R15 C5 Sample - 2 Concentrated Extract - Analysis by GC/MS

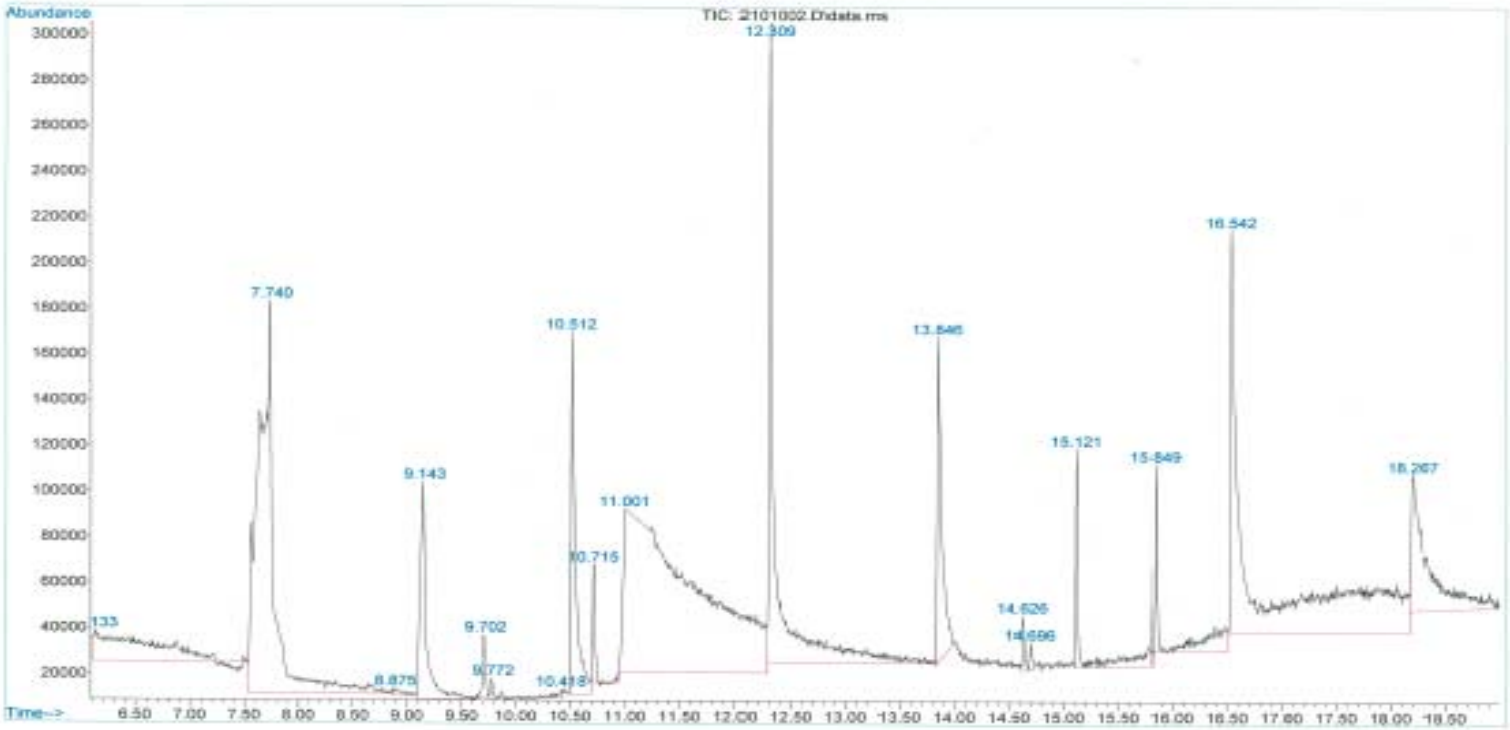
```
File        :F:\GCMS\svoa\112014A\2001001.D
Operator    :
Acquired    : 20 Nov 2014  18:02     using AcqMethod SVOA35
Instrument  :   GC/MS Ins
Sample Name: r15C5 Extract, Lab Number 300313811
Misc Info   : BF3 DERIVATIZATION, 0.05 mg Extract
Vial Number: 20
```



GC/MS Analysis of Derivatized Extracts

BF_3 – Butanol Derivative of Parent Drum Debris Concentrated Extract – Analysis by GC/MS

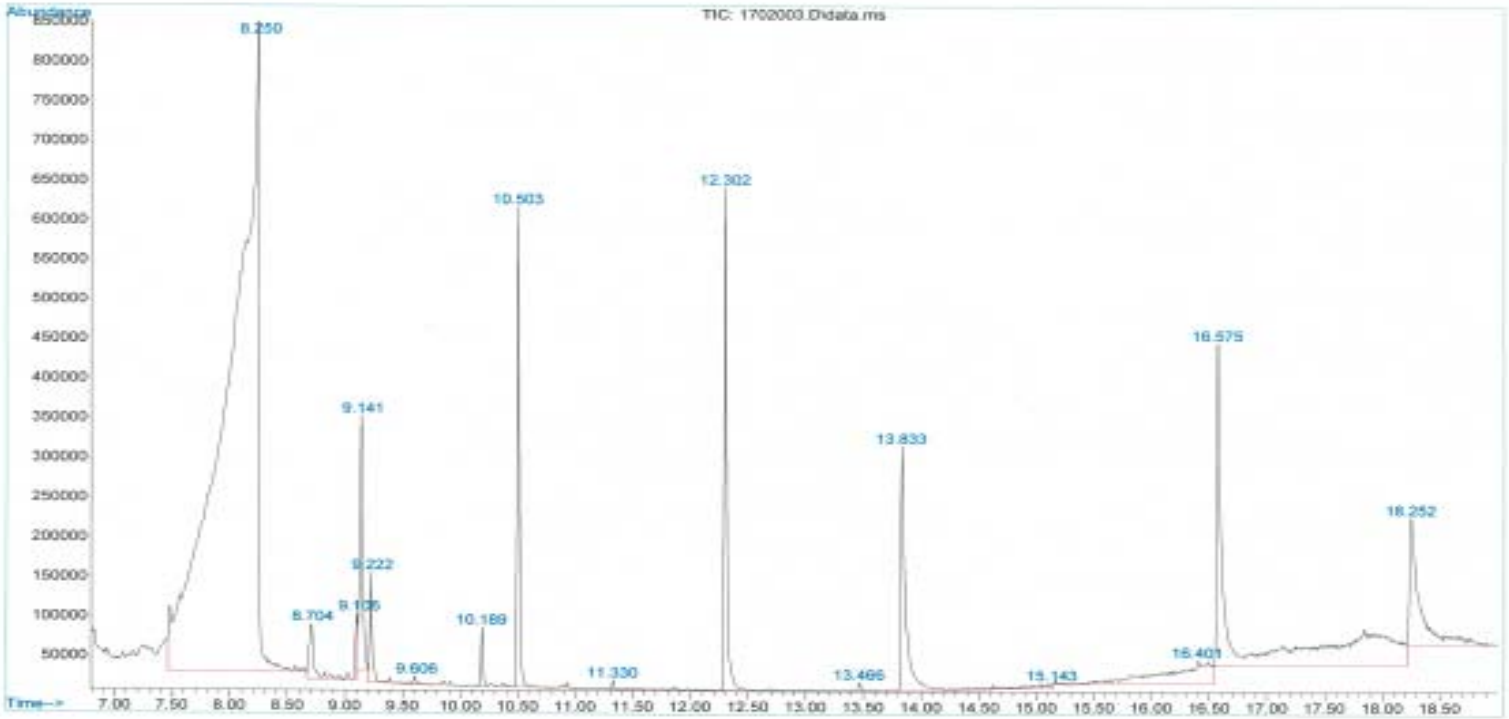
```
File        :F:\GCMS\svoa\112014A\2101002.D
Operator    :
Acquired    : 20 Nov 2014  18:29     using AcqMethod SVOA35
Instrument  :   GC/MS Ina
Sample Name: Debris Sample Extract, Lab Number 300313607
Misc Info   : BF3 DERIVATIZATION, 0.55 mg Extract
Vial Number: 21
```



GC/MS Analysis of Derivatized Extracts

MBTFA Derivative of WIPP R15 C5 Sample - 2 Concentrated Extract - Analysis by GC/MS

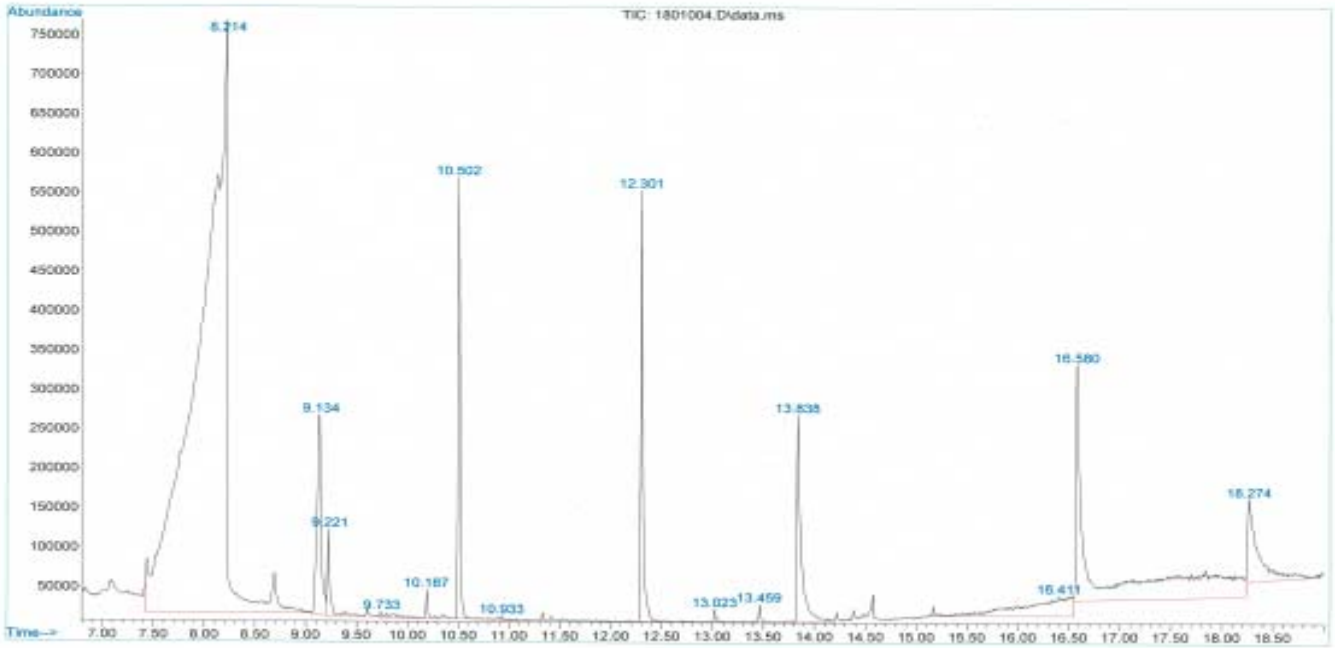
```
File        :F:\GCMS\svoa\112014A\1702003.D
Operator    :
Acquired    : 20 Nov 2014    9:40      using AcqMethod SVOA35
Instrument  :    GC/MS Ins
Sample Name: R15C5 Extract, Lab Number 300313811
Misc Info   :  MBTFA DERIVATIVE, 0.05 mg Extract
Vial Number: 17
```



GC/MS Analysis of Derivatized Extracts

MBTFA Derivative of Parent Drum Debris Concentrated Extract – Analysis by GC/MS

```
File        :F:\GCMS\svoa\112014A\1801004.D
Operator    :
Acquired    : 20 Nov 2014  10:07      using AcqMethod SVOA35
Instrument  :   GC/MS Ins
Sample Name: Debris Sample, Lab Number 300313607
Misc Info   : MBTFA DERIVATIVE, 0.55 mg Extract
Vial Number: 18
```



GC/MS Data – Analysis for TEA

Customer ID	Lab No.	Extractable Mass, mg	Sample Mass, grams	MRL, ppm
Sample_2 (R15 C5, WIPP UG 8/15)	300313811	unknown	0.2369	2
Archive B, Solid (Debris Extract)	300313607	unknown	1	0.5

Discussion of Results

- Two samples were analyzed for semivolatile organic compounds (SVOC), specifically to determine if triethanolamine (TEA) was present, and no significant findings were made. The method reporting limits (MRL) for this study are in the table above. The method reporting limit for TEA is approximately the same as for hydrocarbons (at least within an order of magnitude), even though the compound chomatogaphs poorly.
- No TEA was detected in either sample extract.

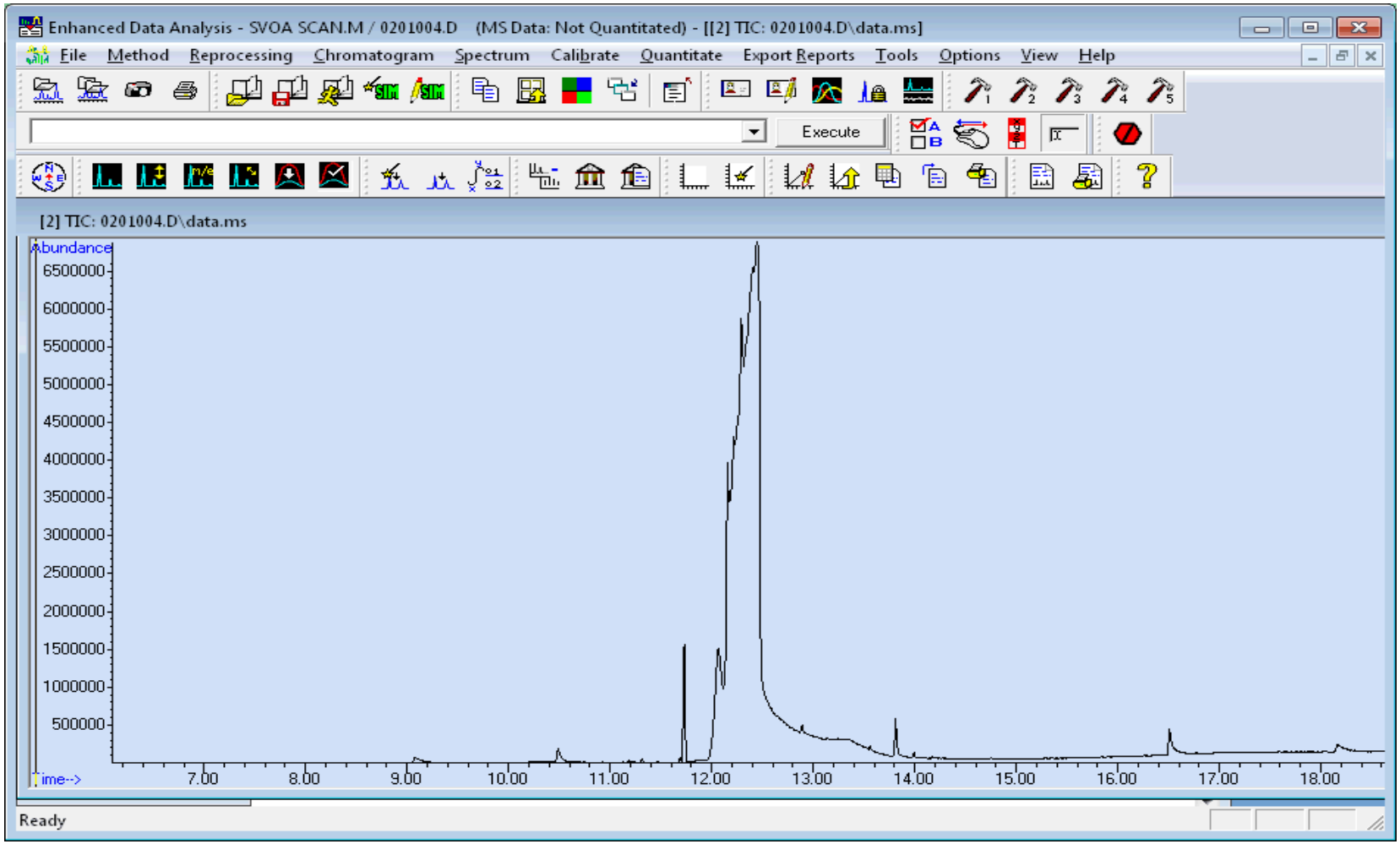
Notes:

1. Samples 300313607 and 300313811 were received as CDCl3 extracts of the original sample materials because of high dose associated with the samples.
2. The one sigma error associated with each value is +/- 10%.
3. The MRL is based on the weight of sample material extracted.
4. Data files were searched for presence of TEA based on the mass spectrum below, using m/z = 46) and H4PO4+ (Phosphate ion, m/z = 99), and neither ion was found. This indicates the absence of nitrated organics, as well as organic phosphates
5. Because of the basicity of TEA the extraction of this compound from a solid matrix sample would be adversely influenced by the presence of acidic components in the solid matrix. The reference chromatogram shown in Figure 1 was obtained from a methylene chloride solution of TEA.

The chromatogram and mass spectrum for TEA are shown below in Figures 1 and 2, respectively.

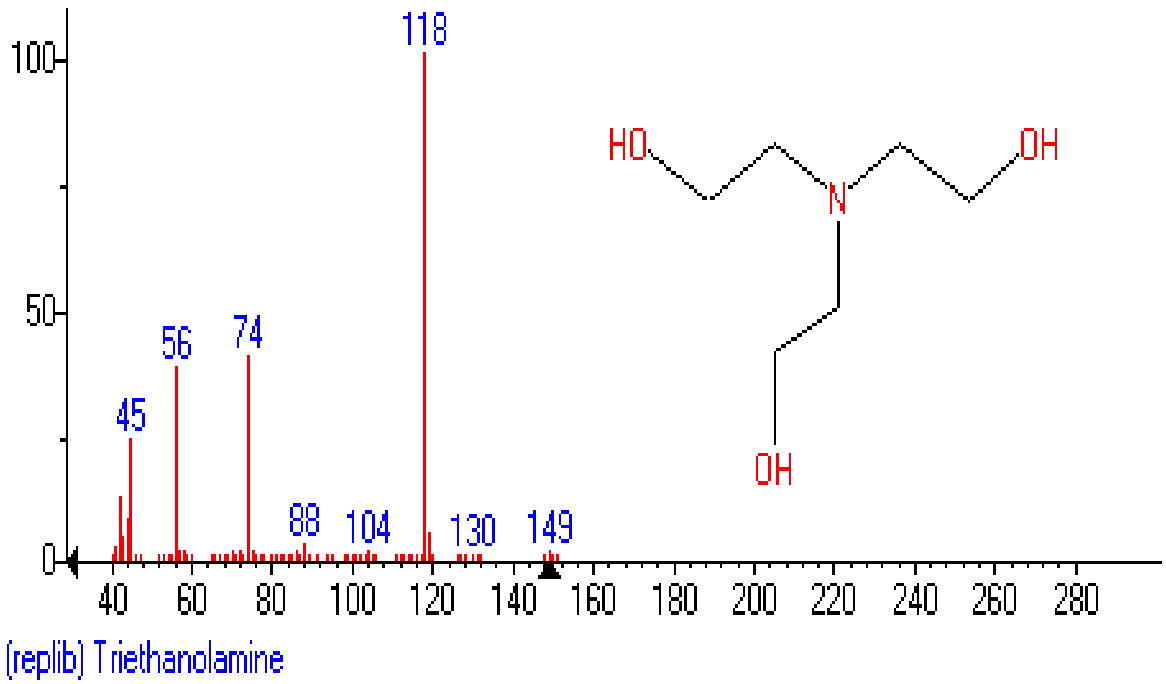
GC/MS Data – Analysis for TEA

Figure 1. Chromatogram of High Concentration TEA in Methylene Chloride.



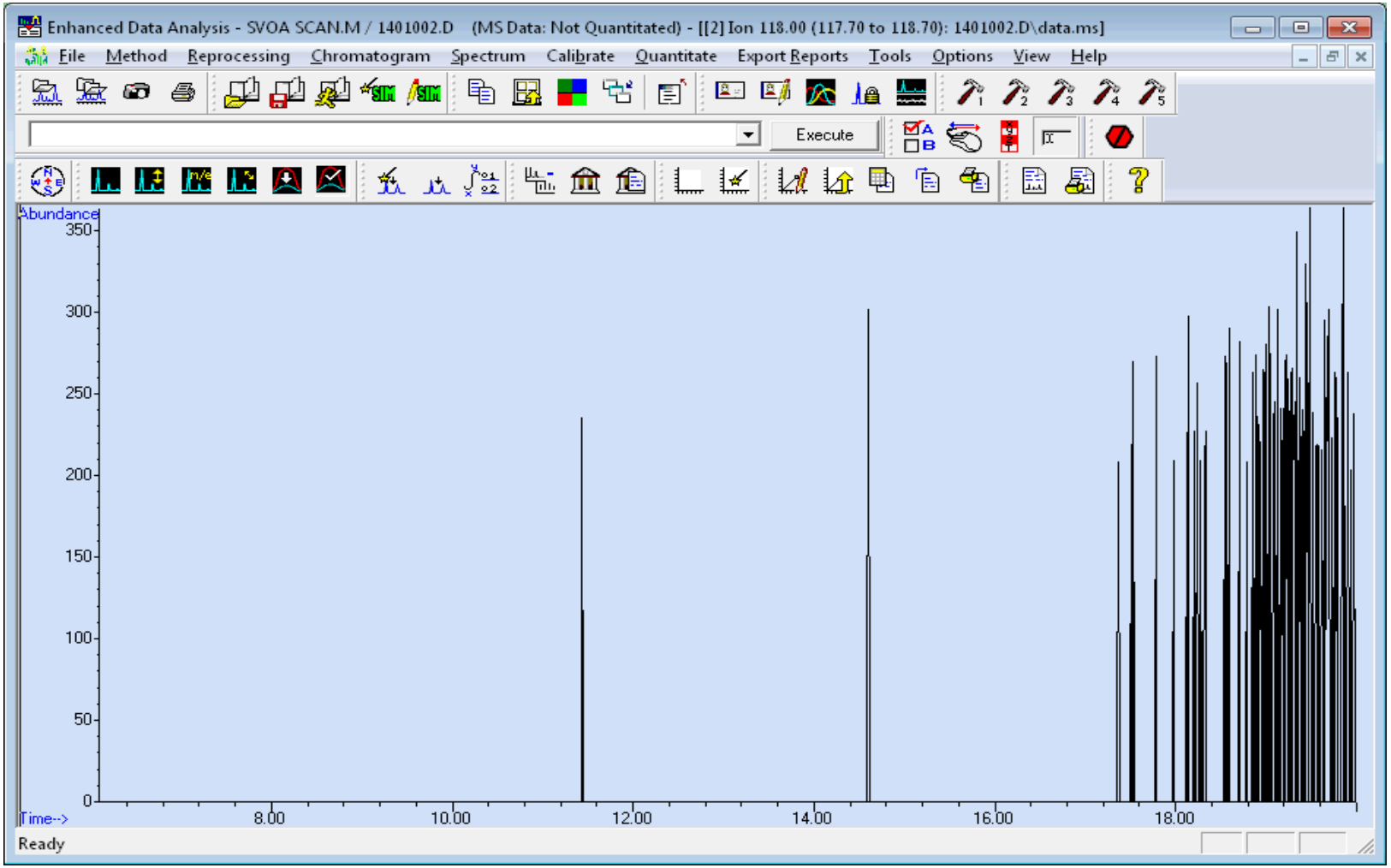
GC/MS Data – Analysis for TEA

Figure 2. Mass Spectrum of TEA from the NIST Mass Spectral Database.



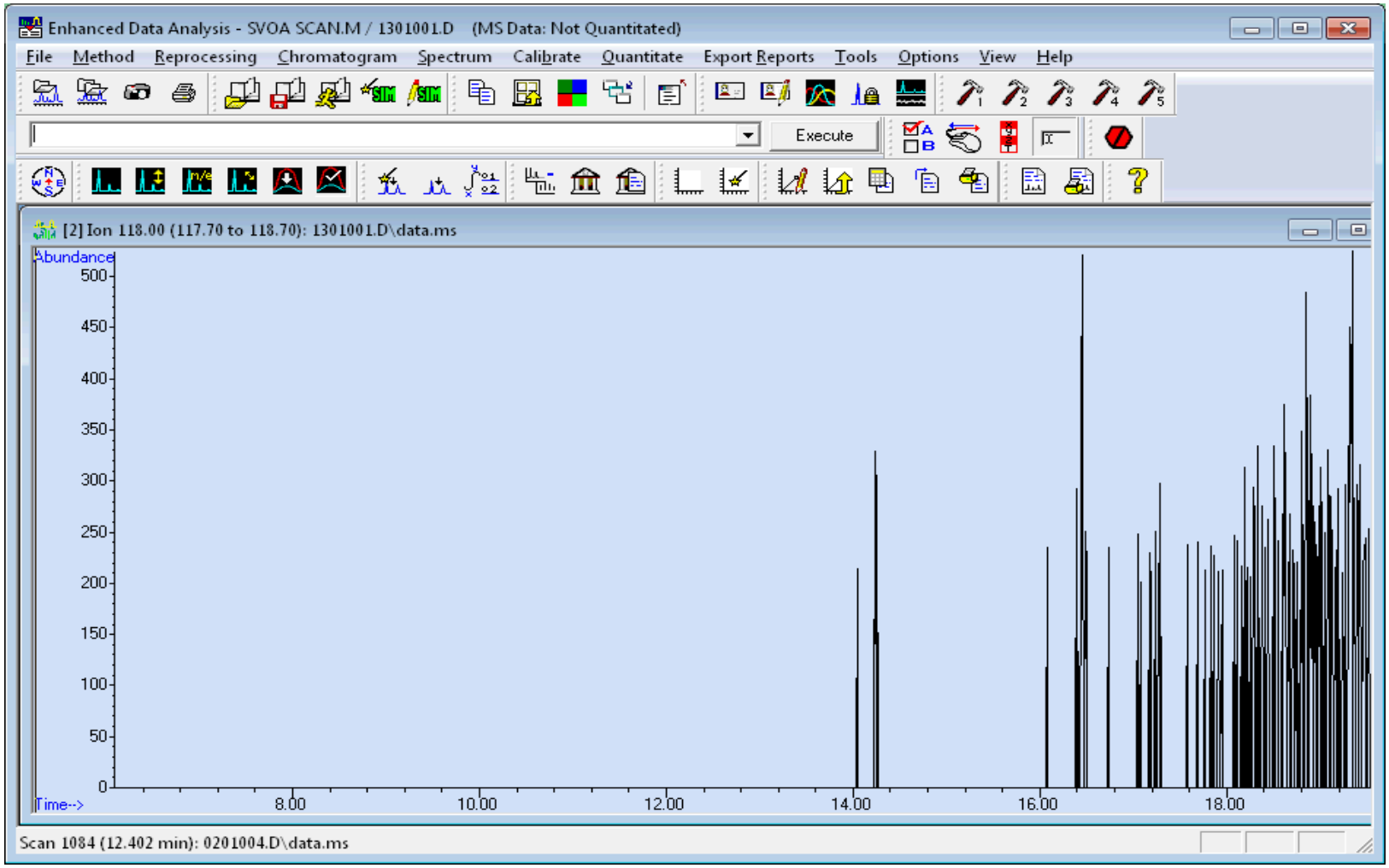
GC/MS Data – Analysis for TEA

Figure 3. Extracted Ion Chromatogram of R15C5 Extract Sample, Evaluating Presence of m/z 118 ion (TEA - [CH2=OH]+)



GC/MS Data – Analysis for TEA

Figure 4. Extracted Ion Chromatogram of the Debris Extract Sample, Evaluating Presence of m/z 118 ion



FTIR Data

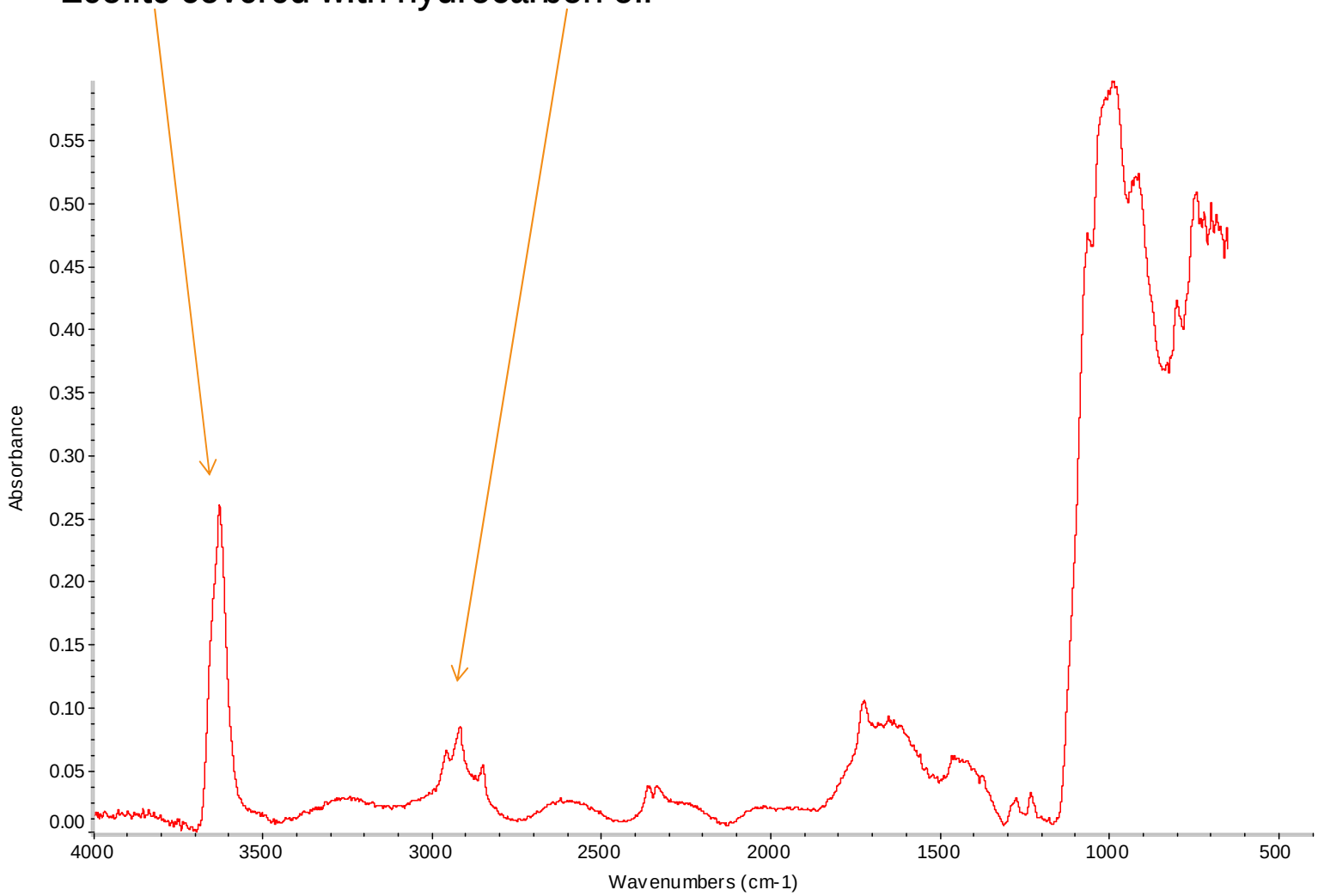
WIPP R16C4 Sample #1 Extract by FTIR (LIMS 300313914)

- Material Preparation by Extraction Method
  - Approximately 0.1 g of as received granular material was placed in a Teflon capped glass containing 5 mL of Dichloromethane. The glass vial was spun end over end every 10 seconds for 48 hours. After which the solution was decanted and collected, leaving behind the granular material. The decanted solution was allowed to dry on a KBr disc in a desiccator. The KBr was then placed on a microscope stage.

- Instrument Conditions
  - Sealed radioactive materials were placed in a Nicolet Continuum FTIR microscope connected to a Nicolet Nexus Spectrometer. Each spectrum is the co-addition of 128 scans at resolution 4 cm^{-1} and apodized with a box car function.

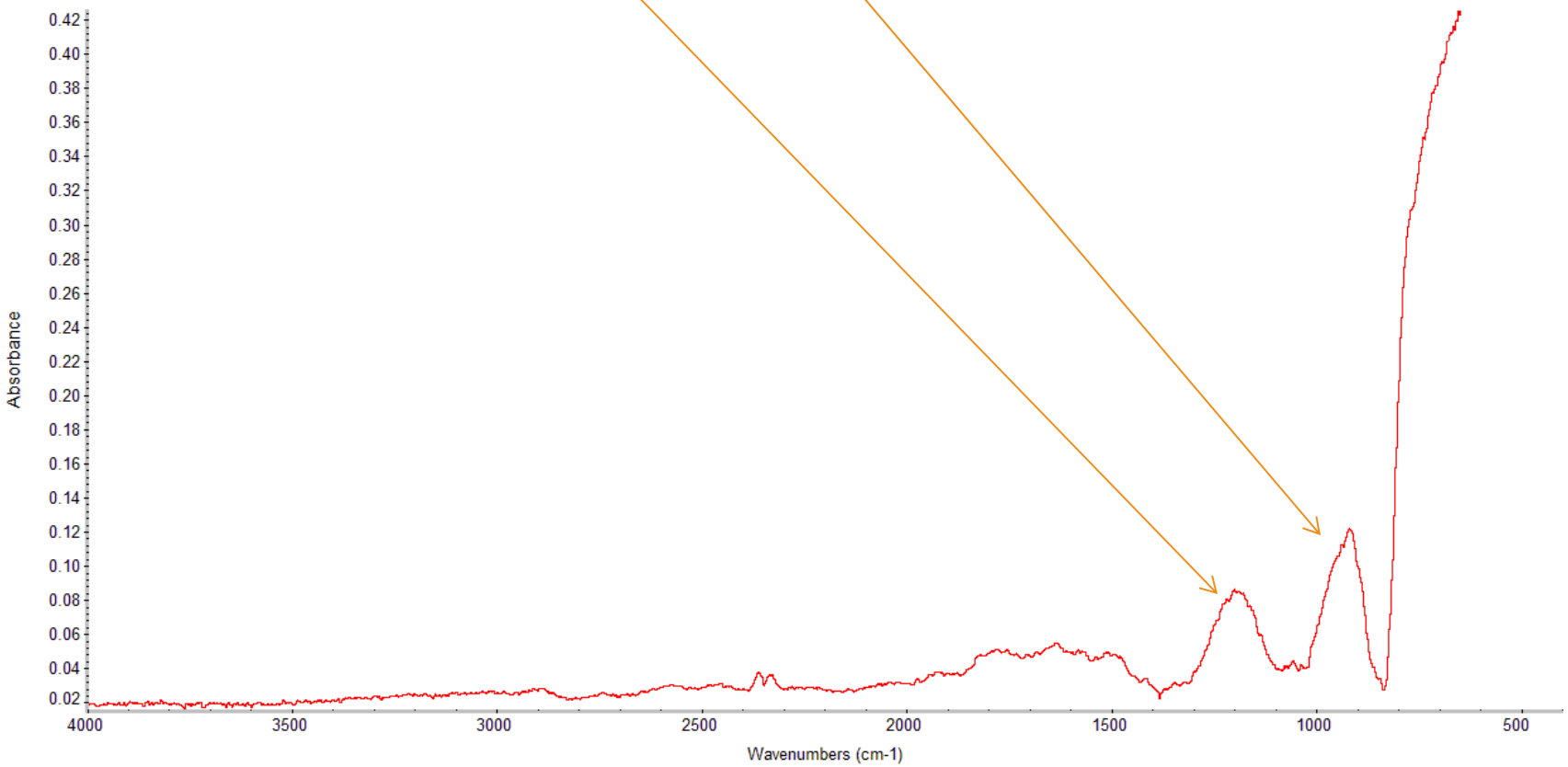
WIPP R16C4 Sample #1 Extract by FTIR (LIMS 300313914)

- Zeolite covered with hydrocarbon oil



WIPP R16C4 Sample #1 Extract by FTIR (LIMS 300313914)

- MgO with some carbonate and silicates



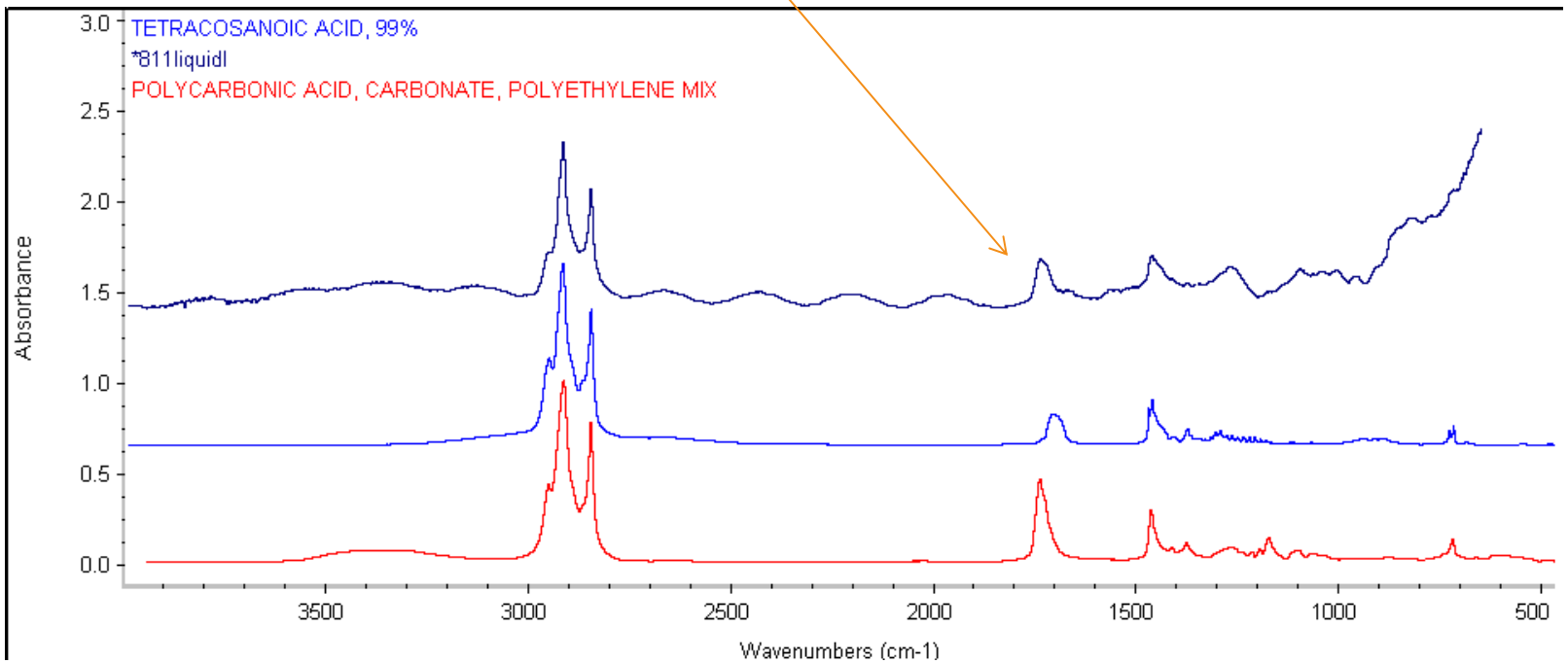
WIPP R15C5 Sample #2 Extract by FTIR (LIMS 300313811)

- Material Preparation by Extraction Method
  - Approximately 0.1 g of as received granular material was placed in a Teflon capped glass containing 5 mL of Dichloromethane. The glass vial was spun end over end every 10 seconds for 48 hours. After which the solution was decanted and collected, leaving behind the granular material. The decanted solution was allowed to dry on a KBr disc in a desiccator. The KBr was then placed on a microscope stage.

- Instrument Conditions
  - Sealed radioactive materials were placed in a Nicolet Continuum FTIR microscope connected to a Nicolet Nexus Spectrometer. Each spectrum is the co-addition of 128 scans at resolution 4 cm^{-1} and apodized with a box car function.

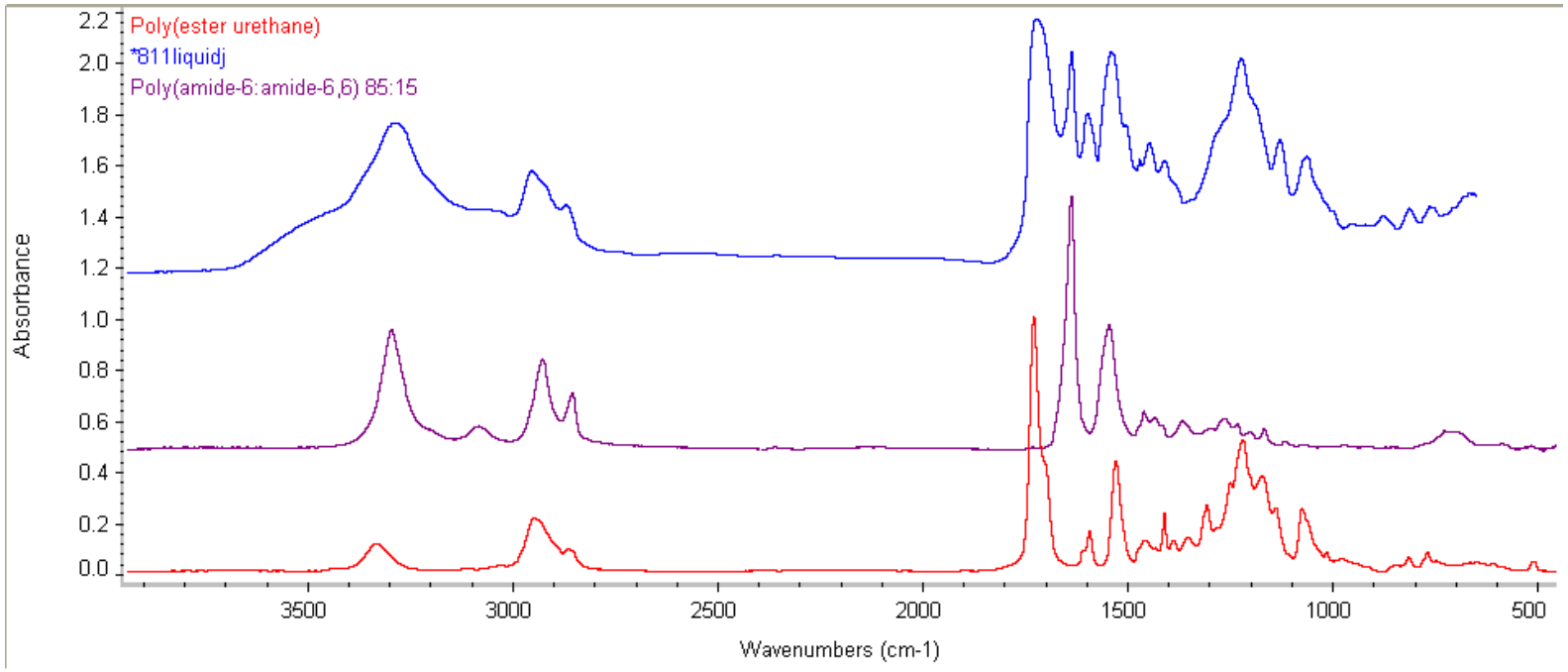
WIPP R15C5 Sample #2 Extract by FTIR (LIMS 300313811)

- (Organics – of Extract) : Ester Oil. The oscillation is due to birefrigence or light interference that results from having two closely-spaced discs.



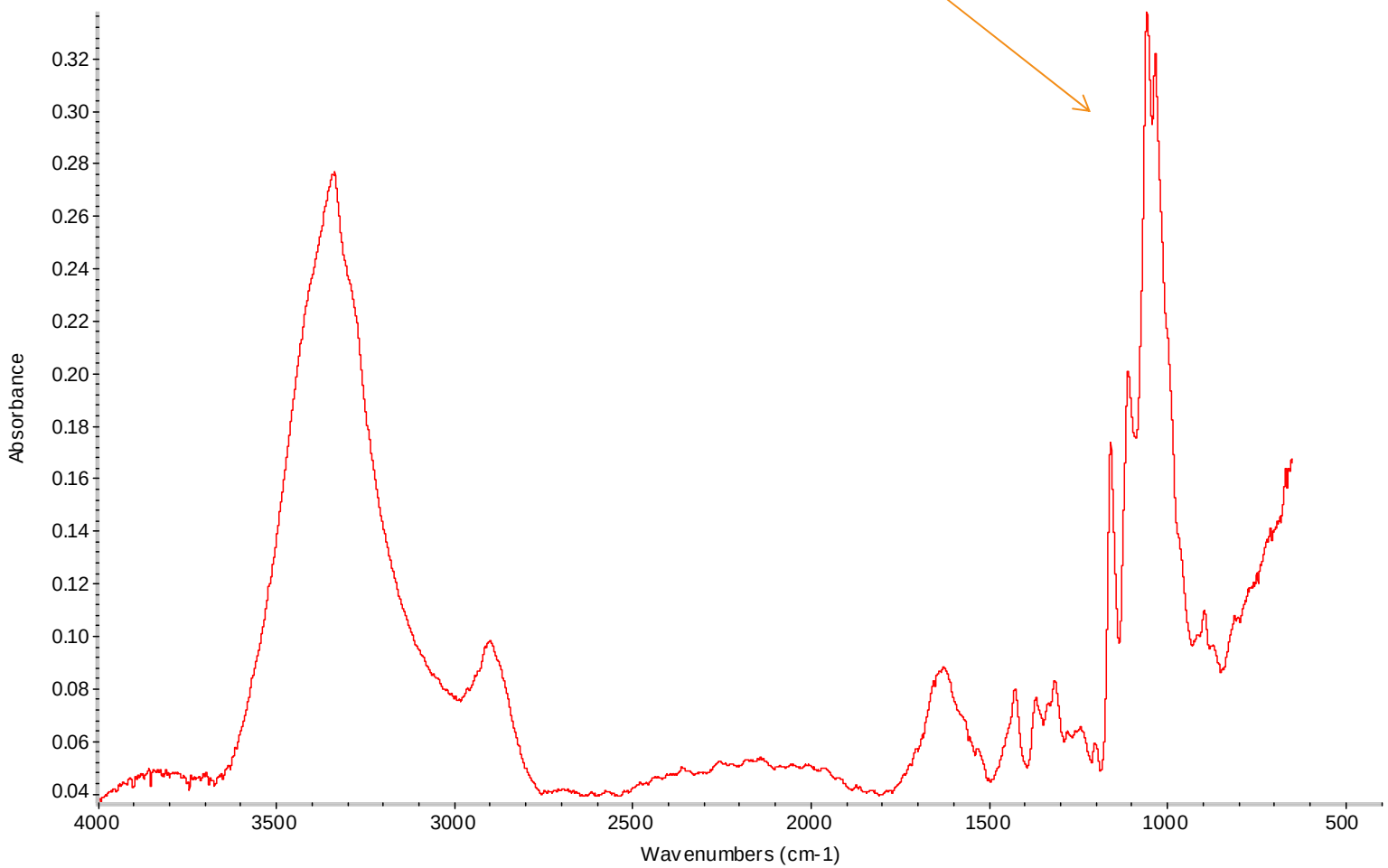
WIPP R15C5 Sample #2 Extract by FTIR (LIMS 300313811)

- (Organics - Solid in Extract): A few particles of a copolymer or mixture of ester urethane and amide)



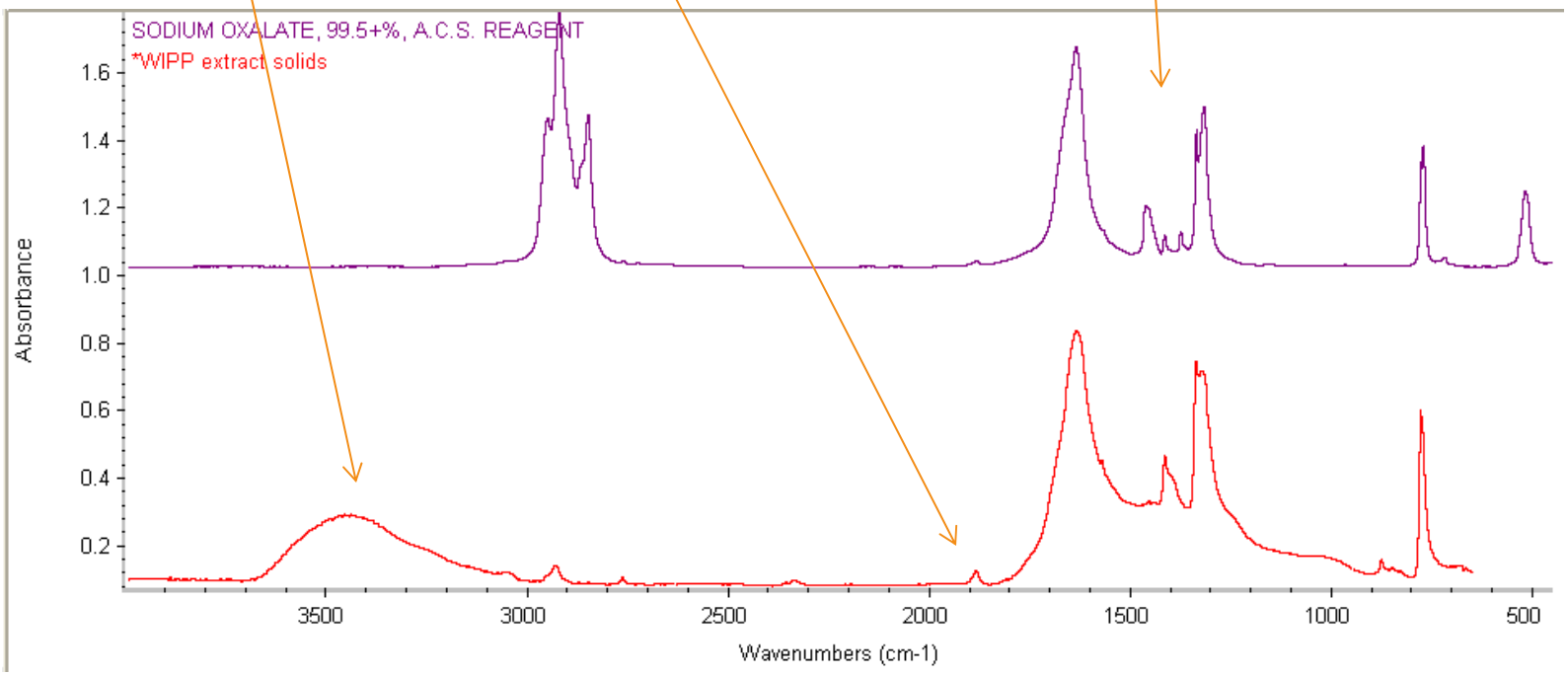
WIPP R15C5 Sample #2 Extract by FTIR (LIMS 300313811)

- (Solid from Extraction): A few particles of Unmodified Cellulose



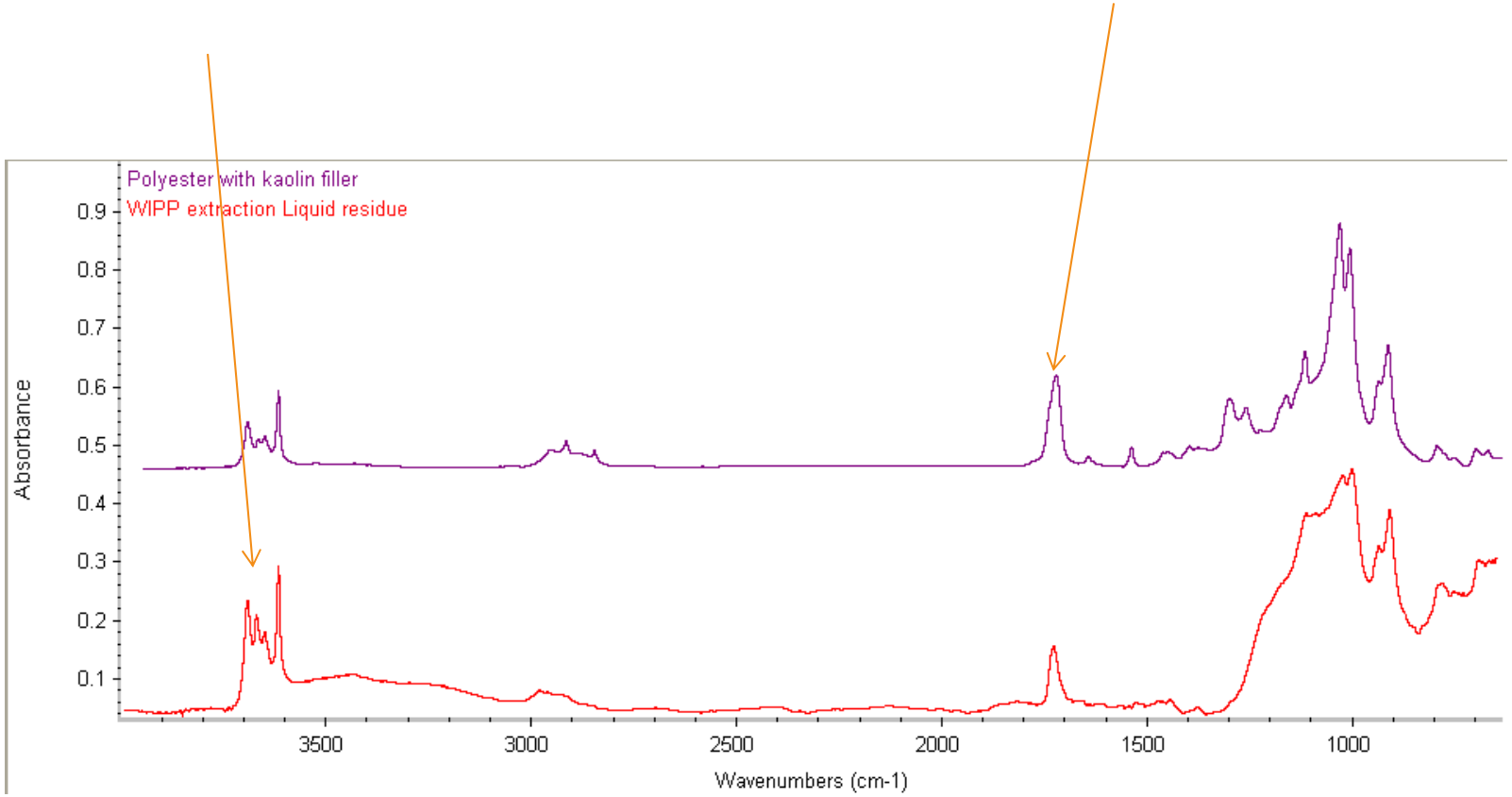
WIPP R15C5 Sample #2 Extract by FTIR (LIMS 300313811)

- (Solid in Extract – Inorganics): A few particles of Sodium Oxalate, Sodium Hydroxide and Sodium Carbonate



WIPP R15C5 Sample #2 Extract by FTIR (LIMS 300313811)

- (Solid in Extract – Inorganics) : A few particles of Polyester containing kaolin filler



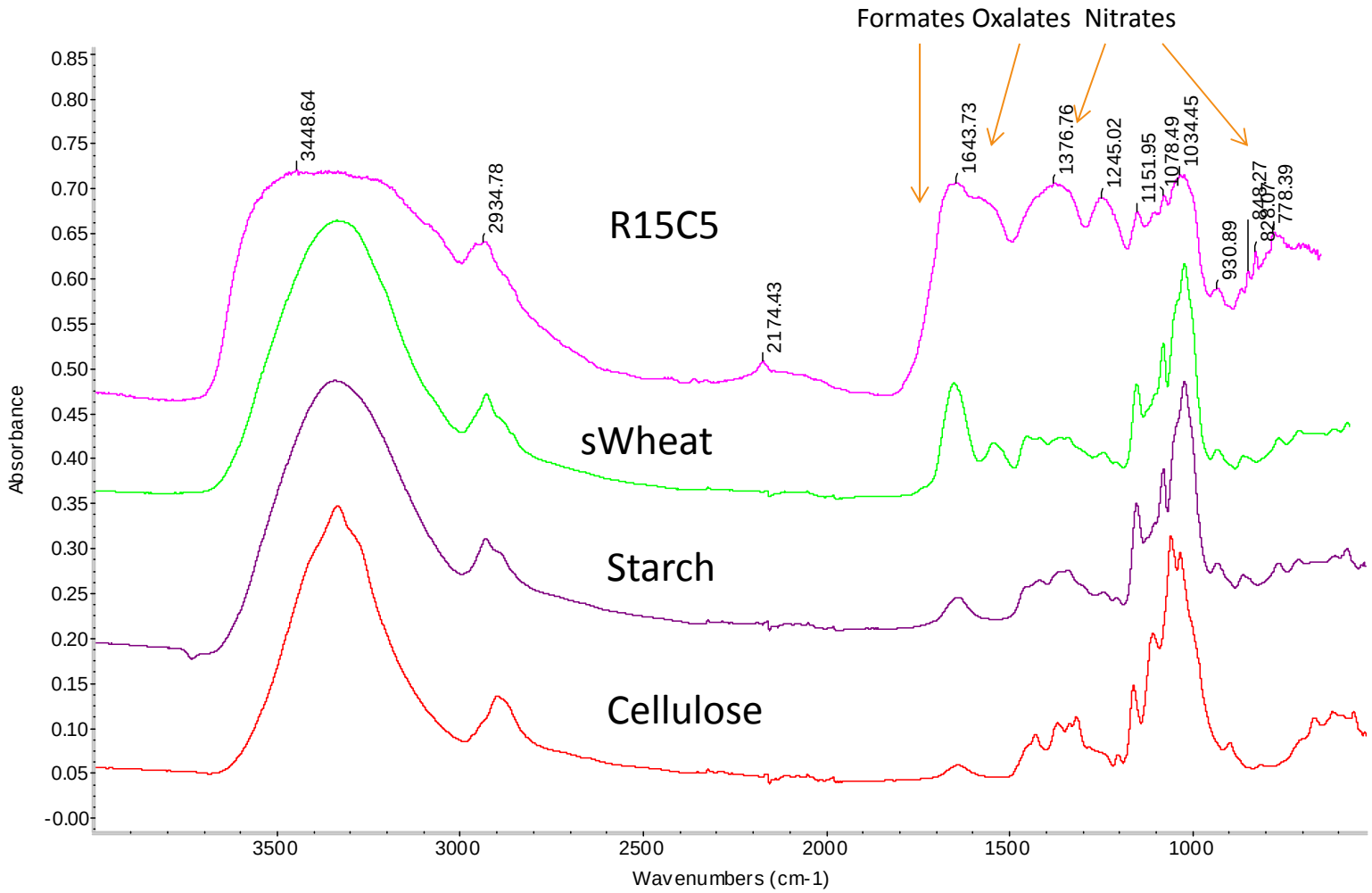
WIPP R15C5 Sample #2 Solid by FTIR (LIMS 300313811)

- Solid Material Preparation
  - As received granular material was contained (sandwiched) between two KBr discs and placed on top a FTIR microscope stage

- Instrument Conditions
  - Sealed radioactive materials were placed in a Nicolet Continuum FTIR microscope connected to a Nicolet Nexus Spectrometer. Each spectrum is the co-addition of 128 scans at resolution 4 cm^{-1} and apodized with a box car.

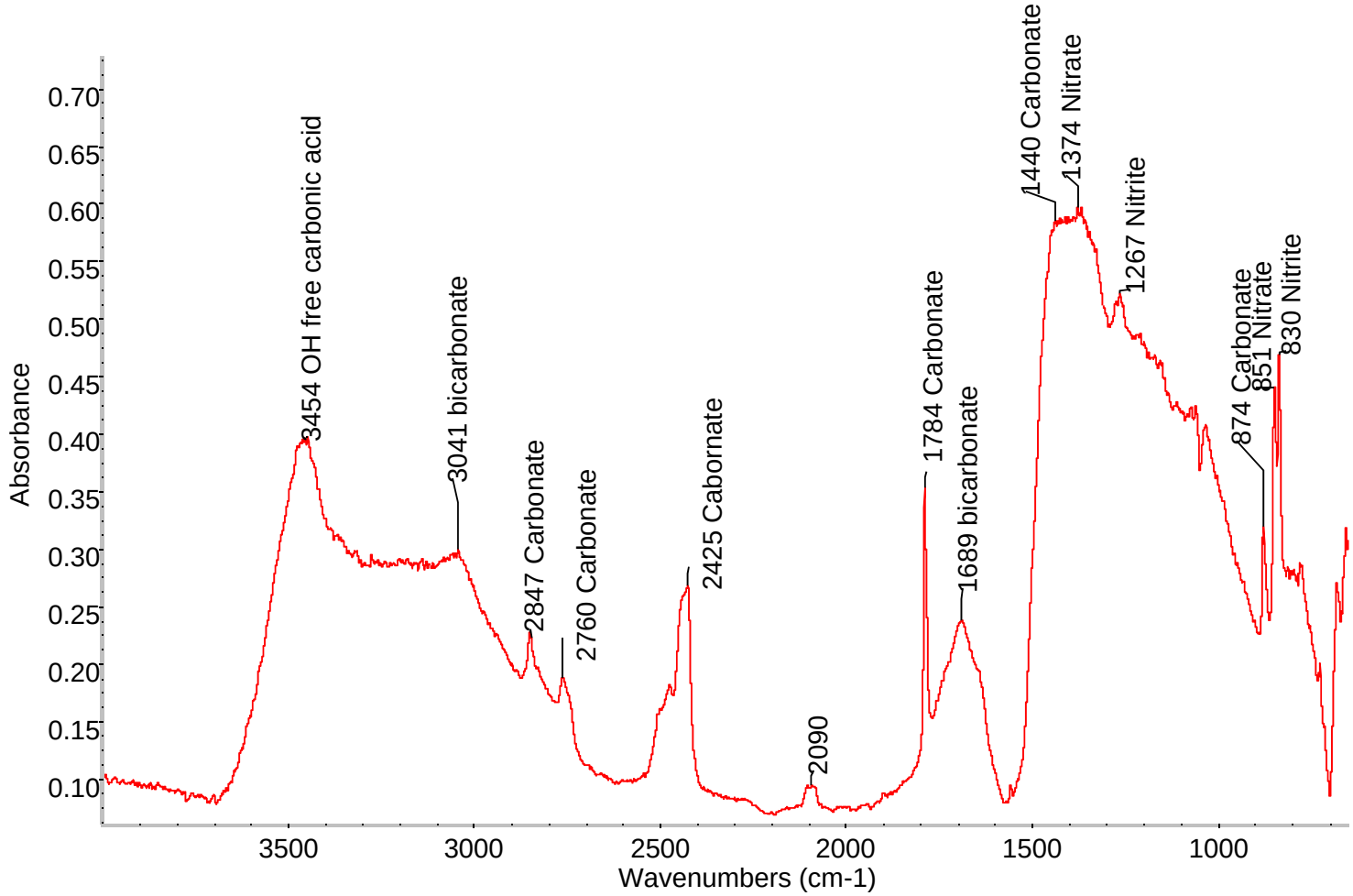
WIPP R15C5 Sample #2 Solid by FTIR (LIMS 300313811)

- (Organics): The hydrocarbon in R15C5 appears to be Starch-like sWheat



WIPP R15C5 Sample #2 Solid by FTIR (LIMS 300313811)

- (Inorganics): Sodium Carbonate (and bicarbonate), Sodium Nitrate, and Sodium Nitrite were also found



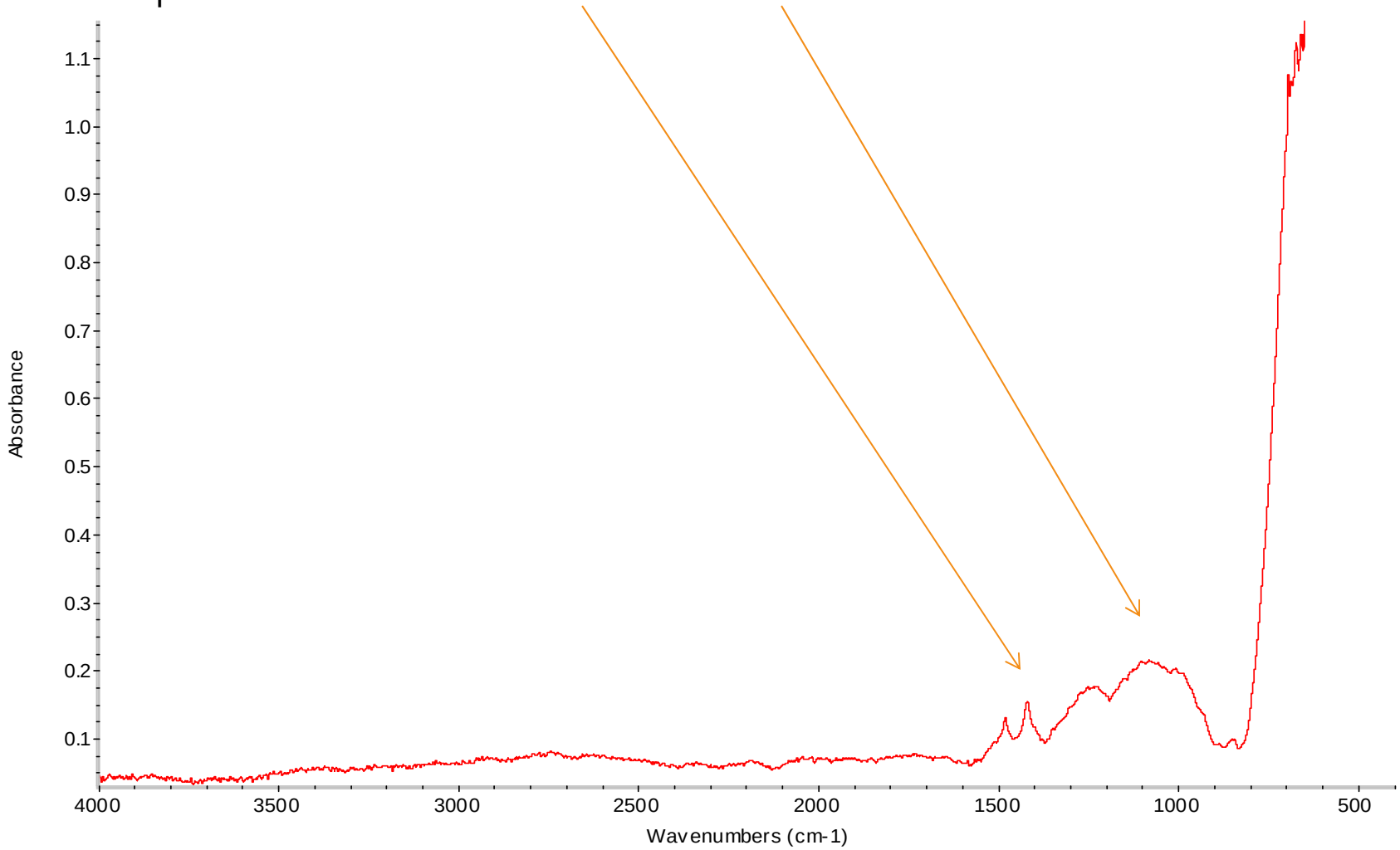
WIPP Super Sack MgO Surface Samples #6 and #7 by FTIR (LIMS 300313916/917)

- Solid Material Preparation
  - As received granular material was contained (sandwiched) between two KBr discs and placed on top a FTIR microscope stage

- Instrument Conditions
  - Sealed radioactive materials were placed in a Nicolet Continuum FTIR microscope connected to a Nicolet Nexus Spectrometer. Each spectrum is the co-addition of 128 scans at resolution 4 cm^{-1} and apodized with a box car.

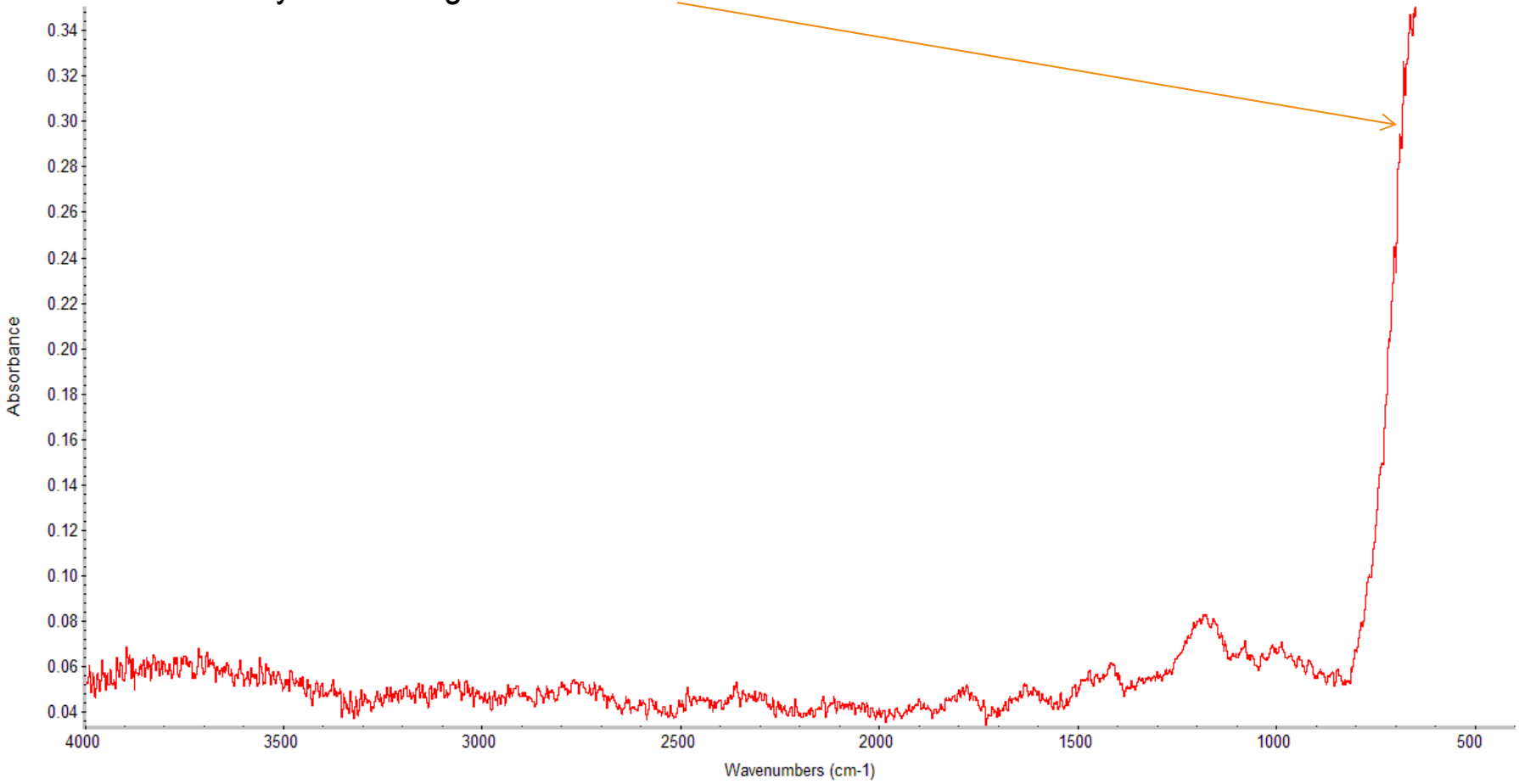
WIPP Super Sack MgO Surface Sample #6 by FTIR (LIMS 300313916)

- This sample shows surface carbonates and silicates



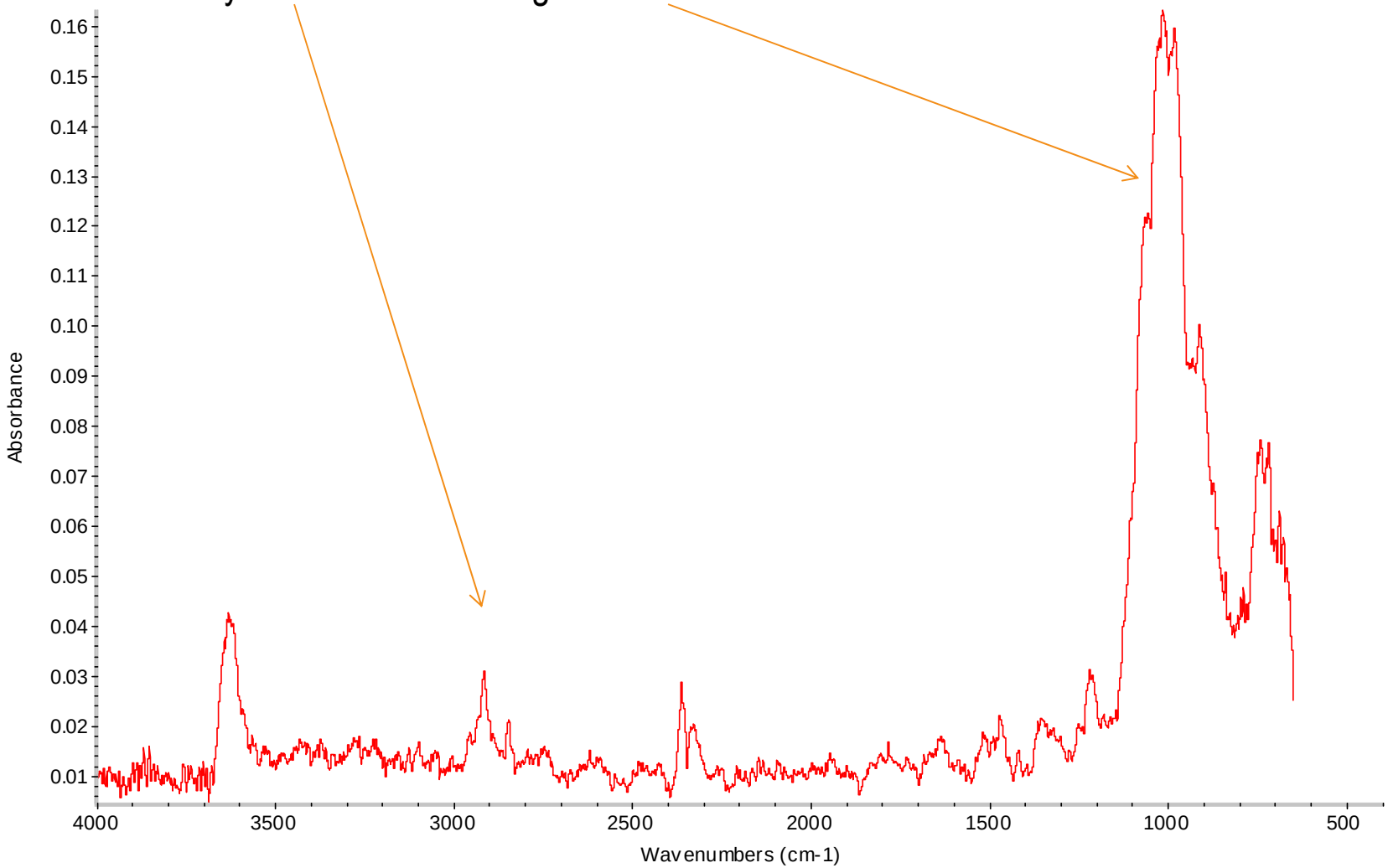
WIPP Super Sack MgO Sub Surface (6-inch) Sample #7 by FTIR (LIMS 300313917)

- Relatively Clean Magnesium Oxide



WIPP Super Sack MgO Sub Surface (6-inch) Sample #7 by FTIR (LIMS 300313917)

- Hydrocarbon oil on magnesium silicate



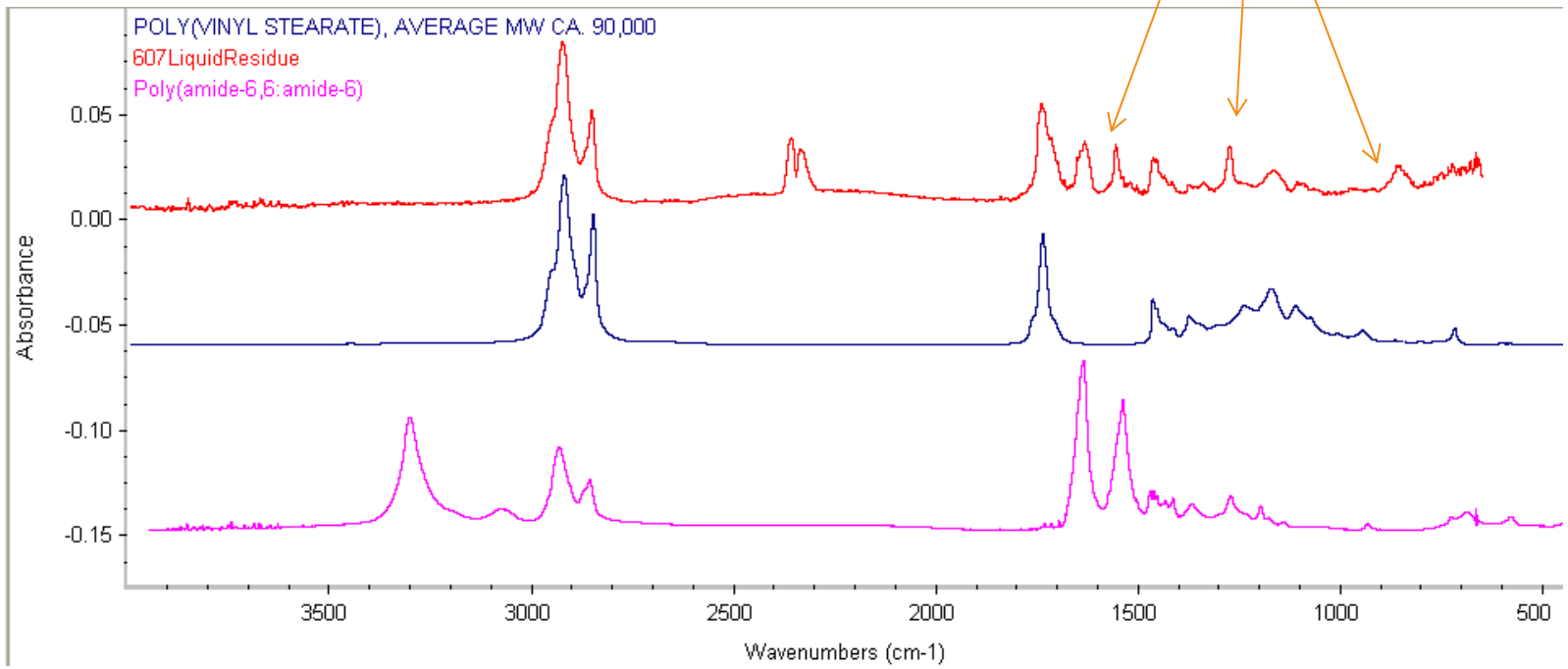
LANL Parent Drum Debris Sample by FTIR (LIMS 300313607)

- Material Preparation by Extraction Method
  - Approximately 0.1 g of as received granular material was placed in a Teflon capped glass containing 5 mL of Dichloromethane. The glass vial was spun end over end every 10 seconds for 48 hours. After which the solution was decanted and collected, leaving behind the granular material. The decanted solution was allowed to dry on a KBr disc in a desiccator. The KBr was then placed on a microscope stage.

- Instrument Conditions
  - Sealed radioactive materials were placed in a Nicolet Continuum FTIR microscope connected to a Nicolet Nexus Spectrometer. Each spectrum is the co-addition of 128 scans at resolution 4 cm^{-1} and apodized with a box car function.

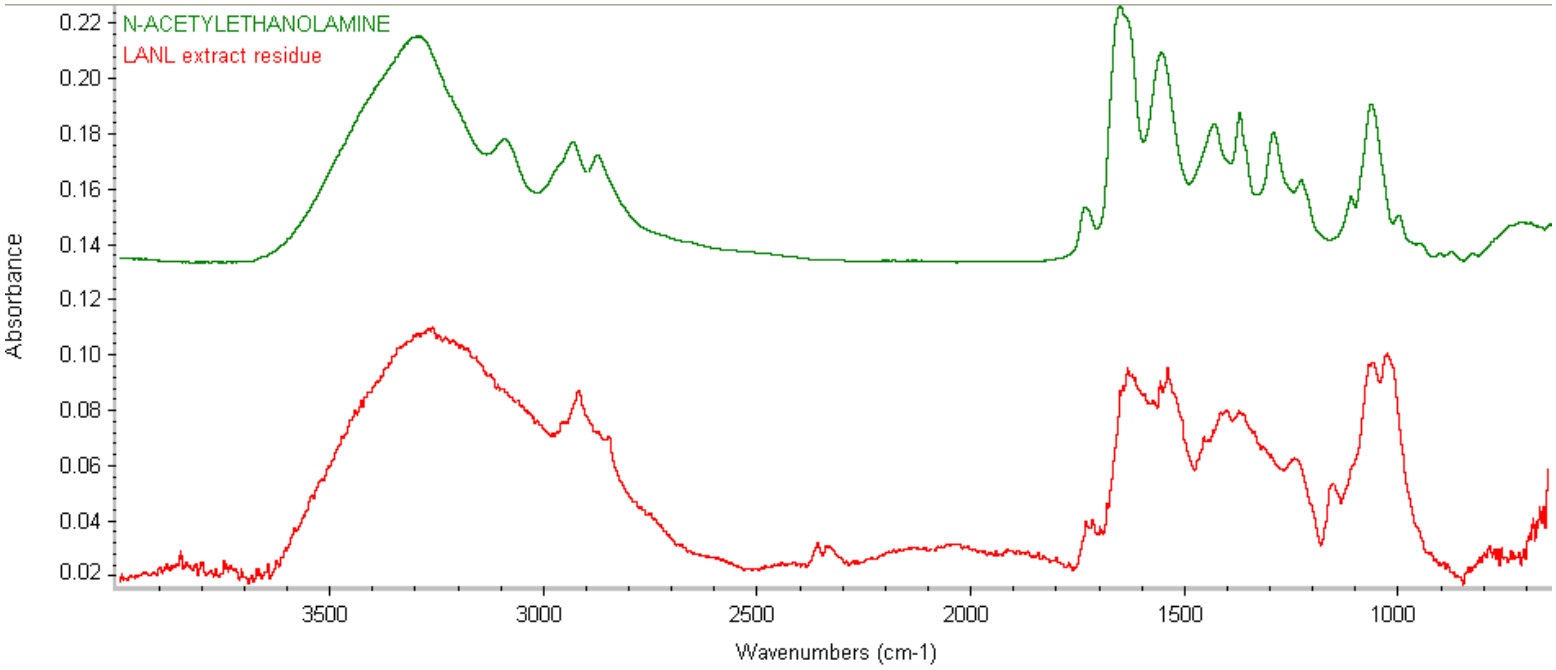
LANL Parent Drum Debris Sample by FTIR (LIMS 300313607)

- Organics Residue from Extraction : Some residues were found to be oxidized Ester oil (may contain nitro groups. Also Found evidence some ethanolamines (acetyl type) and evidence of modified starch was also found. Also shown a library spectrum that resembles the residue but that does not explain entirely the observed sample spectrum.



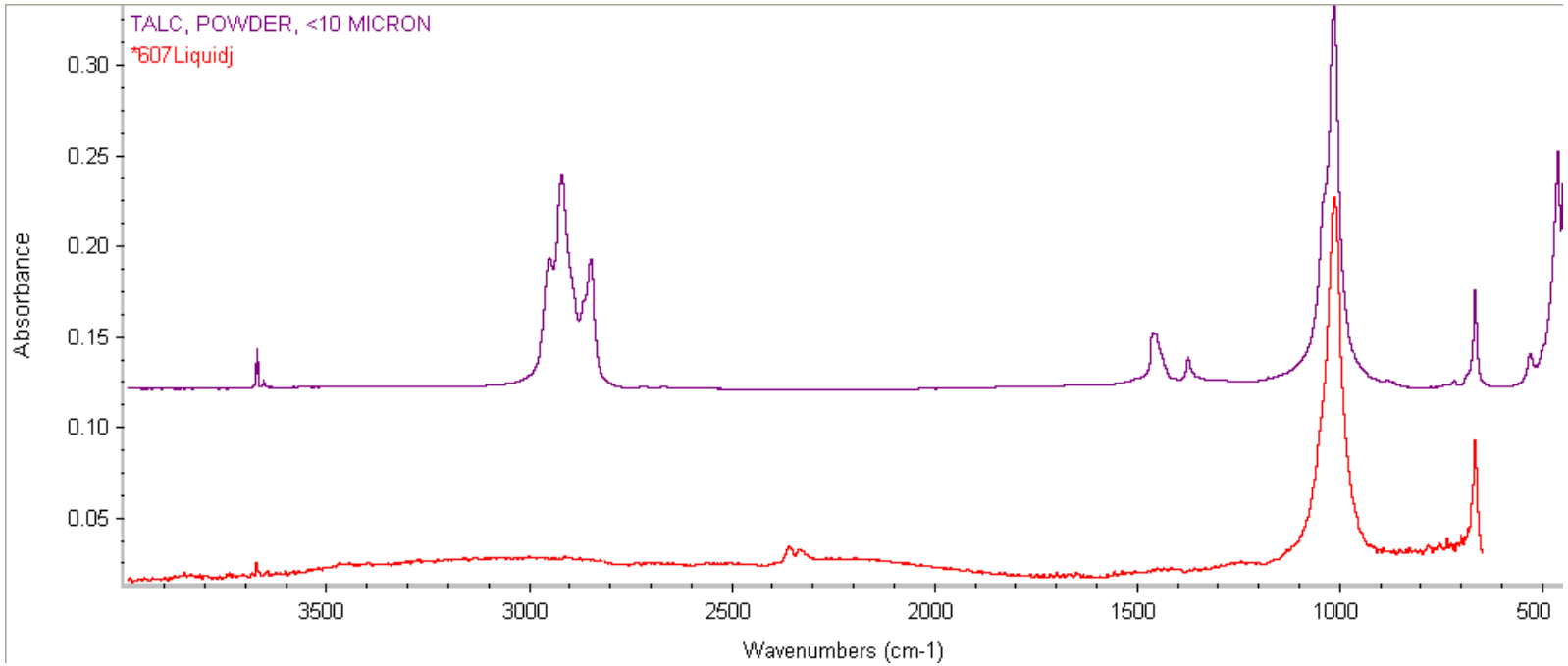
LANL Parent Drum Debris Sample by FTIR (LIMS 300313607)

- Organics - Solid from Extraction: Some residues were found to contain Amine and Secondary Amide as well as modified Cellulose



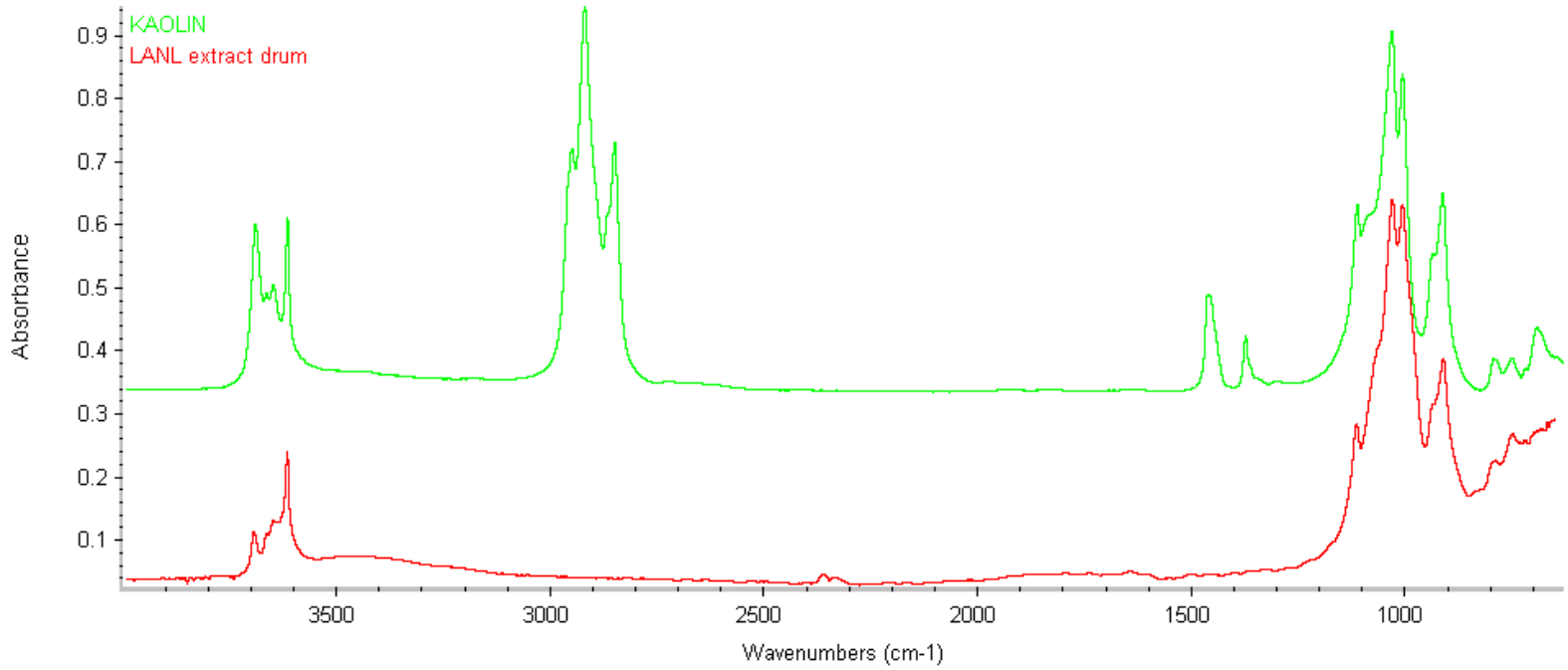
LANL Parent Drum Debris Sample by FTIR (LIMS 300313607)

- Solids from Extraction: Inorganics – Talc [$Mg_2(SiO_3)$]
- Insoluble solid material (suspension) carried by the dichloromethane solution. The standard library peak contains fingerprint from the support baseline.



LANL Parent Drum Debris Sample by FTIR (LIMS 300313607)

- Solid from Extraction: Inorganics – Kaolin [$Al_2Si_2O_5(OH)_4$].
- Part of the solid suspension in the leaching solution. The library spectrum contains peaks from a baseline used to support the standard.



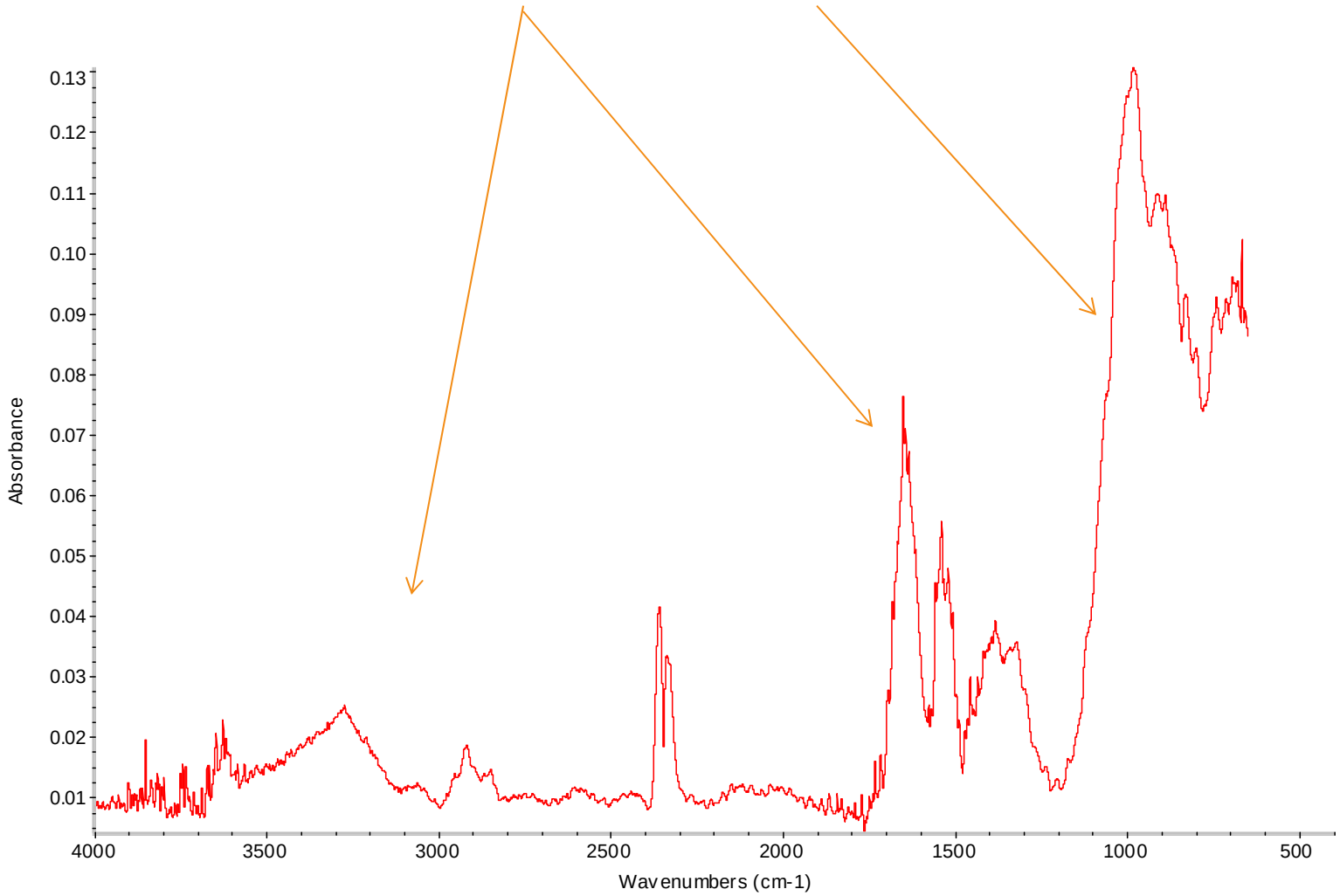
LANL Parent Drum Glass Swipe Sample by FTIR (LIMS 300313712)

- Solid Material Preparation
  - As received granular material was contained (sandwiched) between two KBr discs and placed on top a FTIR microscope stage

- Instrument Conditions
  - Sealed radioactive materials were placed in a Nicolet Continuum FTIR microscope connected to a Nicolet Nexus Spectrometer. Each spectrum is the co-addition of 128 scans at resolution 4 cm^{-1} and apodized with a box car.

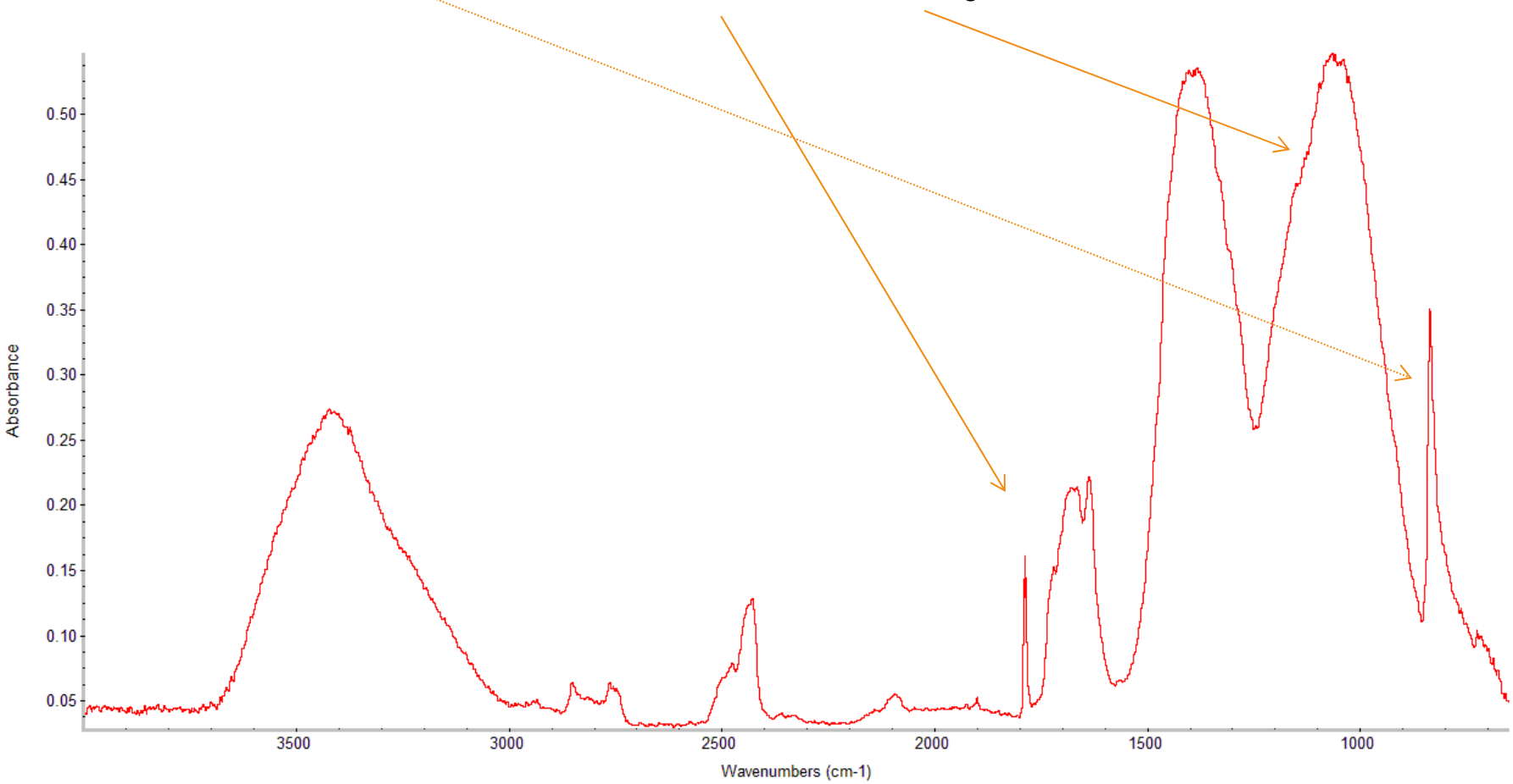
LANL Parent Drum Glass Swipe Sample by FTIR (LIMS 300313712)

- Solid Material: Secondary Amide on Borosilicate glass



LANL Parent Drum Glass Swipe Sample by FTIR (LIMS 300313712)

- Solid Material: Nitrates and Carbonates on Borosilicate glass



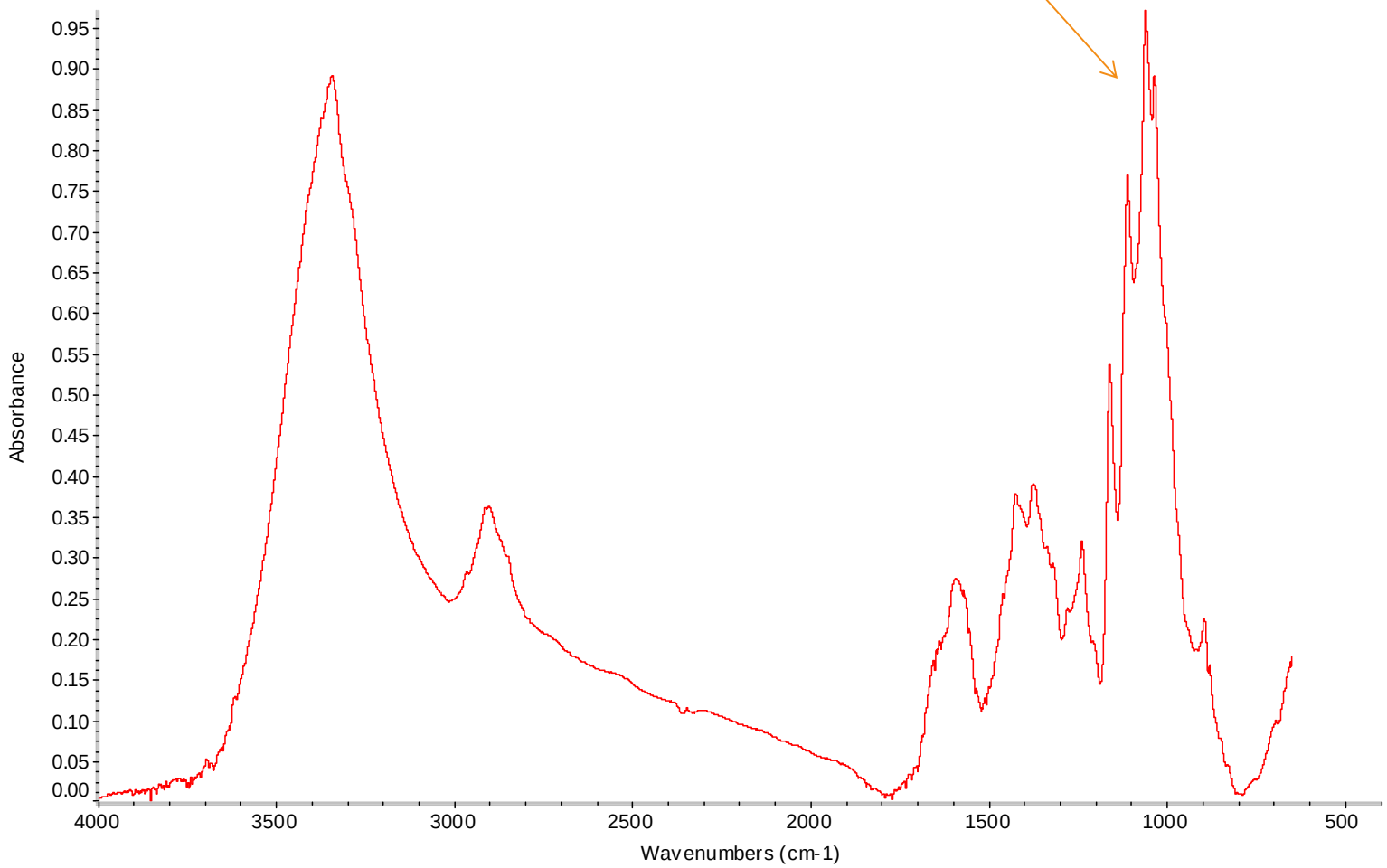
WIPP CAM Filter Cartridge Particulate Extracts by FTIR (LIMS 300311056-59)

- Material Preparation by Extraction Method
  - Approximately 0.1 g of as received granular material was placed in a Teflon capped glass containing 5 mL of Dichloromethane. The glass vial was spun end over end every 10 seconds for 48 hours. After which the solution was decanted and collected, leaving behind the granular material. The decanted solution was allowed to dry on a KBr disc in a desiccator. The KBr was then placed on a microscope stage.

- Instrument Conditions
  - Sealed radioactive materials were placed in a Nicolet Continuum FTIR microscope connected to a Nicolet Nexus Spectrometer. Each spectrum is the co-addition of 128 scans at resolution 4 cm^{-1} and apodized with a box car function.

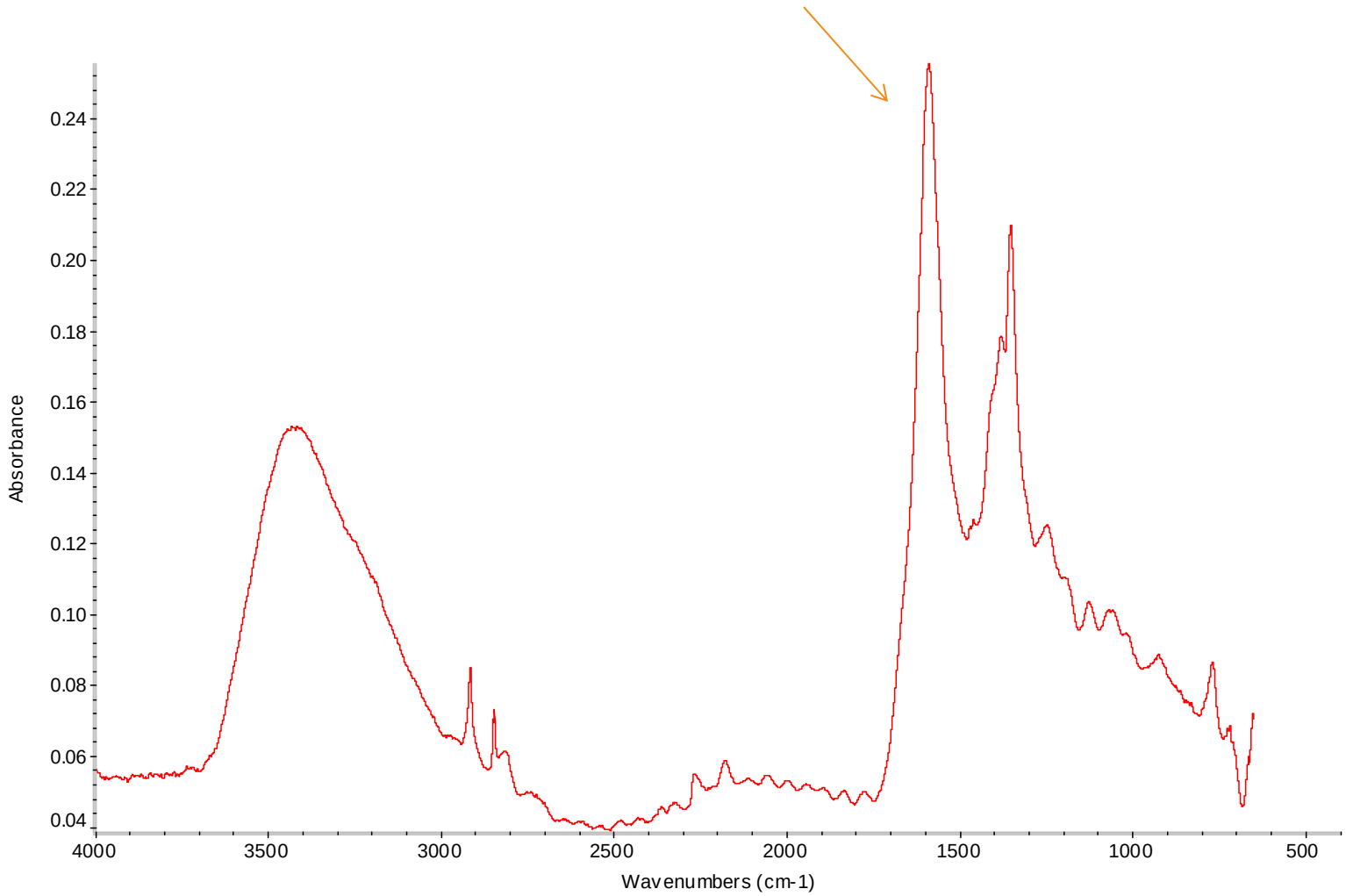
WIPP CAM Filter Cartridge Particulate Extracts by FTIR (LIMS 300311056-59)

- (Extract Solid): Some residues are unmodified Cellulose



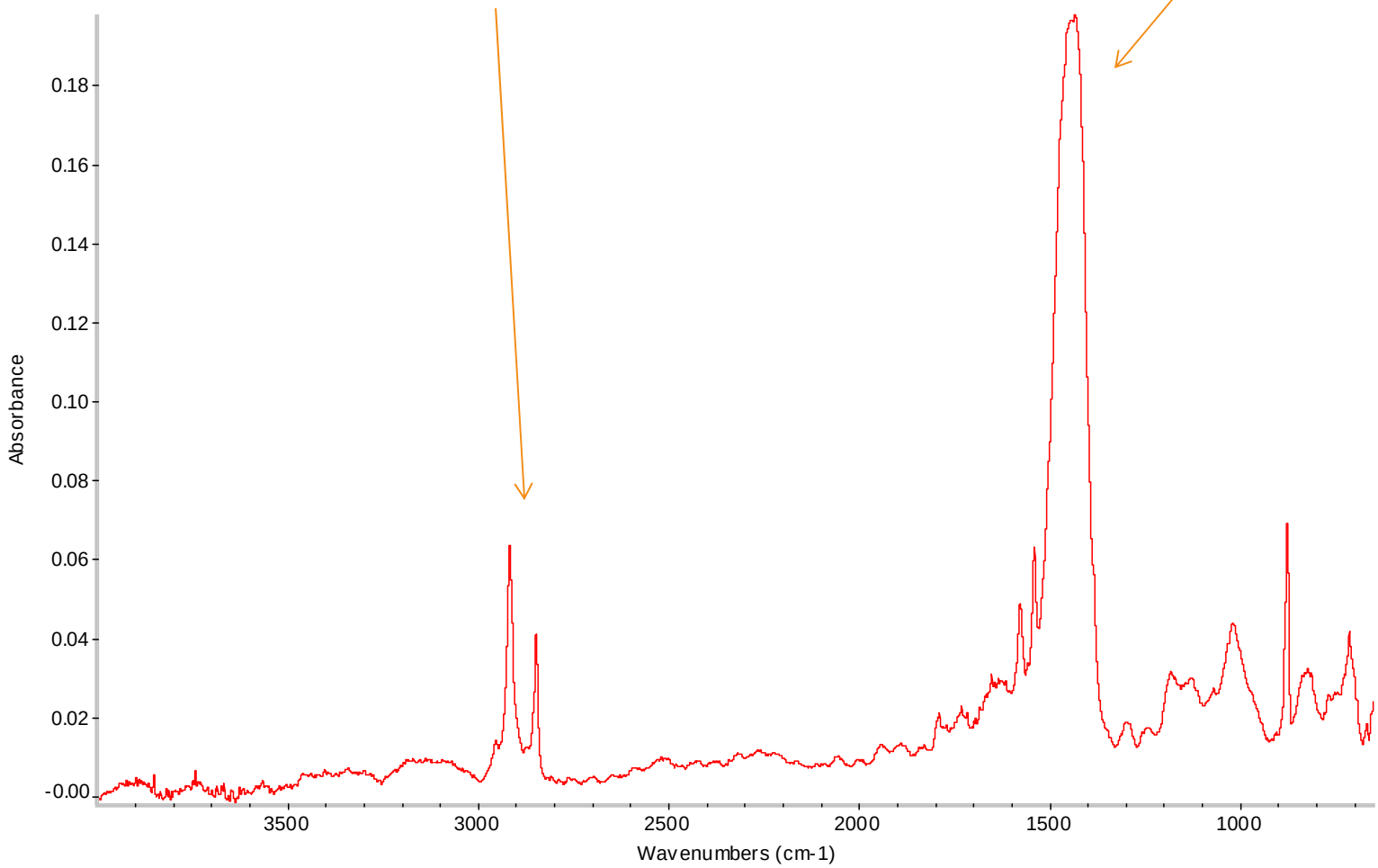
WIPP CAM Filter Cartridge Particulate Extracts by FTIR (LIMS 300311056-59)

- (Extract Solid): A few residues are Sodium Oxalate



WIPP CAM Filter Cartridge Particulate Extracts by FTIR (LIMS 300311056-59)

- (Extract Solid): A significant fraction of the residue are Sodium Nitrate mixed with hydrocarbon oil.



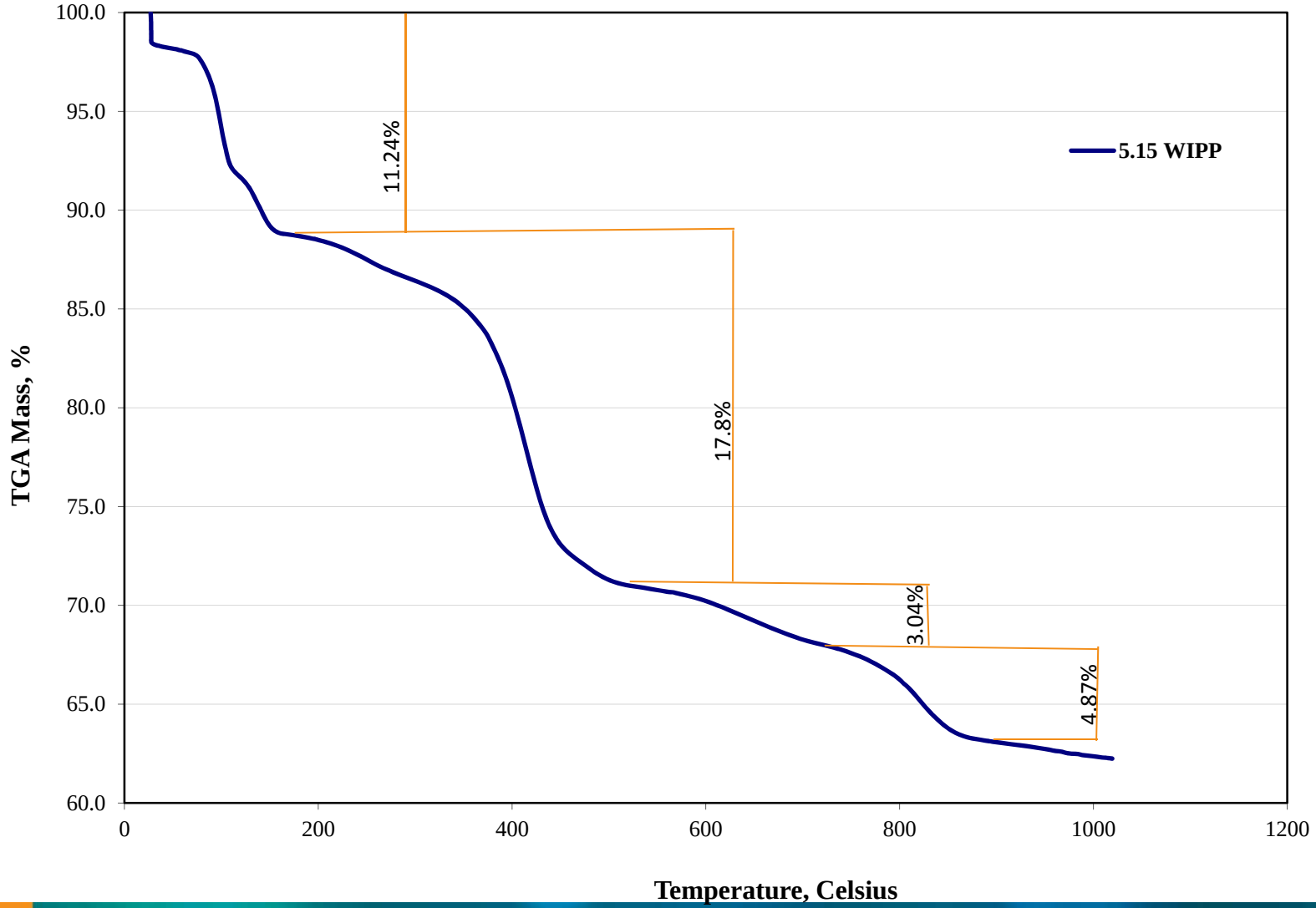
TGA-MS Data

WIPP R15C5 Sample #2 by TGA (LIMS 300313811)

- Solid Material Preparation
  - About 20 mg of sample was place in a zirconia cup and heated at 15 Celsius/min under a 15 mL/min Argon gas. The TGA is Netzsch and the MS is a quadrupole from Thermal Solutions.

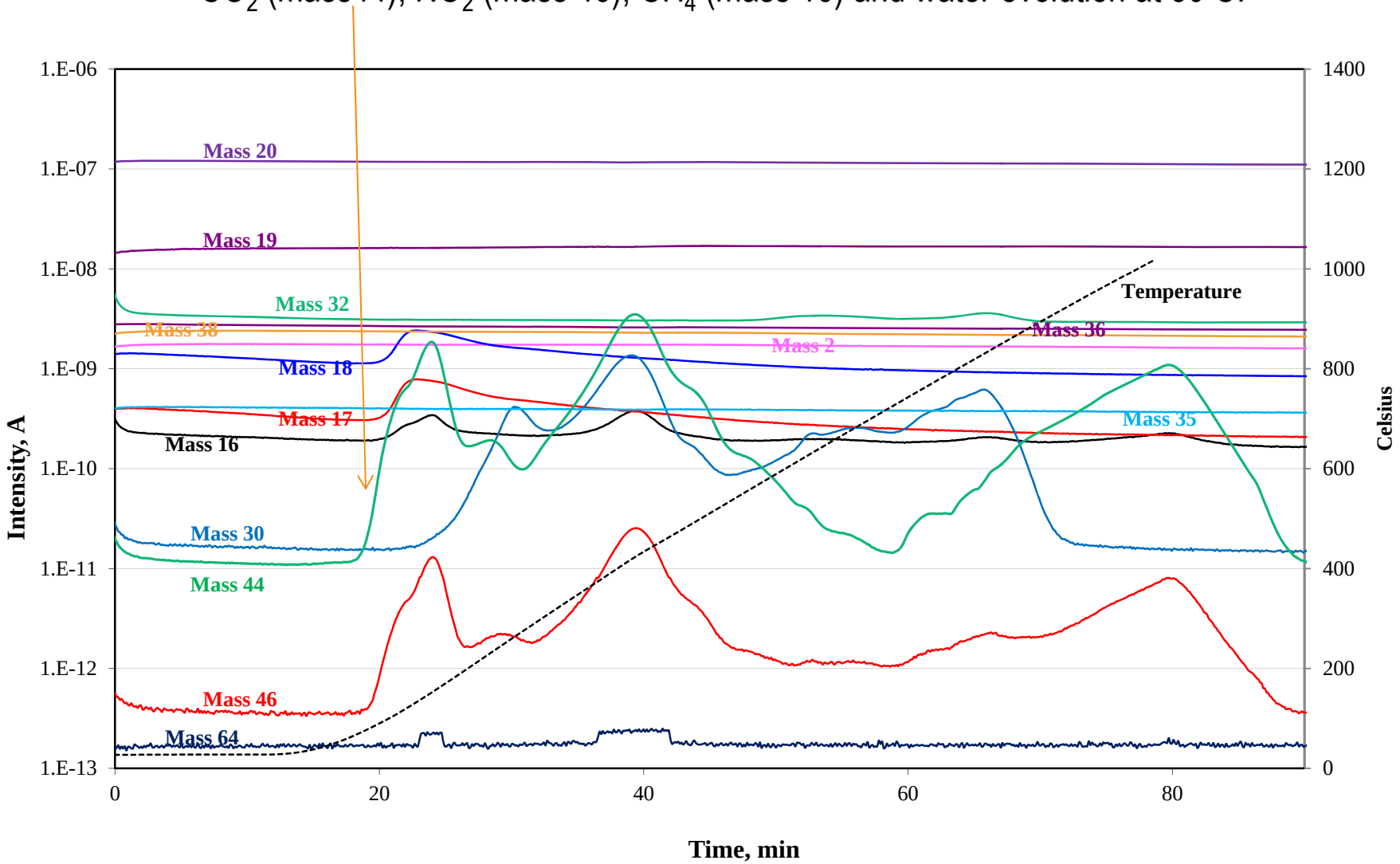
5.15 WIPP sample

- Weight losses reflect water, CO_2, NO_2, possibly NO,formaldehyde and other gases



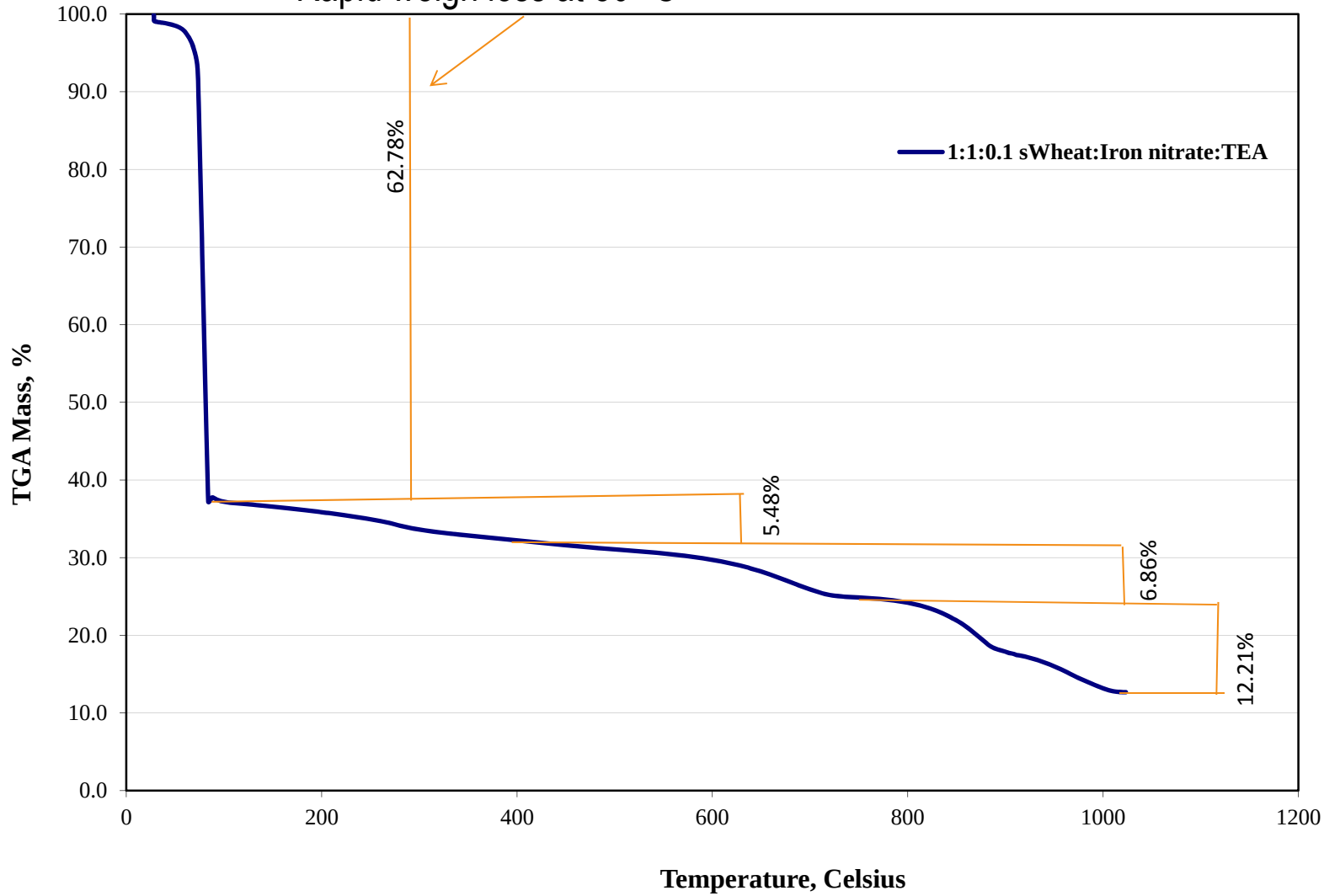
TG-MS spectrum of R15C5

- CO_2 (mass44), NO_2 (mass 46), CH_4 (mass 16) and water evolution at 80°C.



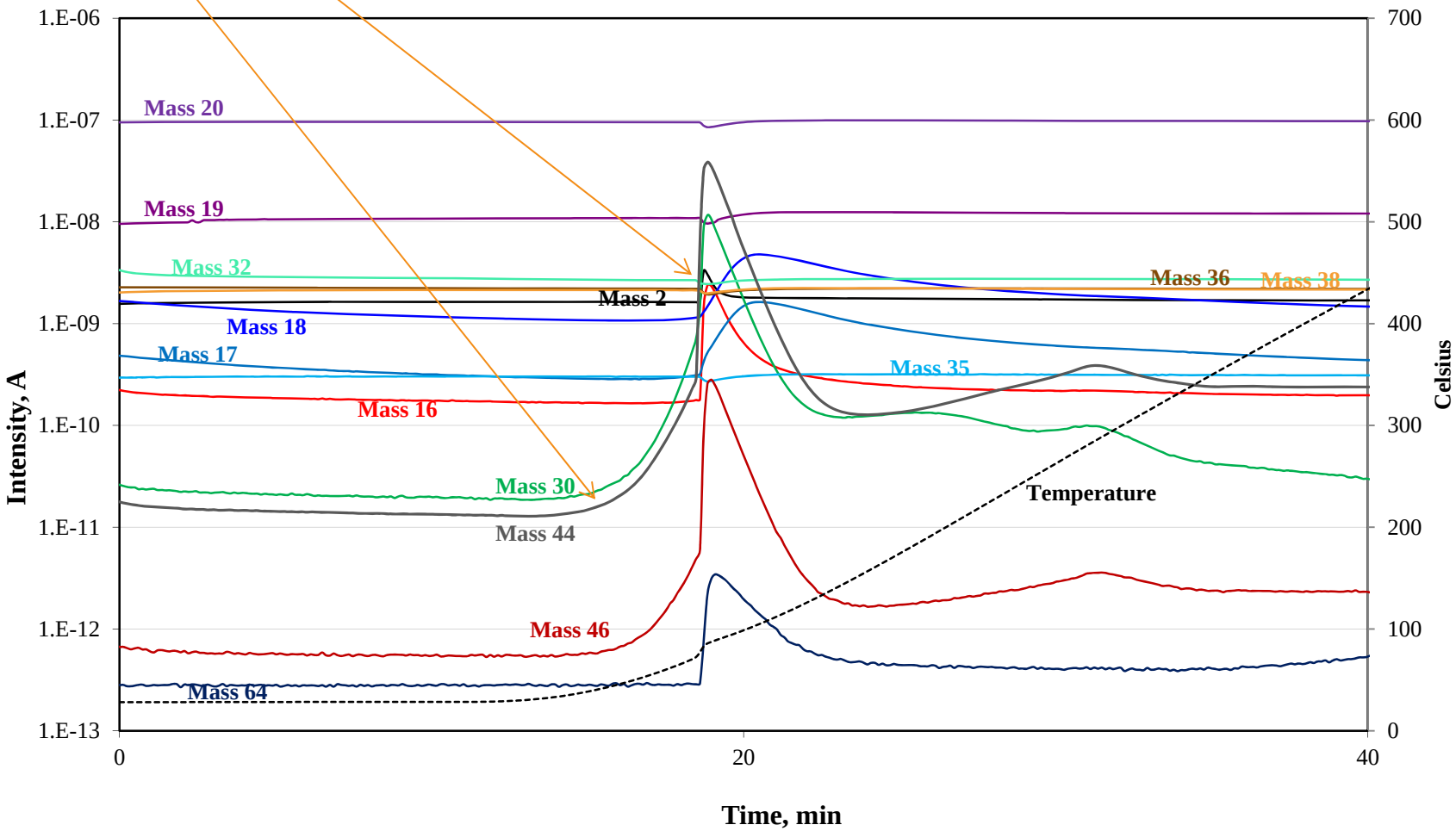
TGA of 1:1:0.1 Swheat : Iron nitrate: TEA

- Rapid weigh loss at 80 ◦C



Mass Spectrometry of Gases Released from 1:1:0.1 Swheat:Iron Nitrate:TEA

- Slow incubation releasing CO_2 and CH_2O (formaldehyde) or NO
- followed by rapid reaction releasing H_2, CH_2O or NO, NO_2, and CO_2 at 80°C.



Contributors

- Amy Ekechukwu
- Charles Coleman
- David Diprete
- David Missimer
- Fernando Fondeur
- John Young
- Henry Ajo
- Mark Jones
- Patrick O'Rourke
- Stephen Crump
- Thomas White

Attachment 2 - Sample ID and Chain of Custody Cross Reference

SAMPLE_ID	TRAVEL COPY	USER_SAMPLEID	COC #
300309393	65453	D13_020514	14-0014
300309392	65453	D12_020514	14-0014
300309391	65453	D11_020514	14-0014
300309390	65453	B13_021414	14-0014
300309389	65453	A33_021414	14-0014
300309388	65453	A23_021514_1510 2_17_14	14-0014
300309387	65453	A23_021514_0840 2_17_14	14-0014
300309386	65453	ADS GENERATED BLANK	14-0014
300309390	65453	B13_021414	14-0014
300309389	65453	A33_021414	14-0014
300309388	65453	A23_021514_1510 2_17_14	14-0014
300309387	65453	A23_021514_0840 2_17_14	14-0014
300309386	65453	ADS GENERATED BLANK	14-0014
300309391	65453	D11_020514	14-0014
300309392	65453	D12_020514	14-0014
300309393	65453	D13_020514	14-0014
300311047	65772	FILTER _9	14-0160
300311044	65772	FILTER _6	14-0160
300311042	65772	FILTER _4	14-0160
300311053	65772	FILTER _15	14-0160
300311052	65772	FILTER _14	14-0160
300311051	65772	FILTER _13	14-0160
300311050	65772	FILTER _12	14-0160
300311049	65772	FILTER _11	14-0160
300311048	65772	FILTER _10	14-0160
300311046	65772	FILTER _8	14-0160
300311045	65772	FILTER _7	14-0160
300311043	65772	FILTER _5	14-0160
300311041	65772	FILTER _3	14-0160
300311040	65772	FILTER _2	14-0160
300311039	65772	FILTER _1	14-0160
300311076	65774	WIPE_8, Masselin	14-0173
300311075	65774	WIPE_7, Masselin	14-0173
300311074	65774	WIPE_6, RPD Smea	14-0173
300311073	65774	WIPE_5, RPD Smea	14-0173
300311072	65774	WIPE_4, RPD Smea	14-0173
300311071	65774	WIPE_3, RPD Smea	14-0173
300311070	65774	WIPE_2, RPD Smea	14-0173
300311069	65774	WIPE_1, RPD Smear	14-0173
300311089	65778	WIPE #2 (300311070)	14-0173
300311088	65778	WIPE #1 (300311069)	14-0173
300311087	65778	ADS GENERATED BLANK	14-0173
300311132	65784	300311038_C(311108)	14-0175
300311131	65784	ADS GENERATED BLANK	14-0175
300311194	65793	300311187_C(#11_311049)	14-0175

Attachment 2 - Sample ID and Chain of Custody Cross Reference

300311193	65793	300311169_C(#7_311045)	14-0175
300311192	65793	300311163_C(#2_311040)	14-0175
300311191	65793	ADS GENERATED BLANK	14-0175
300311440	65834	#4_ROOM 1 EXHAUST DRIFT	14-0175
300311439	65834	#3_SLIP SHEET WASTE FACE	14-0175
300311438	65834	#2_PINK PAD WASTE FACE	14-0175
300311437	65834	#1_TABLE_CHAIR WASTE FACE	14-0175
300311518	65852	#4_CAMERA LENS	14-0175
300311517	65852	#3_TABLE_CHAIRS WASTE FACE	14-0175
300311516	65852	#2_SLIP SHEET	14-0175
300311515	65852	#1_PINK MAT WASTE FACE	14-0175
300313604	66248	CAM FILTER 9, SECTION PF	14-0160
300313603	66248	CAM FILTER 6, SECTION PF	14-0160
300313602	66248	CAM FILTER 4, SECTION PF	14-0160
300313601	66248	WIPP BLANK PF	14-0160
300311203	65795	BAG 5_PANEL 7 ROOM 6	14-0182
300311202	65795	BAG 4_WASTE FACE TABLE	14-0182
300311201	65795	BAG 3_PANEL 7 ROOM 1	14-0182
300311200	65795	BAG 2_WASTE FACE	14-0182
300311199	65795	BAG 1_WASTE FACE PINK PILLOW	14-0182
300311203	65795	BAG 5_PANEL 7 ROOM 6	14-0182
300311201	65795	BAG 3_PANEL 7 ROOM 1	14-0182
300312405	65975	R_14 C_2	14-0182
300312404	65975	R_14 C_6	14-0182
300312403	65975	R_14 C_4	14-0182
300312402	65975	R_16 C_4	14-0182
300312714	65979	MGO STD-XRDF	14-0182
300312527	65979	R_14 C_6 XPD	14-0182
300312526	65979	R_14 C_6 XPL	14-0182
300312525	65979	R_14 C_4 XPD	14-0182
300312524	65979	R_14 C_4 XPL	14-0182
300312523	65979	R_14 C_2 XPD	14-0182
300312522	65979	R_14 C_2 XPL	14-0182
300312714	65979	MGO STD-XRDF	14-0182
300312527	65979	R_14 C_6 XPD	14-0182
300312526	65979	R_14 C_6 XPL	14-0182
300312525	65979	R_14 C_4 XPD	14-0182
300312524	65979	R_14 C_4 XPL	14-0182
300312523	65979	R_14 C_2 XPD	14-0182
300312522	65979	R_14 C_2 XPL	14-0182
300312521	65979	R_14 C_6 SPD (4)	14-0182
300312520	65979	R_14 C_6 SPL (3)	14-0182
300312519	65979	R_14 C_4 SPD (2)	14-0182
300312518	65979	R_14 C_4 SPL (1)	14-0182
300312517	65979	R_14 C_2 SPD (6)	14-0182
300312516	65979	R_14 C_2 SPL (5)	14-0182

Attachment 2 - Sample ID and Chain of Custody Cross Reference

300312532	66045	R_14 C_6 1_F	14-0182
300312530	66045	R_14 C_4 1_F	14-0182
300312529	66045	R_14 C_2 2_F	14-0182
300312528	66045	R_14 C_2 1_F	14-0182
300313027	66045	Undeployed MgO VOA	NA
300313026	66045	R_14 C_6 VOA	14-0182
300313029	66045	Undeployed SVOA	NA
300313028	66045	R_14 C_6 SVOA	14-0182
300313031	66045	Undeployed MgO IC	NA
300313030	66045	R_14 C_6 IC	14-0182
300312834	66094	R_16 C_4 FTIR	14-0182
300312833	66094	MGO STD FTIR	14-0182
300312748	66094	R_16 C_4 XRDF	14-0182
300312747	66094	MGO STD XRDF	NA
300312746	66094	R_16 C_4 SEM	14-0182
300312745	66094	MGO STD SEM	NA
300312966	66125	MgO Off Color Particle #2	14-0182
300312941	66125	MGO REFERENCE MATERIAL #2	NA
300312940	66125	MGO REFERENCE MATERIAL #1	NA
300312965	66125	MgO Off Color Particle #1	14-0182
300312971	66101	R14 C2 A	14-0182
300312970	66101	R14 C6 A_4	14-0182
300312969	66101	R14 C6 A_3	14-0182
300312968	66101	R14 C6 A_2	14-0182
300312967	66101	R14 C6 A_1	14-0182
300313600	66247	CAM FILTER 9, SECTION MA	14-0160
300313599	66247	CAM FILTER 6, SECTION MA	14-0160
300313598	66247	CAM FILTER 4, SECTION MA	14-0160
300313597	66247	WIPP BLANK MA	14-0182
300313720	66282	69120_F_FTIR	14-0182
300313716	66282	69120_E_FTIR	14-0182
300313712	66282	69120_D_FTIR	14-0182
300313721	66282	69120_F_GCMS	14-0182
300313717	66282	69120_E_GCMS	14-0182
300313713	66282	69120_D_GCMS	14-0182
300313732	66284	ADS GENERATED BLANK	14-0182
300313734	66284	69120_H_PF	14-0182
300313733	66284	69120_A_PF	14-0182
300313735	66285	ADS GENERATED BLANK	14-0182
300313736	66285	69120_B_MA	14-0182
300313741	66287	69120_B_PF	14-0182
300313740	66287	ADS GENERATED BLANK	14-0182
300313743	66288	69120_B_IC	14-0182
300313742	66288	ADS GENERATED BLANK	14-0182
300313744	66288	69120_B_TITR	14-0182
300313742	66288	ADS GENERATED BLANK	14-0182

Attachment 2 - Sample ID and Chain of Custody Cross Reference

300313744	66288	69120_B_TITR	14-0182
300313744	66288	69120_B_TITR	14-0182
300313743	66288	69120_B_IC	14-0182
300313814	66295	SAMPLE #7	14-0182
300313813	66295	SAMPLE #6	14-0182
300313812	66295	SAMPLE #3	14-0182
300313811	66295	SAMPLE #2	14-0182
300313810	66295	SAMPLE #1	14-0182
300313909	66301	ADS GENERATED BLANK	14-0182
300313921	66301	SAMPLE_7_XRD	14-0182
300313920	66301	SAMPLE_6_XRD	14-0182
300313919	66301	SAMPLE_2_SEM	14-0182
300313918	66301	SAMPLE_1_SEM	14-0182
300313917	66301	SAMPLE_7_FTIR	14-0182
300313916	66301	SAMPLE_6_FTIR	14-0182
300313914	66301	SAMPLE_1_FTIR	14-0182
300313912	66301	SAMPLE_1_GCMS	14-0182
300313911	66301	SAMPLE_2_MA	14-0182
300313910	66301	SAMPLE_1_MA	14-0182
300313922	66302	ADS GENERATED BLANK	14-0182
300313922	66302	ADS GENERATED BLANK	14-0182
300313924	66302	SAMPLE_2_PF	14-0182
300314333	66417	UNUSED SWHEAT - PF	NA
300314332	66417	ADS GENERATED BLANK	14-0182
300314333	66417	UNUSED SWHEAT - PF	NA
300314335	66418	UNUSED SWHEAT _MA	NA
300314334	66418	ADS GENERATED BLANK	NA
300314374	66424	ADS GENERATED BLANK	NA
300314375	66424	WIPP #3 (SPARE 15_5)_MA	14-0182
300314377	66425	WIPP #3 (SPARE 15_5)_PF	14-0182
300314376	66425	ADS GENERATED BLANK	14-0182
300314453	66439	ADS GENERATED BLANK	14-0182
300314454	66439	WIPP SAMPLE #2 (R15_C5)	14-0182
300314454	66439	WIPP SAMPLE #2 (R15_C5)	14-0182
300314453	66439	ADS GENERATED BLANK	14-0182
300314454	66439	WIPP SAMPLE #2 (R15_C5)	14-0182
300314453	66439	ADS GENERATED BLANK	14-0182
300314962	66523	IC SWHEAT LEACH #1	NA
300314964	66523	WET CHEM SWHEAT LEACH #2	NA
300314963	66523	WET CHEM BLANK #2	NA
300309400	65454	D13_020514	14-0182
300309399	65454	D12_020514	14-0182
300309398	65454	D11_020514	14-0182
300309397	65454	B13_021414	14-0182
300309396	65454	A33_021414	14-0182
300309395	65454	A23_021514_1510	14-0182

Attachment 2 - Sample ID and Chain of Custody Cross Reference

300309394	65454	A23_021514_0840	14-0182
300311110	65771	300311038-E	14-0182
300311055	65773	FILTER _ 17	14-0160
300311054	65773	FILTER _ 16	14-0160
300311092	65779	WIPE #5 (300311073)	14-0175
300311091	65779	WIPE #3 (300311071)	14-0175
300311090	65779	ADS GENERATED BLANK	14-0175
300311130	65783	300311038_D(311109)	14-0175
300311129	65783	ADS GENERATED BLANK	14-0175
300311171	65791	CAM FILTER#7 (311045)_E	14-0160
300311168	65791	CAM FILTER#7 (311045)_B	14-0160
300311165	65791	CAM FILTER#2 (311040)_E	14-0160
300311162	65791	CAM FILTER#2 (311040)_B	14-0160
300311198	65794	300311188_D(#11_311049)	14-0160
300311197	65794	300311170_D(#7_311045)	14-0160
300311196	65794	300311164_D(#2_311040)	14-0160
300311195	65794	ADS GENERATED BLANK	14-0160
300313607	66250	ARCHIVE B, SOLID DEBRIS	14-0182
300313605	66250	ARCHIVE A, IAEA SWIPE	14-0182
300313610	66250	ARCHIVE D, GLASS SWIPE	14-0182
300313725	66283	ADS GENERATED BLANK	14-0182
300313728	66283	69120_A_ARC2	14-0182
300313727	66283	69120_A_ARC1	14-0182
300313726	66283	69120_A_MA	14-0182
300313729	66283	69120_H_MA	14-0182