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Date: MAR 2 1 2016
Symbol: ADESH-16-043
LA-UR: 16-21587
Locates Action No.: Not Applicable

Mr. John E. Kieling, Chief
Hazardous Waste Bureau
New Mexico Environment Department
2905 Rodeo Park Drive East, Building 1
Santa Fe, NM 87505-6303

Dear Mr. Kieling:

Subject: Response to Ordered Action 2/3, Attachment A to Settlement Agreement and Stipulated Final Order HWB-14-20, Los Alamos National Laboratory

This letter provides the response to Ordered Action No. 2/3, pages 8 and 9 in Attachment A to the Settlement Agreement and Stipulated Final Order HWB-14-20 (SFO) entered into by the New Mexico Environment Department (NMED) and the U.S. Department of Energy and Los Alamos National Security, LLC (Respondents) on January 22, 2016. Paragraph 35 of the SFO requires the Respondents to complete corrective actions and submit required evidence of completion to the NMED for approval by specified deadlines as described in Attachment A of the SFO. The requirements for the Respondents' evidence of completion in response to each of the NMED's Ordered Actions are specified in the "Response Actions" column of Attachment A to the SFO. Ordered Action No. 2/3 requires that:

> *No later than 60 days after this Order becomes final, Respondents shall submit to NMED for review and comments the following:*
>
> *A. Reports on the scientific studies Respondents have conducted regarding LANL nitrate salt waste streams since February 14, 2014.*
>
> *B. The nitrate salt waste treatment options assessment report.*
>
> *C. A Plan to determine treatment methods for the nitrate salt waste streams. The Plan shall include a proposed schedule for submission of the following:*
>
> *i. Sampling and analysis plan for unremediated nitrate salt waste*
>
> *ii. Surrogate waste testing plan*
>
> *iii. Report on surrogate waste tests*
>
> *iv. Safe handling and treatment plan for both remediated and unremediated nitrate salt wastes*

The documentation necessary to provide evidence of completion for Ordered Action No. 2/3 consists of several submittals to the NMED. This letter and enclosures either reference past submittals or include information that provides evidence of completion for each of the response actions. Enclosure 1 includes a crosswalk of information required by Ordered Action 2/3 and how each of the response actions is addressed within this submittal.

In fulfillment of Item A above, Enclosure 2 includes a list of reports on the scientific studies that have been conducted on nitrate salt waste streams. The enclosure either includes a reference to previously submitted reports or includes the report as an appendix to the enclosure. For completeness, the list included in Enclosure 2 encompasses all studies that have been completed to date associated with the treatment of nitrate salt waste streams, including the document referenced by Item B and some required for fulfillment of Item C.

Fulfilling the requirements listed in Item C above, Enclosure 3 provides a schedule and an overview of the status of the Respondents' current proposed plan for treatment of nitrate salt-bearing waste within containers at LANL. As applicable, Enclosure 3 provides the overall plan to finalize characterization of nitrate salt waste, test and determine the treatment methodologies for these wastes, and a description of the treatment path for these wastes at LANL. Documents that have been finalized are included or referenced in Enclosure 2. Schedules for submittal of the remaining anticipated documents, that are in draft form or have not been developed because key determinations have not yet been made, are also included within Enclosure 3.

The Respondents would be pleased to meet with the NMED upon request to discuss and explain the documentation included herein. If you have comments or questions regarding this submittal, please contact Mark P. Haagenstad (LANS) at (505) 665-2014 or David Nickless (EM-LA) at (505) 665-6448.

Sincerely,

3/15/16

Michael T. Brandt, DrPH, CIH
Associate Director
Environment, Safety & Health
Los Alamos National Security, LLC
Los Alamos National Laboratory

Sincerely,

Kimberly Davis Lebak

Kimberly Davis Lebak
Manager
Los Alamos Field Office
U.S. Department of Energy

MTB:KBL/

Enclosures:
(1) Summary for Ordered Action 2/3, Attachment A of the Settlement Agreement and Stipulated Final Order
(2) Studies Related to Nitrate Salt Waste Streams and Treatment of Nitrate Salt Waste
(3) Los Alamos National Laboratory Nitrate Salt-Bearing Waste Treatment Planning Schedule

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CERTIFICATION

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

3/15/16

Michael T. Brandt, DrPH, CIH
Associate Director
Environment, Safety, and Health
Los Alamos National Security, LLC
Los Alamos National Laboratory
Operator

Date Signed

3/17/16

Kimberly Davis Lebak
Manager
Los Alamos Field Office
U.S. Department of Energy
Owner/Operator

Date Signed

Response to Ordered Action 2/3, Attachment A to Settlement Agreement and Stipulated Final Order HWB-14-20, Los Alamos National Laboratory (LANL), EPA ID# NM0890010515

March 2016

LA-UR-16-21587

ENCLOSURE 1

Summary for Ordered Action 2/3, Attachment A of the Settlement Agreement and Stipulated Final Order

ADESH-16-043

LA-UR-16-21587

Date: MAR 2 1 2016

Crosswalk for Attachment A of the Settlement Agreement and Stipulated Final Order and Location of Documentation

This enclosure consists of a table populated with information on Ordered Action 2/3 and how each of the response actions is addressed within this submittal. The following table is presented in two parts. The first documents completed actions and the second documents the actions in progress.

Topic Addressed by Response Action	Applicable Response Actions	Documentation to Provide as Evidence of Completion	Location of Discussion and Evidence of Completion
Items Listed as Complete			
A. Reports on the scientific studies Respondents have conducted regarding LANL nitrate salt waste streams since February 14, 2014.	A. Remediated Nitrate Salt Chemical Reactivity Study	*Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes, D. L. Clark, D.J. Funk, LA-UR-15-22393*	Response to Ordered Action 2/3, Enclosure 2 includes a link to this document
A. Reports on the scientific studies Respondents have conducted regarding LANL nitrate salt waste streams since February 14, 2014. (Supplemental)			Although the "Documentation to Provide as Evidence of Completion" column includes only one document to be provided, the Respondents have included all reports currently available for completeness and in support of the final determination for treatment effectiveness Response to Ordered Action 2/3, Enclosure 2, Table (List of Studies Related to Nitrate Salt Waste and Treatment of Nitrate Salt Waste)

Crosswalk for Attachment A of the Settlement Agreement and Stipulated Final Order and Location of Documentation

Topic Addressed by Response Action	Applicable Response Actions	Documentation to Provide as Evidence of Completion	Location of Discussion and Evidence of Completion
B. The nitrate salt waste treatment options assessment report.	B. Nitrate Salt Waste Options Assessment Report	*Nitrate Salt Options Assessment Report* LA-UR-15-25355	Response to Ordered Action 2/3, Enclosure 2, Appendix 3 Please note: the final title for this document is, *Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory* and the LA-UR number changed prior to finalization of the report
C. A Plan to determine treatment methods for the nitrate salt waste streams. The Plan shall include a proposed schedule for submission of the following: i. Sampling and analysis plan for unremediated nitrate salt waste ii. Surrogate waste testing plan iii. Report on surrogate waste tests iv. Safe handling and treatment plan for both remediated and unremediated nitrate salt wastes	C. Remediation/Scheduling Plan as discussed in technical meetings. The Plan shall include referenced plans and a schedule for the surrogate waste test report.	*Remediation/Scheduling Plan*, including the following: [See the following two rows for 1. and 2. details] AND *3. A schedule for the Final Report on Surrogate Waste Tests* (Final Title TBD)	Response to Ordered Action 2/3, Enclosure 3 Please note: the final title for this document is, *Los Alamos National Laboratory Nitrate Salt-Bearing Waste Treatment Planning Schedule*

Crosswalk for Attachment A of the Settlement Agreement and Stipulated Final Order and Location of Documentation

Topic Addressed by Response Action	Applicable Response Actions	Documentation to Provide as Evidence of Completion	Location of Discussion and Evidence of Completion
C. i. Sampling and analysis plan for unremediated nitrate salt waste		*1. Sampling and Analysis Plan for Unremediated Nitrate Salt Waste, ENV-DO-15-0248, LA-UR-15-26357*	Response to Ordered Action 2/3, Enclosure 2 includes a link to this document Please note: the final title for this document is, *Sampling and Analysis Plan Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory* Description of the intended use of this plan is discussed in Response to Ordered Action 2/3, Enclosure 3, Section 2
C.ii. Surrogate waste testing plan		*2. Treatment Study Work Plan for Nitrate salt Transuranic (TRU) Wastes*	Response to Ordered Action 2/3, Enclosure 2, Appendix 5 Please note: the final title for this document is, *Treatment Study Plan for Nitrate Salt Waste Remediation* Description of the plan's use is discussed in Response to Ordered Action 2/3, Enclosure 3, Section 3

Crosswalk for Attachment A of the Settlement Agreement and Stipulated Final Order and Location of Documentation

Topic Addressed by Response Action	Applicable Response Actions	Documentation to Provide as Evidence of Completion	Location of Discussion and Evidence of Completion
Additional information requested by NMED	C. Remediation/Scheduling Plan as discussed in technical meetings. The Plan shall include referenced plans and a schedule for the surrogate waste test report. NMED requested LA-CIN01.001 Liquid Sampling Information Letter (ENV-DO-15-0313, LA-UR-1528468)	*NMED requested LA-CIN01.001 Liquid Sampling Information Letter* (ENV-DO-15-0313, LA-UR-15-28468) with the following attachments: • List of Containers sampled; • List of Containers not sampled, but attempted; • List of Containers of Interest; • RTR Videos of Containers of Interest; • Analytical results	Response to Ordered Action 2/3, Enclosure 2 includes a link to this document Please note: the final title for this document is, *Sampling and Analysis Information for LA-CIN01 Waste Containers Los Alamos National Laboratory*
Items Listed as In-Progress/Ongoing			
C.iii. Report on surrogate waste tests	Final Report on Surrogate Waste Tests (Final Title TBD) NOTE: The response to item C.iii [Final Report on Surrogate Waste Tests (Final Title TBD)] will include UNS and SWERI analytical results.		Discussion and schedule for the submittal of information for these reports is discussed in Response to Ordered Action 2/3, Enclosure 3, Sections 3 & 4, and Table 1
C.iv. Safe handling and treatment plan for both remediated and unremediated nitrate salt wastes		[NOTE: Safe Handling and Treatment Plan for Remediated Nitrate Salt (RNS) and Unremediated Nitrate Salt (UNS) Wastes is addressed under the Remediation/Scheduling Plan]	Discussion and schedule for the submittal of this plan is discussed in Response to Ordered Action 2/3, Enclosure 3, Section 5 Please note: This is considered to be part of the Respondents' future permit modification request

ENCLOSURE 2

Studies Related to Nitrate Salt Waste Streams and Treatment of Nitrate Salt Waste

ADESH-16-043

LA-UR-16-21587

Date: MAR 2 1 2016

List of Studies Related to Nitrate Salt Waste and Treatment of Nitrate Salt Waste

Document Title	**Location**
Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes LA-UR-15-22393	ENV-DO-15-0097: Transmittal of Referenced Report on Remediated Nitrate Salt Wastes http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600350
Interpretation of Headspace Gas Observations in Remediated Nitrate Salt Waste Containers Stored at Los Alamos National Laboratory LA-UR-15-22661	ESHID-600373: HSG Data Report and Presentation Slides for NMED-LANL Meeting held on Thursday, April 16, 2015 http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600373
Sampling and Analysis Information for LA-CIN01 Waste Containers Los Alamos National Laboratory LA-UR-15-28468	ENV-DO-15-0313: Sampling and Analysis Information for LA-CIN0l Waste Containers, Los Alamos National Laboratory http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-601010 http://eprrdata.lanl.gov/eprrdata/Files/ESHID-601010-2.zip
Sampling and Analysis Plan Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory LA-UR-15-26357, Rev. 1	ENV-DO-15-024: Transmittal of Sampling and Analysis Plan for Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600920
Remediated Nitrate Salt Surrogate Formulation, Aging, and Testing Procedure, PLAN-TA9-2443(U), Rev. B LA-UR-16-21746	Appendix 1
Data Report for the Drum-scale Thermal Transport Characterization LA-UR-16-20004	Appendix 2
Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory LA-UR-15-27180	Appendix 3
The Path to Nitrate Salt Disposition LA-UR-16-21760	Appendix 4
Treatment Study Plan for Nitrate Salt Waste Remediation Revision 2.1 LA-UR-15-27971	Appendix 5

List of Studies Related to Nitrate Salt Waste and Treatment of Nitrate Salt Waste

Document Title	**Location**
Statistical Modeling Effects for Headspace Gas LA-UR-16-21293	Appendix 6
Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing LA-UR-15-28900	Appendix 7
Engineered Option Treatment of Remediated Nitrate Salts: Surrogate Batch-Blending Testing LA-UR-16-21653	Appendix 8

Appendix 1

Remediated Nitrate Salt Surrogate Formulation, Aging, and Testing Procedure

LA-UR-16-21746

Approved for public release; distribution is unlimited.

Title: PLAN-TA9-2443(U), Rev. B Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing Standard Procedure

Author(s): Brown, Geoffrey Wayne

Intended for: Needed for the NMED Submittal UNS Nonsample Technical Justification

Issued: 2016-03-16

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LA-UR-16-21746

Los Alamos NATIONAL LABORATORY EST. 1943

PLAN-TA9-2443(U), Rev. B

TITLE: Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing Standard Procedure

This section to be completed by Document Control Team
EFFECTIVE DATE: 2/16/16
EXPIRATION DATE: 2/16/17

Review Cycle:	☒ 1 Year	☐ 2 Years	☐ 3 Years
Procedure Usage Designation:	☒ Reference	☐ Use Every Time	☐ UET Sections:

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YOU ARE RESPONSIBLE FOR VERIFYING THAT YOU ARE WORKING TO THE MOST CURRENT REVISION OF THIS DOCUMENT.

Revision History

Revision	Date	Description of Change
A	7/27/15	Initial release
B	2/16/16	Corrected typographical errors and minor inaccuracies in introduction, formulation section, testing section, and quality assurance section. Removed Vacuum Thermal Stability.

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1.0 INTRODUCTION

1.1 Purpose

This document identifies scope and some general procedural steps for performing Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing.

LANL created 600 barrels of nuclear waste with a combination of different nitrate salts mixed with Swheat Scoop cat litter. The resulting product is a fuel/oxidizer mixture that tests positive for RCRA ignitability (D001 characteristic). The hazard of this situation became evident when Drum 68660 spontaneously breached and contaminated panel 7 at the Waste Isolation Pilot Plant (WIPP) on February 14, 2014. Vast experimental and theoretical effort has been pursued to arrive at a reasonable recipe for a simulant with similar proportions of nitrate salts and fuel as that represented by Drum 68660. Quantification of the likely sensitivity of the barrel contents is necessary in preparation for remediation of the waste to a non-ignitable form (removal of RCRA characteristic D001). For that purpose, a surrogate formulation must be chosen that should represent the energetic properties of the waste without including any radioactive hazard.

This Test Plan describes the requirements, responsibilities, and process for preparing and testing a range of chemical surrogates intended to mimic the energetic response of waste created during processing of legacy nitrate salts. The surrogates developed are expected to bound[1] the thermal and mechanical sensitivity of such waste, allowing for the development of process parameters required to minimize the risk to worker and public when processing this waste. Such parameters will be based on the worst-case kinetic parameters as derived from APTAC measurements as well as the development of controls to mitigate sensitivities that may exist due to friction, impact, and spark. This Test Plan will define the scope and technical approach for activities that implement Quality Assurance requirements relevant to formulation and testing. This Test Plan conforms to ASME NQA-1-2009A, Subpart 4.2, “Guidance on Graded Application of the Nuclear Quality Assurance (NQA) Standard for Research and Development”.

1.2 Scope

This document covers the requirements for preparation of material and sensitivity testing to gauge the response of remediated nitrate salt waste that used Swheat Scoop cat litter as an absorbent. Previous testing has indicated that at least two factors are critical for ignition of the formulation. These include the ratio of Swheat scoop cat litter to the nitrate salt and the concentration of lead salts in the formulation. The ratio of Swheat to salt influences the oxygen balance of the formulation and therefore the thermodynamic ability to combust without added oxygen. We determined through previous testing that lead nitrate is a catalyst for the ignition process. The amount of lead actually present in the waste is difficult to estimate precisely due to the complexity of its formation. In addition, heating and partially drying the materials will result in additional worst-case scenarios: prior testing has indicated that the dried material is more thermally sensitive.

[1] Bound is defined as “exhibiting thermal sensitivities that are consistent with the observed behavior of drum 68660 within room 7 of panel 7 at WIPP.”

2.0 PRECAUTIONS AND LIMITATIONS

- All work described above is covered by IWDs that have had ES&H review for all hazards and processes. The IWDs covering this work are:
  1. IWD-TA9-022, Novel Energetic Material Synthesis and Small Scale Formulation
  2. IWD-TA9-2309, WX-7 Chemical Operations
  3. TP-IWD-TA9-134, Mixing, Formulation, Preparation, and Scale-up of Composite Energetic Materials
  4. TP-IWD-TA9-193, Small-Scale Sensitivity Testing of Energetic Materials
  5. TP-IWD-TA9-2189, Thermal Analysis
- Test Plan Changes: Changes to this Test Plan that redefine work scope or processes will be documented in an approved revision. Release of the revision will require new signatures on the coversheet. Administrative changes or changes to the experimental details that do not affect the purpose or scope of the plan shall be documented in a scientific notebook.

3.0 PREREQUISITES

3.1 Prerequisite Actions

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

3.2 Training

Applicable training requirements are to be found in the IWDs required to carry out this work in the M-7 laboratories.

Qualification and Approval of specific workers for activities in the IWDs in Section 2 are achieved through the Worker Qualification and Authorization System in the Utrain System. When a worker is Approved for a given IWD or IWD subtask in WQAS, the RLM has acknowledged that the worker is qualified for the task.

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

4.0 PROCEDURE

This procedure describes the formulation of RNS surrogate salt and salt/organic formulations at lab scale (2-60 g) as well as the sensitivity testing of the surrogate. This formulation scale is adequate for all small-scale sensitivity testing that will be performed as part of the safety basis analysis. All activities described below are peer reviewed for technical accuracy and quality of records as evidenced by appropriate signature authorities on the coversheet of this plan. Peer Review of individual tasks within an IWD follows the guidelines of P101-8, Explosives Safety. Peer Review of full IWD documents follows guidelines of AP-JDIV-1019, Integrated Work Documents. Analytical reports are peer reviewed before release. Analytical Reports and other technical Memoranda are archived in PDMLink. For this activity, both types of documents will include copies of lab notebooks, as applicable.

Nitrate salts, oxalic acid, and potassium carbonate will be acquired from IESL-approved vendors and be 99% or higher purity. Often this means the materials meet standards for chemical purity in accordance with the ACS as identified by "ACS reagent grade" or "ACS Certified", which imply 99% or higher purity. Water is obtained through reverse osmosis of tap water or IESL-procured, 99% or higher purity LCMS-grade water is used. For purchased chemicals, upon receipt the item will be checked against the packing slip and the lot number and ChemDB inventory number will be noted on the packing slip. The packing slip will then be signed to confirm inspection and receipt. The Certificate of Analysis for the particular lot of material will be obtained from the vendor and archived along with the signed packing slip as part of a M-7 memorandum in PDMLink.

Swheat Scoop cat litter is procured through commercial sources. All glassware that is not disposable will be prepared the day before use by cleaning according standard laboratory procedures until they are free from contamination by visual inspection and then allowed to dry overnight.

4.1 Surrogate Salt Formulation

Through previous testing, analysis of waste records and simulations of process streams, a surrogate recipe was developed that has small scale thermal properties expected to be similar to Drum 68660 and which also represents an average of the contents of that drum. This recipe was also tested at a 55 gallon-drum scale with results similar to what is thought to have happened with Drum 68660[2]. The work in this test plan is based on that recipe with variations in the Swheat (fuel) content and Pb content (catalyst) to determine the most sensitive surrogate formulation. These variations will be formulated with respect to the nominal formulation where all relative proportions are held constant. The nominal recipe for preparing the independent surrogate shall be as follows:

[2] G. R. Parker, M. D. Holmes, E. M. Heatwole, P. Leonard, and C. P. Leibman, "The Thermolytic Response of a Surrogate Remediated Nitrate Salts (RNS) Waste Mixture at the Drum Scale," (Draft) LA-UR-15-29229 (2015).

Nominal Formulation

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

[a] **Masses are +/- 1 mg**

[b] **Weight % values are +/- 0.01 %**

To this formulation will be added lead nitrate ($Pb(NO_3)_3$) and Swheat according to the following matrix where percentages refer to the weight % of the material in the final product formulation. Attachment A has all recipes listed in detail.

4% $Pb(NO_3)_3$; 15% Swheat	4% $Pb(NO_3)_3$; 25% Swheat	4% $Pb(NO_3)_3$; 35% Swheat
2% $Pb(NO_3)_3$; 15% Swheat	2% $Pb(NO_3)_3$; 25% Swheat	2% $Pb(NO_3)_3$; 35% Swheat
1% $Pb(NO_3)_3$; 15% Swheat	1% $Pb(NO_3)_3$; 25% Swheat	1% $Pb(NO_3)_3$; 35% Swheat

All of the formulations in the matrix above will initially be made and tested once. After that first round, the matrix will be made and tested two more times so that, in the end, everything will have been done in triplicate.

4.2 Formulation

4.2.1 The masses of nitrate salt components are measured in a plastic or aluminum weigh-boat or on waxed-paper using a balance calibrated to +/- 10 mg uncertainty. The quantity of material measured will be within 10 mg of the desired quantity of material.

4.2.2 The weighed portion of nitrate salt will be transferred to a ceramic mortar.

4.2.3 Once all of the nitrate salts have been measured and placed into the mortar they will be ground together using a pestle for about one minute.

4.2.4 The mass of Swheat Scoop cat litter is measured in a plastic or aluminum weigh-boat or on waxed-paper using a balance calibrated to +/- 10 mg uncertainty. The quantity of material measured will be within 10 mg of the desired quantity of material.

4.2.5 The weighed portion of Swheat Scoop cat litter will be transferred to a second ceramic mortar.

4.2.6 Swheat Scoop cat litter will be ground in the mortar using a pestle for about one minute.

4.2.7 The mass of oxalic acid dihydrate and potassium carbonate will be measured in a plastic or aluminum weigh-boat or on waxed-paper using a balance calibrated to +/- 10 mg uncertainty. The quantity of material measured will be within 10 mg of the desired quantity of material.

4.2.8 Water will be measured into a tared glass beaker using a balance calibrated to +/- 10 mg uncertainty.

4.2.9 The oxalic acid dihydrate and potassium carbonate will be added to the water and stirred until well mixed.

4.2.10 The potassium oxalate mixture formed above will be added to the ground nitrate salts and manually mixed for approximately 1 minute, or until homogenous, using a spatula.

4.2.11 The Swheat Scoop cat litter will be added to the wetted nitrate salt mixture and the resulting formulation mixed for approximately 1 minute, or until homogenous, using a spatula.

4.2.12 The mixture of wetted nitrate salt and Swheat Scoop cat litter is transferred to a glass container.

4.2.13 Samples will be labeled with their designated name, the date and time of preparation, and all appropriate hazard labels.

4.2.14 The glass container is heated using a hotplate with a surface temperature of approximately 60 ˚C for 4 hours. The container is loosely covered and heated in a ventilation hood.

4.2.15 The cover is removed and the material is allowed to stand overnight at room temperature in a ventilation hood.

4.2.16 The material is transferred to a plastic container and submitted for testing

4.2.17 Samples will be stored with caps secure in a normal laboratory environment.

4.2.18 Each test will be started no earlier than 24 hours after formulation and no later than 4 days after formulation. The actual formulation and testing dates will be recorded in the documentation. If all testing cannot be started within this 3-day window, the formulation will be re-made and all tests re-performed.

4.3 Sensitivity Testing

4.3.1 Technical details of the various sensitivity tests are provided in Appendix 2. The quality of each of the tests relies on different aspects of the testing. These are noted in the following subsections.

4.3.2 Sensitivity testing will include differential scanning calorimetry (DSC), Drop Weight Impact testing, Friction sensitivity, Electrostatic Spark Discharge testing, and Automatic Pressure-Tracking Adiabatic Calorimetry testing (APTAC).

4.3.3 Vacuum Thermal Stability (VTS) testing was included in the initial release of this document. After the first few formulations however, it was determined that VTS did not provide any useful information for these materials. The materials are being evaluated for their low thermal sensitivity and concomitant high gas generation rates, which makes this test moot. Furthermore, similar data up to much higher pressures is obtained from the APTAC instrument described below.

4.3.4 The DSC procedure is documented in WX-7-AC-11-002, “Standard DSC Procedure”. Drop Weight, Friction, and Spark testing procedures are documented in TP/IWD-TA9-193, “Small-Scale Sensitivity Testing of Energetic Materials.” The APTAC testing procedure is described below.

4.3.5 The DSC instrument and software operation are verified using an Indium standard supplied by the vendor and traceable to the National Physical Laboratory in the UK. The indium scan verifies the

temperature measurement capability of the instrument and the enthalpy measurement capability. For this work we will request that the instrument operation be checked by indium both before and after running the samples. There are no other process aids or equipment that significantly influence the temperatures and enthalpies measured by DSC. The model and serial number of the DSC and balance used for the testing will be recorded in the laboratory report.

4.3.6 The VTS instrument and software operation are verified using one or more internal explosive standards with known gas generation properties based on repeated historical measurements. For this work we will request standards to be run concurrently with the samples. There are no other process aids or equipment that significantly influence the temperatures and gas generation measured by VTS. The model and serial number of the VTS instrument and balance used for this work will be recorded in the laboratory report. The lot numbers of the internal standards are part of the analytical lab report data.

4.3.7 Verification of the Drop Weight Impact testing machine is accomplished by testing internal explosive standards with known DWI properties based on repeated historical measurements. The DWI result is only meaningful relative to the response of these standards. The 50% reaction level is established using Commercial-Off-the-Shelf software: the SenTest software package from Neyer software. When this software was purchased several years ago, its operation was checked against a number of known internal standards to see that it produced expected results. This testing and the periodic checks with internal standards verify the operation of the instrument and software. There are no other process aids or equipment that significantly influence the sample response. For this measurement we will request standards to be run both before and after the samples. The lot numbers of the standards are part of the analytical report data.

4.3.8 Verification of the Friction testing machine is accomplished by testing internal explosive standards with known friction response properties based on repeated historical measurements. The Friction sensitivity result is only meaningful relative to the response of these standards. The 50% reaction level is established using Commercial-Off-the-Shelf software: the SenTest software package from Neyer software. When this software was purchased several years ago, its operation was checked against a number of known internal standards to see that it produced expected results. This testing and the periodic checks with internal standards verify the operation of the instrument and software. There are no other process aids or equipment that significantly influence the sample response. For this measurement we will request standards to be run both before and after the samples. The lot numbers of the standards are part of the analytical report data.

4.3.9 Verification of the Electrostatic Spark Discharge testing machine is accomplished by testing internal explosive standards with known ESD properties based on repeated historical measurements. The ESD result is only meaningful relative to the response of these standards. There are no other process aids or equipment that significantly influence the sample response. For this measurement we will request standards to be run both before and after the samples. The lot numbers of the standards are part of the analytical report data.

4.4 APTAC Testing

4.4.1 Temperature verification: The instrument thermocouple that measures the sample temperature is verified and corrected by measuring its response relative to a more precise thermocouple that is calibrated. Attach both thermocouples to a metal block, and in contact with each other, and record their responses at approximately 10 ˚C steps from approximately 40 ˚C to over 150 ˚C.

4.4.2 Pressure verification: The instrument pressure transducers are verified by measuring their response relative to a more precise gauge that is calibrated. This gauge is accurate to 2 psi. Pressure readings will be verified at 100 psi intervals from near atmospheric pressure (open vessel) to 500 psi.

4.4.3 Instrument verification: Following the APTAC instrument acceptance manual, verify that DTBP shows the expected exothermic behavior as defined in that manual. The DTBP and toluene must be purchased from an IESL vendor and certificates of analysis must be obtained. The instrument and software operation are verified by the DTBP results meeting manufacturer's specifications.

4.4.4 Unless otherwise noted below, follow the general APTAC manual instructions for setting up and running the required type of test (Heat-Wait-Search or Isothermal).

4.4.5 A 10 ml titanium sample holder is to be used for the testing. The sample holder should be cleaned with acetone and dried overnight at 200 ˚C. If there is residue remaining from a previous test, obtain a new sample holder.

4.4.6 Record the weight of the sample bomb to the nearest 10 mg using a calibrated scale (+/- 10 mg). Weigh approximately 4 grams of the sample into the bomb and record the loaded sample weight to the nearest 10 mg. Record the weight of foil and any other items attached to the bomb for testing.

4.4.7 Following the instrument manual, prepare the sample bomb and instrument for testing. Load the experimental parameters into the APTAC instrument software. For Heat-Wait-Search testing, use steps of 2 ˚C.

4.4.8 After the test is completed, use the APTAC data analysis software to determine the onset of self-heating, the heat of reaction, and kinetic parameters.

4.4.9 The onset of self-heating is evident from the temperature before the exothermic segment begins. The heat of reaction is determined from a Horizontal Step measurement of the exothermic segment. The kinetic parameters are determined by the analyst through visual best fit of the available models to the data.

4.4.10 After all sample testing is completed, or earlier if deemed necessary, repeat the DTBP instrument check described above.

4.4.11 The two software packages used in this testing are integral to the instrument. Both are from the instrument manufacturer and are COTS and proprietary. The expected test results from the DTBP sample indicate that the instrument and software are functioning properly.

5.0 QUALITY ASSURANCE

ASME NQA-1-2009A, Subpart 4.2, "Guidance on Graded Application of the Nuclear Quality Assurance (NQA) Standard for Research and Development" guided the development of this Test Plan. The test plan conforms to SD330, Los Alamos National Laboratory Quality Assurance Program. SD330 is implemented within M Division using PLAN-WXDIV-2142, WX Division Quality Assurance Plan.

As part of the Quality Assurance activities for this work, the QA-SME may request table top and walk down reviews of documents and tasks prior to the start of formulation and analysis. The QA-SME may also request to observe the actual formulation and analysis of recipes listed in Attachment A. Due to the limited scope of this plan, surveillances will be performed by Environmental Program deployed QA SMEs utilizing QPA-DO-FSD-007.006 Quality Assurance Surveillances.

Examples of documents that the QA-SME may choose to review include calibration records for specific items, chemical receipt records, and Lot Certificates of Analysis. Formal calibration records are available from S&CL. Chemical receipt records and Lot Certificates of Analysis will be provided in a M-7 memorandum.

6.0 NONCONFORMANCES

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

7.0 DOCUMENT MANAGEMENT

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

8.0 TEST PLAN REVIEW AND APPROVAL

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

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

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

9.0 TEST PLAN CHANGES

Changes to the issued Test Plan that redefine work scope or processes will be documented in an approved revision to this Test Plan. Administrative changes or changes to the experimental details that do not affect the purpose or scope of the plan shall be documented in a scientific notebook.

10.0 RECORDS AND RECORD REQUIREMENTS

Records compiled or generated by this process include:

- Receipt documentation for the process chemicals
- Certificates of analysis for the process chemicals
- Calibration records for the balances and equipment used in formulation and testing (if noted in section 4 above)
- Signed notebook pages showing the formulation process outlined above and the actual masses used for the formulation/testing
- Analytical Testing reports for the sensitivity testing.

Records will be compiled into M-7 memoranda or reports that will be uploaded to PDMLink for archival purposes.

A final memo will include a list of the Analytical Reports, memoranda, and SQM documents that fulfill the requirements of this test plan.

11.0 SOFTWARE QUALITY MANAGEMENT

Software used with the instruments described above is managed through Software Quality Management Plans controlled by M Division. Before testing begins, SQM documents will be released for the following software:

- Differential Scanning Calorimeter control software
- Differential Scanning Calorimeter data analysis software
- APTAC control software
- APTAC data analysis software
- APTAC reporting software
- SenTest sensitivity testing software

12.0 ENVIRONMENT, SAFETY, AND HEALTH

All work described above is covered by IWDs that have had ES&H review for all hazards and processes.

13.0 RESPONSIBILITIES

13.1 Responsible Line Manager

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

13.2 Principal Investigator

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

13.3 Technical Reviewer

13.3.1 Confirms accuracy, adequacy, and completeness of this Test Plan

13.4 Document Control

- Assigns document number and effective date for this Test Plan

13.5 Worker

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

14.0 ACRONYMS

Term	Description
ACS	American Chemical Society
DOE	United States Department of Energy
DSC	Differential Scanning Calorimetry

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

15.0 ATTACHMENTS

Number	Title
A	Surrogate Recipes
B	Test Descriptions
C	Quality Implementation Matrix

Attachment A: SURROGATE RECIPES

Recipes with 15% SWheat.

Material	Milligrams	grams	wt %	
Al(NO3)3 * 9 H20	2145	2.145	3.57	
Ca(NO3)2 * 4 H2O	8530	8.530	14.22	
Cr(NO3)3 * 9H2O	105	0.105	0.17	Actual
Fe(NO3)3 * 9H2O	3258	3.258	5.43	Pb salt as
Mg(NO3)2 * 6H2O	23939	23.939	39.90	% of salts
NaNO3	5307	5.307	8.85	and acid
Pb(NO3)2	1884	1.884	3.14	4.00
(COOH)2*2H20	1936	1.936	3.23	
K2CO3	1010	1.010	1.68	
Swheat	9000	9.000	15.00	
Water	2886	2.886	4.81	

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	2189	2.189	3.65	
Ca(NO3)2 * 4 H2O	8708	8.708	14.51	
Cr(NO3)3 * 9H2O	107	0.107	0.18	Actual
Fe(NO3)3 * 9H2O	3326	3.326	5.54	Pb salt as
Mg(NO3)2 * 6H2O	24438	24.438	40.73	% of salts
NaNO3	5418	5.418	9.03	and acid
Pb(NO3)2	942	0.942	1.57	2.00
(COOH)2*2H20	1976	1.976	3.29	
K2CO3	1010	1.010	1.68	
Swheat	9000	9.000	15.00	
Water	2886	2.886	4.81	

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	2212	2.212	3.69	
Ca(NO3)2 * 4 H2O	8797	8.797	14.66	
Cr(NO3)3 * 9H2O	108	0.108	0.18	Actual
Fe(NO3)3 * 9H2O	3360	3.360	5.60	Pb salt as
Mg(NO3)2 * 6H2O	24687	24.687	41.15	% of salts
NaNO3	5473	5.473	9.12	and acid
Pb(NO3)2	471	0.471	0.79	1.00
(COOH)2*2H20	1997	1.997	3.33	
K2CO3	1010	1.010	1.68	
Swheat	9000	9.000	15.00	
Water	2886	2.886	4.81	

Attachment A: SURROGATE RECIPES (cont'd)

Recipes with 25% SWheat.

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	1892	1.892	3.15	
Ca(NO3)2 * 4 H2O	7527	7.527	12.54	
Cr(NO3)3 * 9H2O	92	0.092	0.15	Actual
Fe(NO3)3 * 9H2O	2875	2.875	4.79	Pb salt as
Mg(NO3)2 * 6H2O	21123	21.123	35.20	% of salts
NaNO3	4683	4.683	7.80	and acid
Pb(NO3)2	1663	1.663	2.77	4.00
(COOH)2*2H20	1708	1.708	2.85	
K2CO3	891	0.891	1.48	
Swheat	15000	15.000	25.00	
Water	2546	2.546	4.24	

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	1932	1.932	3.22	
Ca(NO3)2 * 4 H2O	7683	7.683	12.81	
Cr(NO3)3 * 9H2O	94	0.094	0.16	Actual
Fe(NO3)3 * 9H2O	2935	2.935	4.89	Pb salt as
Mg(NO3)2 * 6H2O	21563	21.563	35.94	% of salts
NaNO3	4780	4.780	7.97	and acid
Pb(NO3)2	831	0.831	1.39	2.00
(COOH)2*2H20	1744	1.744	2.91	
K2CO3	891	0.891	1.48	
Swheat	15000	15.000	25.00	
Water	2546	2.546	4.24	

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	1951	1.951	3.25	
Ca(NO3)2 * 4 H2O	7762	7.762	12.94	
Cr(NO3)3 * 9H2O	95	0.095	0.16	Actual
Fe(NO3)3 * 9H2O	2965	2.965	4.94	Pb salt as
Mg(NO3)2 * 6H2O	21783	21.783	36.30	% of salts
NaNO3	4829	4.829	8.05	and acid
Pb(NO3)2	416	0.416	0.69	1.00
(COOH)2*2H20	1762	1.762	2.94	
K2CO3	891	0.891	1.48	
Swheat	15000	15.000	25.00	
Water	2546	2.546	4.24	

Attachment A: SURROGATE RECIPES (cont'd)

Recipes with 35% SWheat.

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	1640	1.640	2.73	
Ca(NO3)2 * 4 H2O	6523	6.523	10.87	
Cr(NO3)3 * 9H2O	80	0.080	0.13	Actual
Fe(NO3)3 * 9H2O	2492	2.492	4.15	Pb salt as
Mg(NO3)2 * 6H2O	18306	18.306	30.51	% of salts
NaNO3	4058	4.058	6.76	and acid
Pb(NO3)2	1441	1.441	2.40	4.00
(COOH)2*2H20	1481	1.481	2.47	
K2CO3	772	0.772	1.29	
Swheat	21000	21.000	35.00	
Water	2207	2.207	3.68	

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	1674	1.674	2.79	
Ca(NO3)2 * 4 H2O	6659	6.659	11.10	
Cr(NO3)3 * 9H2O	82	0.082	0.14	Actual
Fe(NO3)3 * 9H2O	2544	2.544	4.24	Pb salt as
Mg(NO3)2 * 6H2O	18688	18.688	31.15	% of salts
NaNO3	4143	4.143	6.90	and acid
Pb(NO3)2	720	0.720	1.20	2.00
(COOH)2*2H20	1511	1.511	2.52	
K2CO3	772	0.772	1.29	
Swheat	21000	21.000	35.00	
Water	2207	2.207	3.68	

Material	milligrams	grams	wt %	
Al(NO3)3 * 9 H20	1691	1.691	2.82	
Ca(NO3)2 * 4 H2O	6727	6.727	11.21	
Cr(NO3)3 * 9H2O	83	0.083	0.14	Actual
Fe(NO3)3 * 9H2O	2570	2.570	4.28	Pb salt as
Mg(NO3)2 * 6H2O	18879	18.879	31.46	% of salts
NaNO3	4185	4.185	6.98	and acid
Pb(NO3)2	360	0.360	0.60	1.00
(COOH)2*2H20	1527	1.527	2.54	
K2CO3	772	0.772	1.29	
Swheat	21000	21.000	35.00	
Water	2207	2.207	3.68	

Attachment B: TEST DESCRIPTIONS

Differential Scanning Calorimetry (DSC)

DSC measures the thermal response of a material by monitoring the heat flow into or out of that material as it is heated at a constant ramp rate. A 1 mg sample of the material is held in a sealed aluminum pan. The pan is placed in an instrumented furnace with an empty reference pan and the furnace is ramped at 10 ˚C/min while heat flow to the sample and reference pans is monitored. Endothermic events require more heat to flow to the sample to keep its temperature increasing at the desired ramp rate. Exothermic events cause the furnace power to be reduced for the same reason. With this method, melts, phase transitions, decomposition, and other features can be quantitatively measured.

Drop Weight Impact (DWI)

DWI is a statistical test to determine the 50% reaction level of a material to impact stimulus. In this test, a fixed volume of material is placed on a sand paper disk on top of a steel anvil. A steel striker is placed on the sample and impacted by a 2.5 kg mass falling from a predetermined height. Microphones record the sound generated by the impact. Sound above the intensity due to a blank sandpaper disk is attributed to a reaction in the material (a GO event). Sound below that intensity indicates no reaction in the material (a NO GO event). Commercial software evaluates the GO and NO GO events and adjusts the required height of the 2.5 kg mass to map out the reaction probability distribution. The 50% level is assessed assuming that the measured reaction is Gaussian.

Friction Sensitivity

Friction sensitivity testing is a statistical test to determine the 50% reaction level of a material to impact stimulus. In this test, a fixed volume of material is placed on a ceramic plate on a movable platform. A ceramic pin on a lever arm is lowered onto the sample and weight is added to the arm to produce a predetermined friction force. The platform is forced to move under the pin by a motor and reaction indications are assessed by the instrument operator. Smoke, sound, or black marks on the ceramic are attributed to a reaction in the material (a GO event). Lack of these features indicates no reaction in the material (a NO GO event). Commercial software evaluates the GO and NO GO events and adjusts the required weight to map out the reaction probability distribution. The 50% level is assessed assuming that the measured reaction is Gaussian.

Electrostatic Spark Discharge Sensitivity (ESD)

ESD is a threshold level determination test that evaluates sensitivity of a material to spark discharge stimulus. In this test, a fixed volume of material is added to a sample holder that insulates the material from everything except the bottom electrode of the platform. A piece of scotch tape is placed over the sample holder, enclosing the sample area. The sample holder is placed on the platform and a needle is charged to a predetermined energy with a capacitor bank. The needle is then pushed through the tape and the energy is discharged to the bottom electrode through the sample. If the sample reacts, gas is generated and the tape is torn and sometimes obliterated. If there is no reaction, the tape is only punctured by the needle. The operator assesses the result of the test and varies the energy over a number of different replicates to determine the energy at which there are 20 consecutive NO GO events

Attachment B: TEST DESCRIPTIONS (cont'd)

with at least one GO event at the next higher energy level. The level of the 20 consecutive NO GO events is reported as the Threshold Initiation Level.

Automatic Pressure Tracking Adiabatic Calorimetry (APTAC)

APTAC is a measurement that determines the temperature at which a material begins to self-heat and monitors the thermal and pressure behavior of that material during the self-heating. In this test, several grams of material are loaded into a titanium sample bomb that is mounted inside a furnace. The bomb is instrumented with a pressure line and thermocouple that is inserted into the sample. In a typical experiment, the sample is heated in 2 ˚C steps and the temperature is monitored at each step for some tens of minutes. If there is no indication of self-heating, the next step is taken. If the sample does begin to self-heat, the instrument switches to its tracking mode and ramps the furnace at the same rate that the sample is self-heating. This produces adiabatic conditions – the sample cannot lose heat to the surroundings. The heating stops when the heating rate exceeds the limit of the instrument, the pressure exceeds limits, or the sample temperature exceeds a predetermined threshold. The onset temperature of the self-heating is an important metric for ranking materials relative to one another in terms of thermal stability. The adiabatic nature of the measurement makes this more relevant to larger masses whose thermal conductivity may inhibit heat loss from a hot spot. The onset and rate of heating can also be used to determine kinetic parameters that allow predictions to be made for the material in other scenarios, enabling the development of process parameters for reprocessing of the remediated nitrate salt waste stream.

Attachment C - QUALITY IMPLEMENTATION MATRIX

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

Attachment C: QUALITY IMPLEMENTATION MATRIX

		the formality associated with the preparation and revision of design and process documentation, and by including in the milestones a plan for evaluating performance at various key junctures during the project. Tests are prescribed with requirements commensurate with the complexity and scale of the work, and with the associated risk to the public, workers, and environment and future success of the project.	
3	Design Control	603.4 Development and Support. For development and support activities, the level of design control should be applied to support the input needs of the design process. In some cases, considerable importance is placed on R&D results to demonstrate the acceptability of innovative design.	Not Applicable. Nothing is being designed.
4	Procurement Document Control	604.4 Development and Support. For development and support activities, the level of procurement document control should be applied to support a commercial design basis, i.e., engineering design system criteria.	SD330, P840-1, PLAN-WXDIV-2142, P1020-2, and P1020-1. In this Test Plan, the relevant procurement documents are the Certificates of Analysis from Fisher for the chemicals. These will be assembled into a memorandum that is archived in PDMLink. See sections 4, 10, and 11 of this Test Plan for more detail on specific procurement document controls.
5	Instructions, Procedures, and Drawings	605.4 Development and Support. Activities should be performed in accordance with documented instructions, procedures, or drawings, as directed by the researcher / developer.	This Test Plan and several IWDs contain the instructions and procedures needed for the work. Refer to Section 2.0 of this Test Plan and other content. P315 Conduct of Operations
6	Document Control	606 NQA-1. Requirement 6; Document Control. This element is applicable to R&D activities. As a minimum, laboratory notebooks should be subject to document control procedures. Also, the process for development of intellectual property documentation should be subject to document control.	SD330, PLAN-WXDIV-2142, P1020-2, and P1020-1. In this Test Plan, Laboratory Notebook pages will be copied and attached to the Analytical Reports that are archived in PDMLink.

Attachment C: QUALITY IMPLEMENTATION MATRIX

7	Control of Purchased Materials, Items, and Services	607 NQA-l, Requirement 7; Control of Purchased Materials, Items, and Services This element is applicable to R&D activities. The degree of application should support the desired results of the work, within the specified performance boundaries. The need to ensure conformance with specified requirements depends on the objectives of the work. If the quality of work results depends on the pedigree of materials, items, or services, the work should be planned to include this Requirement.	SD330. In this Test Plan, chemicals will be purchased from Fisher or VWR through GSS. All three vendors are on the IESL list. Chemicals will be purchased with Certificates of Analysis.
8	Identification and Control of Items	608 NQA-1, Requirement 8; Identification of Control Items. This element is applicable to R&D activities. The degree of application should support the desired results of the work, within the specified performance boundaries. If the quality of work results depends on the pedigree of materials or items (e.g., analytical chemistry), this Requirement applies.	SD330, P301, and PLAN-WXDIV-2142. In this Test Plan, individual items needing specific controls have been identified either specifically or by implication (e.g. the statement that a measurement requires a certain tolerance). For those items, either S&CL control is required or the use of an internal standard to verify operation is used. See sections 4, 10, and 11 of this Test Plan for more detail on specific item control.
9	Control of Processes	609 NQA-l, Requirement 9; Control of Processes, 609.1 General. The control of processes varies considerably as one advances from basic research through development. 609.4 Development and Support. Process control during this phase is formalized. Formalization occurs at the project or program level. Work processes and supporting activities are defined, and work and operating procedures are developed and implemented with respect to safety considerations, quality, cost, schedule, and programmatic mission. Methods of implementation and training requirements are formally defined.	SD330, P301, and PLAN-WXDIV-2142, and Documents referenced in the Test Plan that control work process development at the division level.
10	Inspection	610.1 General. Basic and applied research activities are not amenable to inspection, Consideration may be given to performing inspection-like activities on basic and applied research to establish process or product control limits. 610.4 Development and Support. The researcher/ developer	Inspection of received items is carried out by the receiver checking to ensure that the lot number of the received item matches the lot number on the Certificate of Analysis. Inspection of instruments includes verifying that the internal standards are showing expected results. These activities are

Attachment C: QUALITY IMPLEMENTATION MATRIX

		should anticipate the need and plan for inspection criteria for advanced development work to interface with design process needs.	described in the Test Plan.
11	Test Control	611.1 General. Test control does not apply uniformly to basic and applied research. Where applicable, test methods and characteristics shall be documented and the approaches and procedures recorded. Test control does not apply to basic and applied research activities in which hypotheses are being evaluated. It does apply to support activities associated with the conduct of research. 611.4 Development and Support. Characteristics to be tested and test methods should be specified. The test results should be documented and their conformance to acceptance criteria evaluated. Tests required should be planned, executed, documented, and evaluated.	The specific test methods and outputs are documented above along with descriptions of the evidence used to ensure that they are conforming to expected performance. This Test Plan constitutes the planning of the tests. Test results will be documented in Analytical Reports that are archived in PDMLink. See sections 9 and 10 of this Test Plan for details on test control.
12	Control of Measuring and Test Equipment	612.1 General. The researcher should specify the requirements of accuracy, precision, and repeatability of measuring and test equipment (M&TE). These requirements have different implications for basic, applied, and development work. 612.4 Development and Support. During the process development stage and for all R&D support activities, M&TE should be controlled. The degree of control should be dependent on the application of the measurement.	Specific items needing S&CL calibration are called out in the test plan either specifically or through implication by statement of a required tolerance. Items not called out in those fashions are controlled through the use of internal standards that verify their operation.
13	Handling, Storage, and Shipping	613 Handling. Storage And Shipping. This element is applicable to R&D activities. Good laboratory practices may be defined as instructions used for conducting the activity.	P301 and P101-14 apply. In addition, “handling” in performance of this R&D work is addressed by SD601, Conduct of R&D, the content of this test plan, including Integrated Work Documents (IWDs) incorporated by reference.

Attachment C: QUALITY IMPLEMENTATION MATRIX

14	Inspection, Test, and Operating Status	614.1 General. This criterion has limited applicability for R&D activities. 614.4 Development and Support. The status of items and processes for which inspections and tests are specified, should be identified by tags, markings, inspection and test records, or other suitable means. The authority for application and removal of inspection and test identification should be specified.	P330-2. Items calibrated by S&CL have visible calibration stickers attached. Any item that is "calibrated" per this Test Plan is understood to be on the S&CL program. Other items noted in this test plan have their operational status verified by the use of internal standards as noted in the text. No user performed calibrations are part of this Test Plan.
15	Control of Nonconforming Items	615 This Requirement should apply only to R&D support activities. The results of R&D activities are not expected to meet predetermined requirements; therefore, obtaining unexpected results does not constitute a nonconforming condition. The point at which a nonconformance can be identified is the point at which development work has transitioned into design or production of engineered items.	Per Part IV, Subpart 4.2, para 103.4, this applies to calibrated items. If calibrated items or items checked with internal standards show nonconformances, per this Test Plan, an assessment will be made by the RLM and then, in conjunction with the SME, a path forward will be determined. This may include reformulation and/or retesting. See section 6.0 of this Test Plan for details on nonconforming items.
16	Corrective Action	616.1 General. Conditions adverse to quality can be identified for R&D activities, depending on the certainty of operating assumptions and expected results. The documentation, reporting, and tracking of conditions adverse to quality is done at the discretion of the researcher. 616.4 Development and Support. Responsibility should be defined for the identification, cause, and corrective action for significant conditions adverse to quality; these should be documented and reported to appropriate levels of management. Follow-up actions should be taken to verify implementation and effectiveness of corrective action.	Corrective action will apply items as noted above and to the Test Plan and associated documentation. Item nonconformance corrective action is described above and in the Test Plan. Document nonconformance includes everything from simple typographic errors to incorrect process and procedures. Per this Test Plan, non conformances that do not affect the purpose or scope may be documented in a scientific notebook. Other nonconformances will be documented in an approved revision to the document. This guidance is consistent with the M division Technical Plan and Integrated Work Document policies, AP-WXDIV-2385 and AP-JDIV-1019. See section 6 of this Test Plan for details on Corrective Actions.

Attachment C: QUALITY IMPLEMENTATION MATRIX

17	Quality Assurance Records	617 Quality Assurance Records. This element is applicable to R&D activities. In many cases, the notebook or journal of the researcher is the QA record. Controls are needed for these documents, e.g., maintain copies of critical pages or access-controlled filing when not in use to preserve process repeatability and the QA record. Electronic media may be used to record data and should be subject to appropriate administrative controls for handling and storage of data.	SD 330 and PLAN-WXDIV-2142. Documents will be captured in memoranda or reports that are archived in PDMLink. See sections 7 and 10 of this Test Plan for more detail on records. P1020-1, Laboratory Records Management
19	Audits	618.1 General. Planned requirements are not always defined for R&D work; therefore, audits should be conducted in a graded manner. R&D audit activities include normally accepted assessment practices, peer reviews, or both. 618.4 Development and Support. Responsibility should be defined for audits and the results of these audits should be documented and reported to appropriate levels of management. Follow-up actions should be taken to verify implementation and effectiveness of corrective action.	Section 5.0 of this Test Plan guides the usage of surveillances. Surveillances may include table top and walk down reviews of documents and tasks prior to start of work and during actual execution. Surveillances will be carried out at the discretion of the QA-SME and coordinated with the Principle Investigator. Due to the limited scope of this plan, surveillances will be performed by Environmental Program deployed QA SMEs utilizing QPA-DO-FSD-007.006 Quality Assurance Surveillances.
*	Software QA	Note: the NQA-1 Subpart 4.2 guidance on R&D does not specifically address the use of Software, however, the DOE QA Order 414.1D and EM QA Program, EM-QA-01 Rev. 1, establish requirements for safety and non-safety software using a graded approach. Established LANL Software QA programs and procedures defining controls for the acquisition, development, and/or use of software should be applied. This includes commercial off-the-shelf (COTS) software used for the control of instrumentation and the recording of data obtained by instrumentation.	SD 330 and PLAN-WXDIV-2142. Software quality will be documented in division implemented SQM forms. All software is COTS and is standard software used in many different places. See sections 4 and 11 of this Test Plan for details on Software QA.

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

Appendix 2

Data Report for the Drum-scale Thermal Transport Characterization

LA-UR-16-20004
Approved for public release; distribution is unlimited.

Title: Data Report for the Drum-scale Thermal Transport Characterization Study

Author(s): Parker, Gary Robert Jr.
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Holmes, Matthew David

Intended for: Report

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LA-UR-16-20004

Data Report for the Drum-scale Thermal Transport Characterization Study

Gary Parker, Eric Heatwole & Matt Holmes
WX-6, HE Thermal and Mechanical Response Team

1.0 Summary

In accordance with document PLAN-WXDIV-2406(U), Rev. A "*Test Plan for the Drum-scale Thermal Transport Characterization Study*", four 55-gallon drums, each containing different representative remediated nitrate salt (RNS) surrogate mixtures, were instrumented and monitored while exposed to both cooling and re-warming environments. The objective for this project was to determine the thermal transport rates for the waste drums in a temperature controlled environment.

The study was initiated on December 19, 2014 and completed on January 12, 2015. Work was performed at TA-54, Area L, in a freezer unit located outside Building 39. A complete set of data was collected as prescribed in the plan. Data quality was high and the objectives were met. The purpose of this report is to present and summarize these data.

2.0 Objectives

The objectives of this test were:

- Collect quality thermal data (using a calibrated and certified data collection system) while test drums were cooled from ambient temperature to -10 ˚C.
- Collect quality thermal data while the test drums were re-warmed to 10 ˚C from a uniform and stable initial temperature state below -10 ˚C.
- Determine the duration required to cool and re-warm the drum filled with the lowest bulk thermal diffusivity mixture.

3.0 Test Description

3.1 Drum Fills

Four standard steel 55-gallon drums were filled with mixtures of Swheat™ (organic, wheat-based kitty litter) and SafeStep™ Enviro-Blend Power 6300 rock salt (Fig. 1). The mixtures were loaded inside a plastic bag and cardboard drum liner system (Fig. 2) inside the drums. The ratio of Swheat to salt was either 3:1 or 1:1. Additionally, water was added (6.25 % by volume) to some to produce what were

referred to as the "wet" mixtures, while other mixtures without added water were referred to as "dry". The fill details of the drums can be found in Table 1.

Figure 1. A mixture of Swheat™ kitty litter and SafeStep™ Enviro-Blend Power 6300 rock salt used in this study.

Figure 2. The four drums, nested in the SWB and filled with the kitty litter and rock salt mixture. The plastic bag and cardboard liner system is visible.

Table 1.

TC #	Cal. Lab. Cert. File #	Position	Drum #	Drum fill (Swheat:salt, dry or wet)	Nominal insertion depth (in)	Actual depth from top of lid (in)	Temp. offset from TC extension cable (˚C)	Cumulative error (˚C)
1	102507	Int. top	1	3:1 dry	12	11.00	1.16 ±0.08	± 2.21
2	102517	Int. middle	1	3:1 dry	18	16.64	1.08 ±0.06	± 2.21
3	102522	Int. bottom	1	3:1 dry	24	22.96	1.07 ±0.05	± 2.21
4	102508	Int. top	2	3:1 wet	12	10.93	0.99 ±0.03	± 2.21
5	102520	Int. bottom	2	3:1 wet	24	22.93	0.96 ±0.04	± 2.21
6	102513	Int. middle	2	3:1 wet	18	16.63	1.03 ±0.03	± 2.21
7	102509	Int. top	3	1:1 dry	12	10.93	0.93 ±0.03	± 2.21
8	102518	Int. middle	3	1:1 dry	18	16.64	0.98 ±0.04	± 2.21
9	102519	Int. bottom	3	1:1 dry	24	22.94	0.93 ±0.03	± 2.21
10	102510	Int. top	4	1:1 wet	12	10.92	0.91 ±0.05	± 2.21
11	102512	Int. middle	4	1:1 wet	18	16.62	0.94 ±0.04	± 2.21
12	102521	Int. bottom	4	1:1 wet	24	22.93	0.94 ±0.06	± 2.21
13	102621	Ext. middle	1	3:1 dry	-	17.25	1.11 ±0.06	± 2.21
14	102622	Ext. middle	2	3:1 wet	-	17.25	1.07 ±0.05	± 2.21
15	102623	Ext. middle	3	1:1 dry	-	17.25	1.07 ±0.05	± 2.21
16*	102624 102627	Ext. middle	4	1:1 wet	-	17.25	1.01 ±0.04	± 2.21
17	102625	SWB lid, top center	-	-	-	-	0.71 ±0.06	± 2.21
18	102516	Freezer environ.	-	-	-	-	0.66 ±0.06	± 2.21

*During drum de-nesting on 1/6/15, The original thermocouple (File #102624) in this position was broken and replaced with another of the same type (File #102627).

3.2 Instrumentation

Once filled, the drums were closed and thermocouple probes were inserted through pass-through fittings in the lids to varying depths (Fig. 3). Additionally, a thermocouple was attached the external surface of each drum at mid-height. The locations of the thermocouples are shown in Figure 4 with measured insertion depths recorded in Table 1. All thermocouples were calibrated and certified by the

LANL Calibration Laboratory. The calibration reports can be accessed using the "Cal. Lab. Cert. File #" recorded in Table 1.

Figure 3. A closed drum with 3 thermocouple probes inserted through the lid and 1 thermocouple attached with orange tape on the external surface.

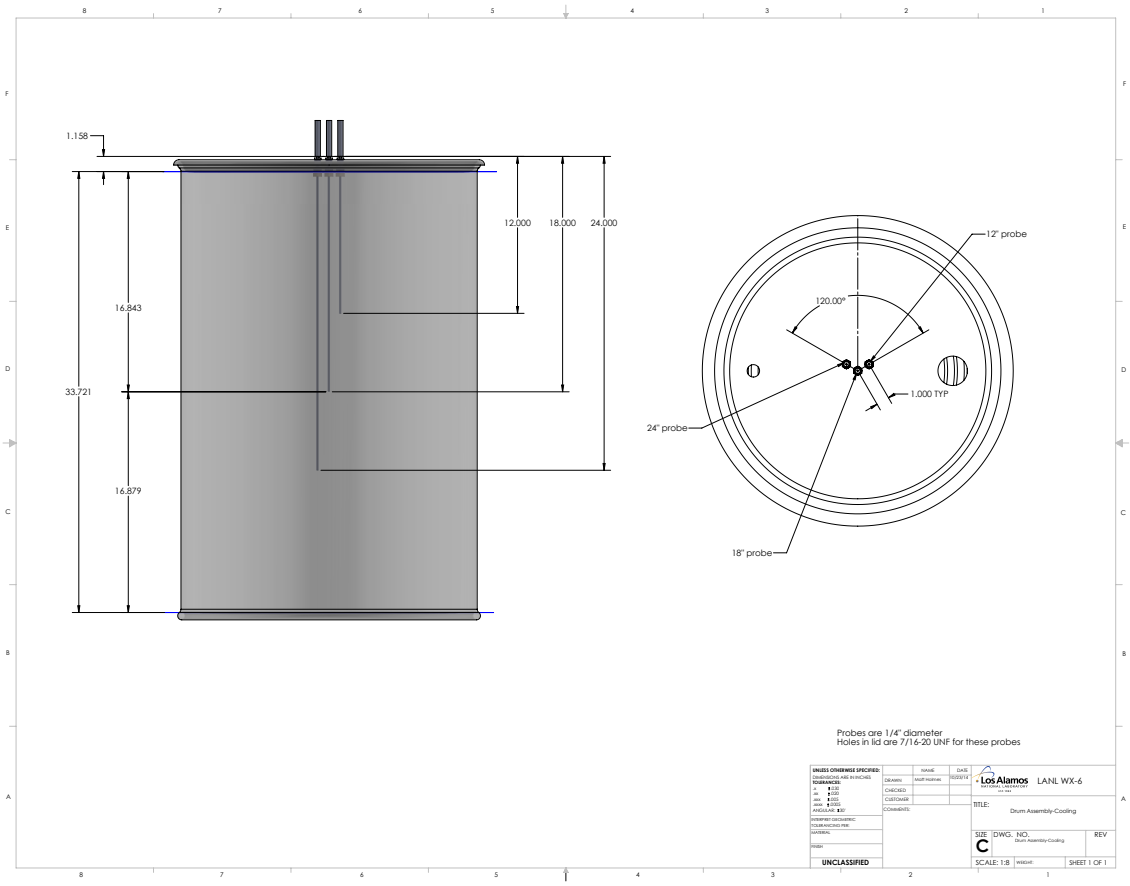

Figure 4. Location diagram of internal thermocouple probes.

The four drums were placed inside a SWB container with lid. Four holes were cut in the SWB lid to allow clearance and access to the thermocouple leads (Fig. 5). The SWB container was then placed inside a walk-in freezer that provided the environmental temperature control for both the cool-down and re-warm phases of this study (Fig. 6). A thermocouple was attached to the top surface, center of the SWB lid. Additionally, a thermocouple probe was located in free-space, mid-height up the SWB, to monitor the air temperature inside the freezer.

Figure 5. The SWB with lid attached.

Figure 6. The SWB placed within the freezer unit. Thermocouple extension cables have been connected.

The thermocouples were connected to a data logger (National Instruments model #cDAQ 9188 outfitted with a model #TB-9214 thermocouple module) located inside TA-54-39 by means of type-K thermocouple extension wire. Because of the voltage drop associated with the employment of extension wire, it was necessary to measure the temperature offset for each channel with a calibrated and certified handheld thermocouple simulator (LANL Cal. Lab. File #102506). Measurements were made with the simulator set at -20, -10, 0 and 10 ˚C. These data were then averaged for each channel to determine the offset and error. The data were corrected by the offset. Cumulative measurement error for each channel in the system arises from the thermocouple unit (±2.2 ˚C), the thermocouple simulator (±0.2 ˚C) and the offset introduced by the wire (error varies). Cumulative error was calculated by the normal method, i.e. the square root of the sum of individual errors squared. Measured offsets and cumulative error can be found in Table 1.

3.3 Thermal Environment Control

The freezer was set to -20 ˚C and temperature was logged until the thermocouples reported a uniform and steady thermal state inside the drums. The next phase began once the freezer was set to 10 ˚C and the temperature was logged until all thermocouples reported ≥8 ˚C. At this point the test was considered complete.

3.4 Timeline

12/19/14, 12:53:35 pm	Started data logging.
12/19/14, 12:59 pm	Freezer doors were closed, cooling phase started.
12/23/14, 1:27 pm	Data logging was paused to save the file. Logging was restarted quickly thereafter.
1/5/15, 1:51 pm	Stopped data logging for the cooling study. Cooling study completed.
1/6/15, 10:18 am	SWB removed from freezer and drums were de-nested.
1/6/15, 10:32 am	De-nested drums were placed inside the freezer. Thermocouple #16 (Cal. Lab. File # 102624) was broken during de-nesting. It was replaced with another thermocouple (Cal. Lab. File #102627).
1/7/15, 10:58 am	Data logging for re-warm phase was started.
1/7/15, 11:08 am	Temperature of freezer was set to 10 ˚C. Re-warming phase started.
1/12/15, 3:10 pm	Data logging for re-warm phase was stopped. Re-warm study was completed.

4.0 Results and Discussion

The data were high quality with neither thermocouple failures, nor unexpected loss of record continuity. Figures 7 and 8 show the complete data sets for the cooling and re-warm phases, respectively. The same data are also displayed in Figures 9-12 grouped by drum number to reduce visual clutter. Qualitatively the thermal response was similar for the four drums. For example, the internal, top thermocouple probes (nominal insertion depth of 12 in.) responded quickly to environmental temperature, whereas the other two internal probes (middle and bottom) tended to respond more slowly. Additionally, the middle and bottom probes tended to track together during the cooling phase. This was not the case during re-warm for the "wet" drums (drums #2 & #4) where an excursion can be seen (Figs. 10 & 12). Hypotheses for this temporary reduction in warming rate include a solid-to-liquid phase transition and/or slumping of material within the drum; unfortunately, there is not enough evidence to explain this response conclusively. The consequence is clear however, especially for drum #4, where the excursion caused the contents to be the slowest to re-warm above the target temperature.

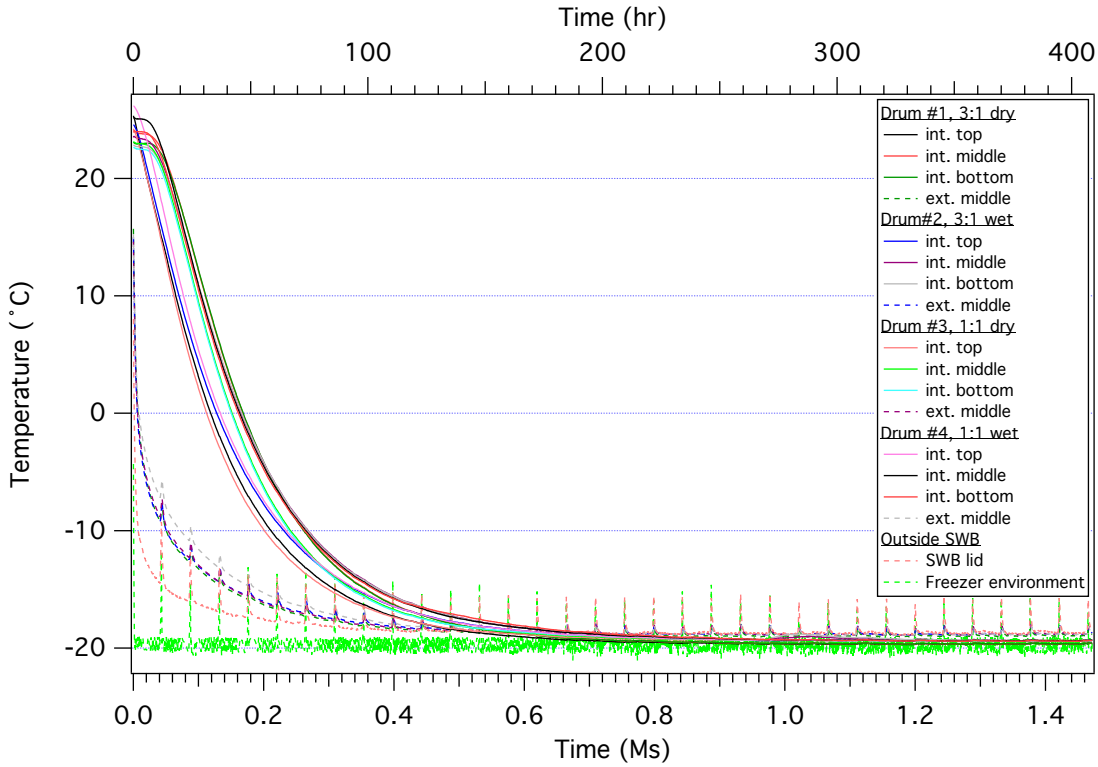

Figure 7. Complete data set for the cooling phase of the project.

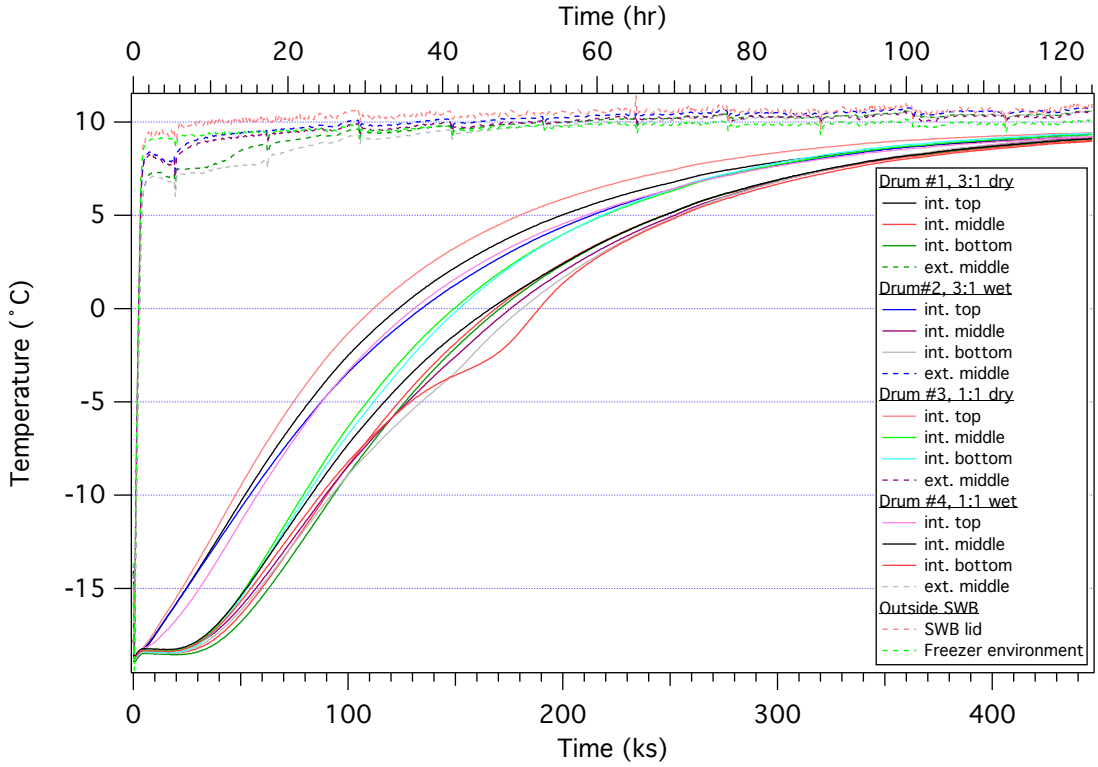

Figure 8. Complete data set for the re-warm phase of the project.

Periodic temperature spikes are evident on the externally located thermocouples. These are the result of a cyclic thaw routine required and pre-programmed by the temperature control hardware on the freezer to prevent buildup of frost from interfering with functioning of the chilling unit. The thermal impulse introduced by these spikes was not sufficiently strong to influence the temperature state at the internal locations as was evidenced by the smoothness of those curves.

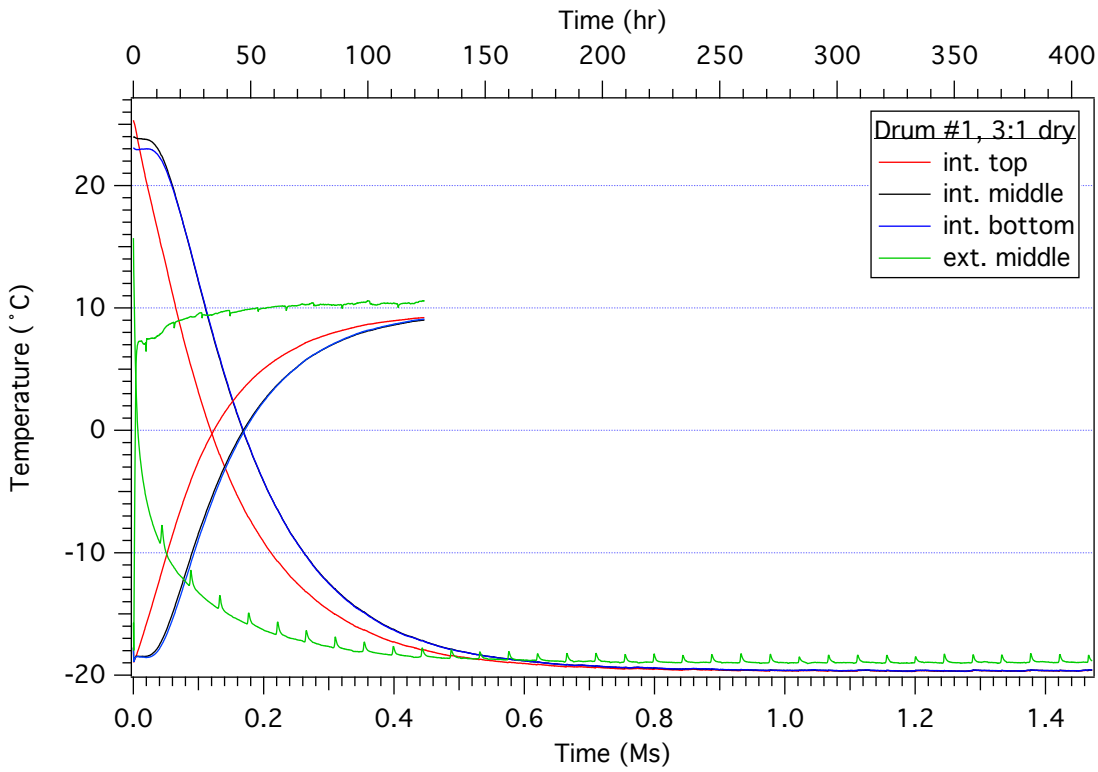

Figure 9. Cooling and warming data for Drum #1.

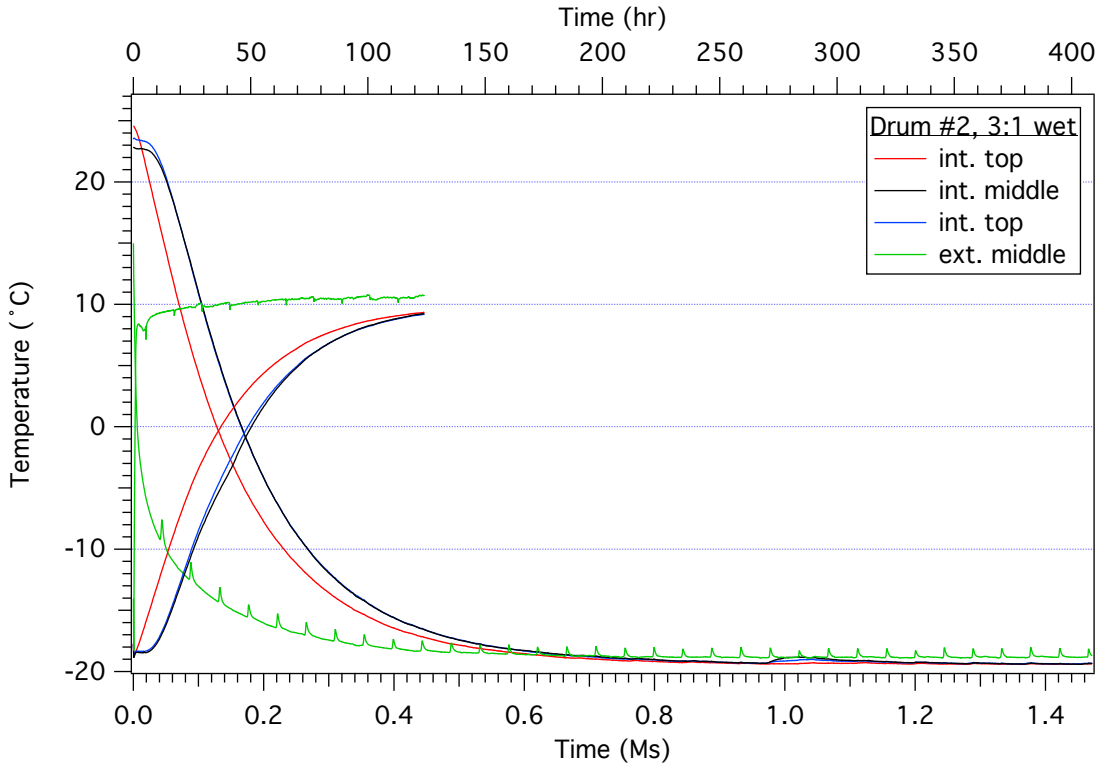

Figure 10. Cooling and warming data for Drum #2.

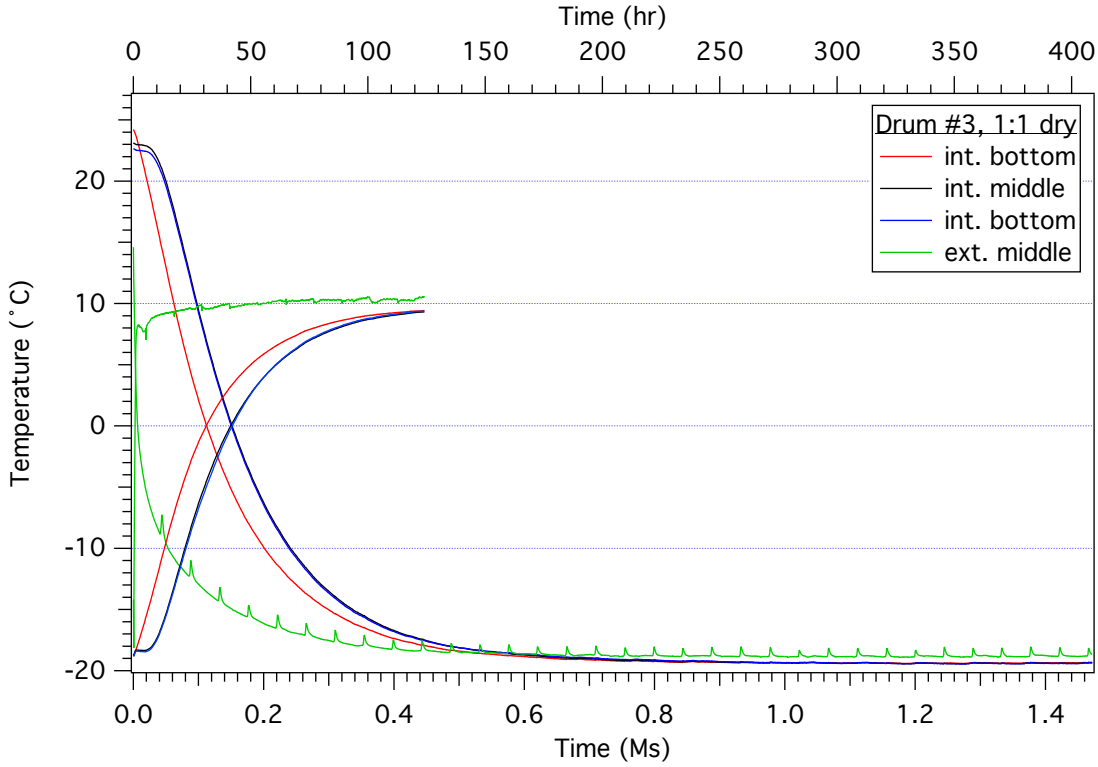

Figure 11. Cooling and warming data for Drum #3.

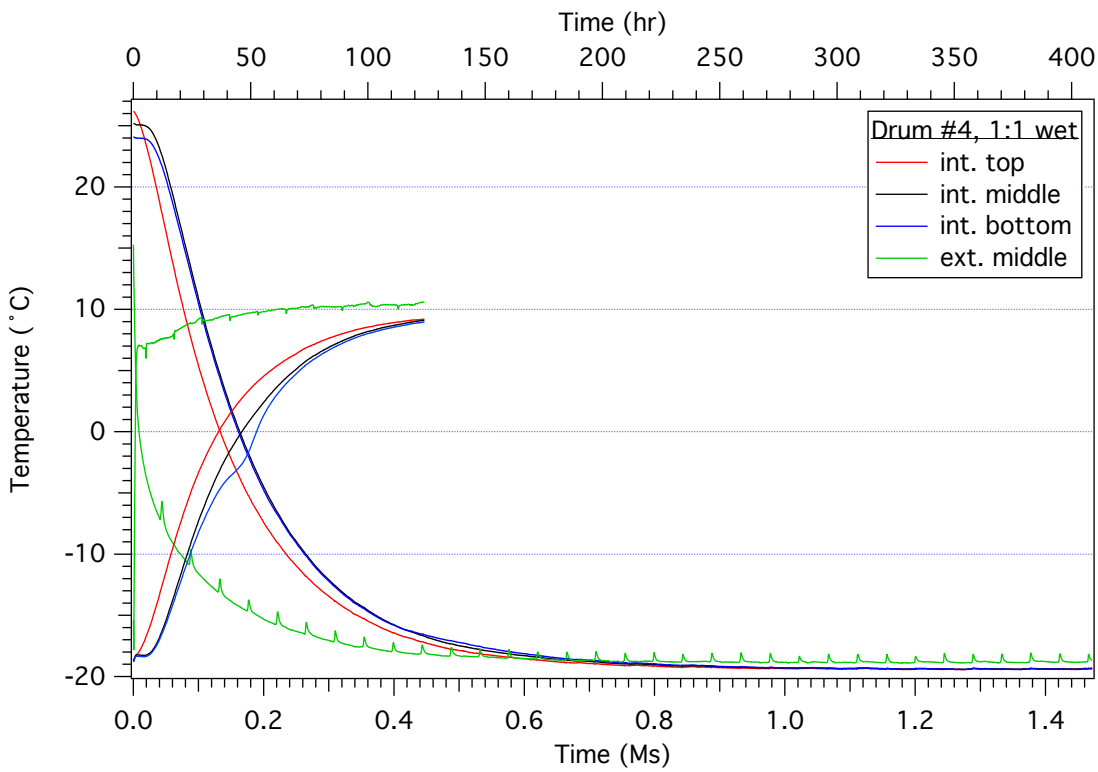

Figure 12. Cooling and warming data for Drum #4.

The objectives of this study were to determine which mixture was the slowest to respond to the imposed environmental temperature conditions and also to determine the duration required to freeze and thaw these contents. The cooling/warming rate will be a function of the bulk thermal diffusivity, α, which is defined as,

$$\alpha = \frac{k}{c_p \rho} \ , \tag{1}$$

where k is thermal conductivity, c_p is specific heat capacity and ρ is density. While bulk thermal diffusivity was not measured directly, the effects of this property were. Figure 13 shows the data scaled to emphasize the salient region of the cooling curve, with error included, for the thermocouple reporting the lowest cooling rate. The contents of drum #2 (3:1 wet mixture) required the greatest duration to reach -10˚C. The location was on the cylindrical axis at approximately mid-height. To illustrate the range of response as a function of fill composition, the thermocouple record for drum #3 (1:1 dry mixture) at the same location was included; this was the quickest-cooling mid-height thermocouple. Note the quickest curve falls within the measurement error of the slowest. The innermost contents of drum #2, with an initial temperature of 22.8 ˚C, required approximately 74 hours to cool to the target temperature. In other words, the 3:1 wet mixture had the lowest bulk thermal diffusivity for this phase of the study.

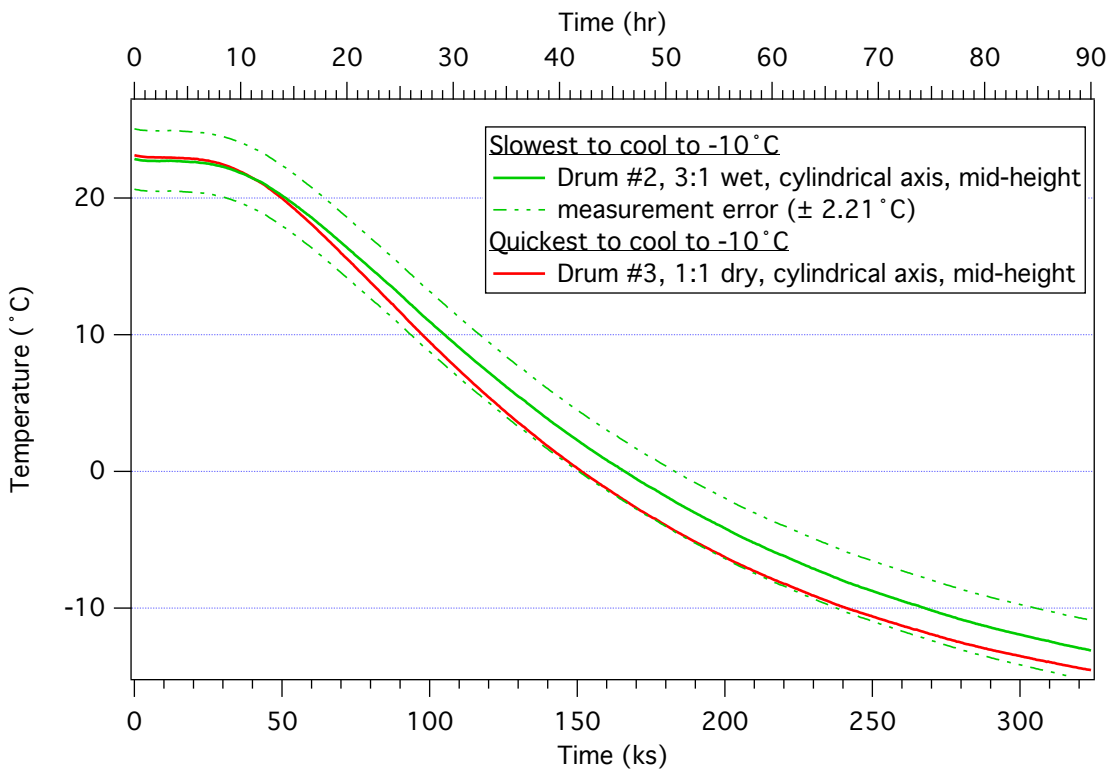

Figure 13. Data from slowest- and quickest-cooling locations.

Figure 14 shows a similar reduction of data emphasizing the slowest and quickest curves during the re-warm phase. Owing to the significant, though temporary, excursion seen on the bottom thermocouple in drum #4, this location is the slowest to reach the target temperature of 8 ˚C. Recall, the slowest location during cooling was in drum #2 at mid-height. Because this location was slow to respond again, and for the sake of continuity, this location record was also included in Figure 14. With the exception of the excursion, these curves track together in late time suggesting the wetted mixtures have similar, and low, bulk thermal diffusivity. Lastly, as was observed during the cooling phase, the thermocouple located in drum #3 at mid-height was the quickest to respond and, with the exception of the period when the excursion occurred, this curve falls within the error bounds of the slowest curve.

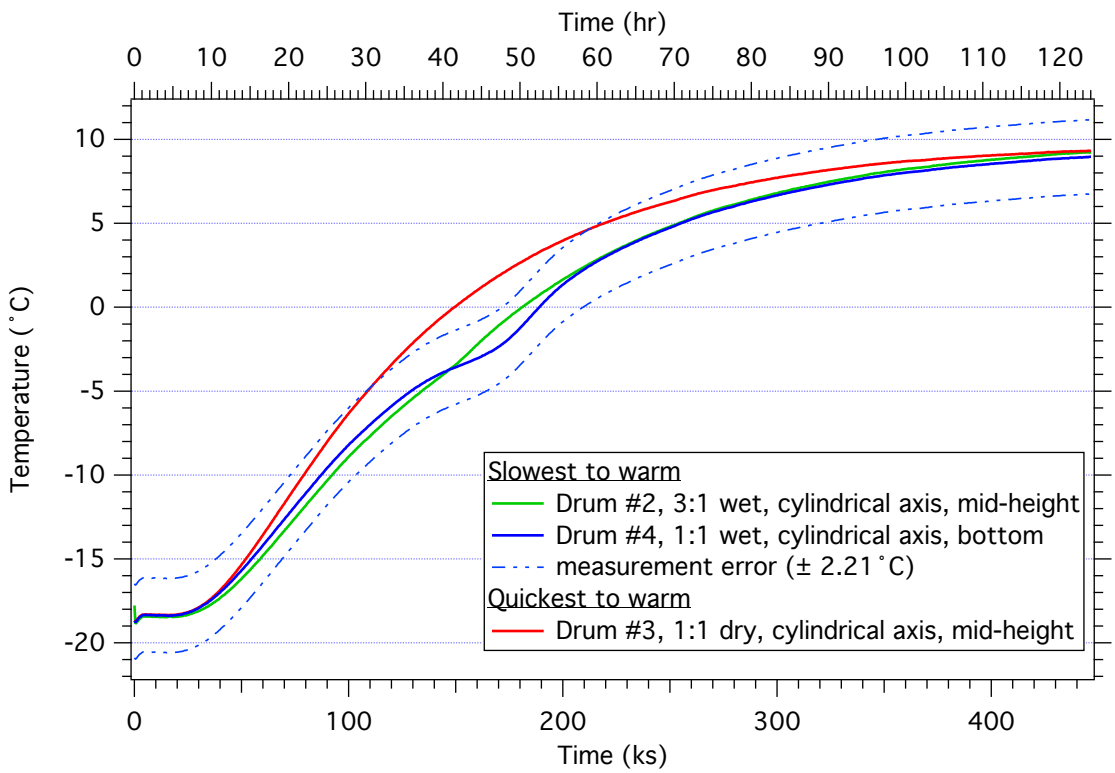

Figure 14. Data from slowest- and quickest-warming locations, including the curve from the mid-height internal probe in drum #2. The thermal excursion is evident between ~40-60 hours.

Owing to the asymptotic approach to the environmental temperature condition, the warming phase was not run for a sufficient duration to reach 10 ˚C. As a result, it is not possible without extrapolation to report the re-warm duration to 10 ˚C. Consequently, a decision was made for this document to report the time required to reach 8 ˚C. This temperature threshold is suitably warm to meet the practical requirements of the re-warm phase, i.e. to warm the frozen mixtures above the freezing point of water and allow the contents of the drum to be easily handled and repackaged. From an initial stable and uniform temperature state of -18.5 ˚C, the slowest responding location required approximately 100 hours to re-warm to 8 ˚C.

5.0 Conclusions

This study successfully adhered to the test plan and met the objectives described therein. It was discovered that the wetted mixtures have the lowest bulk thermal diffusivity and, therefore, require the greatest duration to cool and re-warm.

The durations required for both cooling and warming will depend greatly on the initial temperature, as well as a number of other factors including mixture inhomogeneity, fill volume, convective thermal processes and the presence of exothermic chemical and/or biologically mediated reactions. It is recommended that a numerical model be developed incorporating some or all of these factors to

estimate durations and enhance confidence for determination of safety margins. The data contained in this report would be valuable for validating this model.

6.0 References

PLAN-WXDIV-2406(U), Rev. A "*Test Plan for the Drum-scale Thermal Transport Characterization Study*", 2014.

Appendix 3

Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory

LA-UR-15-27180
Approved for public release; distribution is unlimited.

Title: Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory

Author(s): Robinson, Bruce Alan
Stevens, Patrice Ann

Intended for: Report

Issued: 2015-12-17 (rev.1)

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LA-UR-15-27180

Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory

Rev. 1

Bruce A. Robinson
Patrice A. Stevens

Los Alamos National Laboratory

December 16, 2015

LA-UR-15-27180

Executive Summary

This report documents the methodology used to select a method of treatment for the remediated nitrate salt (RNS) and unremediated nitrate salt (UNS) waste containers at Los Alamos National Laboratory (LANL). The method selected should treat the containerized waste in a manner that renders the waste safe and suitable for transport and final disposal in the Waste Isolation Pilot Plant (WIPP) repository, under specifications listed in the WIPP Waste Acceptance Criteria (DOE/CBFO, 2013). LANL recognizes that the results must be thoroughly vetted with the New Mexico Environment Department (NMED) and that a modification to the LANL Hazardous Waste Facility Permit is a necessary step before implementation of this or any treatment option. Likewise, facility readiness and safety basis approvals must be received from the Department of Energy (DOE). This report presents LANL's preferred option, and the documentation of the process for reaching the recommended treatment option for RNS and UNS waste, and is presented for consideration by NMED and DOE.

After the release of radioactivity from the WIPP on February 14, 2014 and the subsequent recognition that the breached drum was a RNS waste drum processed at LANL (Drum 68660), LANL took a number of precautionary steps to protect workers, the public, and the environment. Drums stored at LANL continue to be maintained in isolated storage. Monitoring results are reported to the NMED under the LANL Nitrate Salt Bearing Waste Container Isolation Plan (Isolation Plan: LANL, 2014). Drums are currently stored under a High Efficiency Particulate Air filtration system and the temperature controls provided by the building, with active fire suppression systems. Monitoring of the drums consists of hourly visual inspections, daily temperature measurements of the standard waste boxes (SWBs) containing the RNS waste drums, and periodic sampling and analysis of the headspace gases within these SWBs. This configuration of the RNS and UNS wastes at LANL represents the "initial state" for subsequent treatment options being considered in this Options Assessment report. The report describes the methods used to evaluate a wide range of potential treatment options to permanently treat the waste, and presents the results of that evaluation.

The scientific underpinning for this assessment is the work of Clark and Funk (2015), which reports the comprehensive set of studies undertaken by LANL to gain an understanding of the chemical reactivity that led to the exothermic reactions and breech of the drum in WIPP. Experimental and modeling studies performed at LANL indicate that mixtures of metal nitrate salts (oxidizer) with Swheat™ organic kitty litter (fuel) create the potential for exothermic chemical reactions. The use of Swheat™ absorbent in the processing of nitrate salt wastes can be pinpointed as the critical processing decision that led to the failure of Drum 68660 in the WIPP repository. Based on their studies, Clark and Funk (2015) proposed a remediation strategy consisting of two steps: 1) cooling of the waste drums during handling to lower the rates of reactions that may be occurring; and 2) stabilizing the waste by mixing the RNS waste into an inorganic matrix of natural mineral zeolite like clinoptilolite to deactivate RCRA characteristics (D001/D002).

To evaluate this recommendation as well as other potential treatment options, LANL assembled a team (the "Core Remediation Team" or "Core Team") consisting of subject matter experts across a wide range of disciplines including scientific, operational, safety and regulatory specialists. The team's goal was to increase the number and diversity of options beyond that considered by Clark and Funk (2015), and to subject those options to an evaluation process that considers a broad set of evaluation criteria, thereby ensuring a more robust, defensible treatment recommendation. Four treatment options previously considered by LANL staff were originally included. These involved zeolite addition, cementation, or both. An additional LANL option was later evaluated including dissolution of the nitrate salts, filtration of the mixture, and final cementation. As part of this study, the Core Team expanded the list of treatment options beyond RCRA stabilization to include nine other general or industry-practice-based technologies recommended in the Resource Conservation and Recovery Act (RCRA) treatment standards (40 CFR Part 268). The full list of treatment options considered is shown in Table ES-1.

A diverse set of eleven criteria was defined to ensure that a broad set of factors was considered in evaluating these options. A twelfth criterion, cost, was also considered for information purposes but not explicitly used in the evaluation. The evaluation process consisted of two steps. First, a pre-screening process was conducted to cull the list on the basis of a decision of infeasibility of certain potential options with respect to one or more of the criteria. Then, the remaining potential options were evaluated and ranked against each of the criteria in a relative fashion, and numerical scores were established by consensus of the review Core Team (with a range of 1 to 5, with higher scores being more favorable). After the ranking process was completed for all criteria and a matrix of scores was determined, the final results were tabulated and the discussion and rationale for the scores was documented. The main report provides definitions of the treatment options and criteria, and narratives explaining the Core Team's rationale for the pre-screening decisions and the justification for the scores awarded for each options against each criteria.

The final results of the evaluation are summarized in Table ES-2. In the pre-screening step, a total of fourteen options were considered. Four RCRA stabilization options were identified using zeolite, zeolite with cementation, and dry-process or wet-process cementation (Options 1 through 4). A fifth stabilization option of combined technologies, filtration and dissolution with cementation of the nitrate salt waste (Option 14), was evaluated as a treatment option, after the initial meeting of the remediation team. All other options were eliminated in this step and screened out. After the determination of the screening, the eliminated options were not ranked. Clearly, this result applies only for the particular nitrate salt waste streams at LANL, and is not a general conclusion. Difficulties in permitting, safety basis, and short-term or long-term effectiveness of the final waste form were typical criteria that led to the elimination of these options. In the subsequent full evaluation of the five stabilization options, Option 1 (Stabilization Using Zeolite) ranked the highest based on the criteria used in the evaluation. Its score is significantly higher than any cementation option; for most of the eleven criteria applied to the evaluation, this option scored equal to or higher than any of the cementation options. Therefore, even if one were to apply unequal weightings to the various criteria, the conclusion that zeolite

addition is the preferred option will not change. Therefore, the recommendation to pursue Option 1 is very robust. The results of the cost criterion, though not used in the analysis, reinforces the results of the evaluation in that the treatment option recommended based on non-monetary criteria is also judged to be the most cost effective option.

Finally, recommendations were developed based on current information and understanding of the scientific, technical, and regulatory situation at the time of writing of this document. Any significant changes to the state of knowledge in any of these areas should be followed up with a qualitative re-evaluation, or a more thorough quantitative evaluation, as appropriate.

Table ES- 1. Summary of potential treatment options considered

Option	Description	Applicability		EPA Technology Code*
		RNS	UNS	
RCRA Stabilization Options				
1. Stabilization Using Zeolite	Mix waste into inorganic natural mineral to eliminate ignitability potential of the waste	X	X	STABL /RHETL
2. Stabilization Using Zeolite With Cementation	Option 1 followed by production of cement waste form	X	X	STABL /RHETL
3. Stabilization Using Dry-Process Cementation	Production of cement waste form with water added only at the time of cementation	X	X	STABL
4. Stabilization Using Wet-Process Cementation	Initial water addition to eliminate potential thermal runaway reactions, followed by production of cement waste form	X		STABL/WTTRx
14. Salt Dissolution With Cementation/ Stabilization	Water addition followed by filtration and cementation process of Swheat™ cake and nitrate salt solution	X		WTRRx/STABL/ RHETL
Other RCRA Recommended Options				
5. Incineration	Burning of waste in a radiological incinerator	X		INCIN
6. Thermal Oxidation of Organics	Treatment of waste in air to oxidize without flame	X		RTHRM
7. Biodegradation	Biological breakdown of organics or non-metallic inorganics under aerobic or anaerobic conditions	X		BIODG
8. Chemical or Electrolytic Oxidation	Breakdown of organics through the addition of oxidation reagents	X		CHOxD
9. Chemical Reduction	Breakdown of nitrate constituents through the addition of reducing reagents	X	X	CHRED
10. Vitrification	Incorporation of waste into a glass waste form	X	X	HLVIT
11. Alternate Macro-Encapsulation	Coating of the waste with an organic polymer to reduce surface exposure	X	X	MACRO
12. Neutralization	Reagent addition to neutralize the pH	X	X	NEUTR
13. Controlled Reaction or Leaching	Removal of soluble salts by leaching with water	X	X	

* EPA Technology Code derived from 40 CFR 268.42.

Table ES-2. Summary of results of the evaluation of treatment options

EVALUATION CRITERIA / POTENTIAL TREATMENT OPTIONS	Robust to waste stream variability	Ease of permitting (permitting difficulties)	Safety basis challenges	Extent of testing required	Reduction of toxicity and mobility	Reduction in volume	Short-term and long-term effectiveness	WCS implications	Scalability and complexity	Facilities challenges	Schedule	Cost (not a primary evaluation criterion) *	SCORE
1 Stabilization Using Zeolite (remediated)	5	3	4	5	4	2	4	2	4	4	4	4	41
Stabilization Using Zeolite (unremediated)	5	3	4	5	4	2	5	N/A	5	5	5	5	43
2 Stabilization Using Zeolite With Cementation (remediated)	5	2	3	3	4	1	4	1	2	2	1	1	28
Stabilization Using Zeolite With Cementation (unremediated)	5	2	3	3	4	1	5	N/A	3	3	2	1	31
3 Stabilization Using Dry-Process Cementation (remediated)	5	2	2	3	4	3	4	1	3	2	2	2	31
Stabilization Using Dry-Process Cementation (unremediated)	5	3	2	3	4	3	5	N/A	4	3	3	3	35
4 Stabilization Using Wet-Process Cementation (remediated)	3	4	1	3	4	2	4	1	1	1	1	2	25
14 Salt Dissolution With Cementation/Stabilization (remediated)	3	3	1	3	4	2	4	1	2	1	2	2	26
5 Incineration													
6 Thermal Oxidation of Organics													
7 Biodegradation													
8 Chemical or Electrolytic Oxidation													
9 Chemical Reduction													
10 Vitrification													
11 Alternate Macro-Encapsulation													
12 Neutralization													
13 Controlled Reaction or Leaching of Reactive Inorganic Chemicals With Water													

Note: Stabilization Options 1-4 and 14 are discussed in Section 4.1 RCRA Stabilization Options. Options developed from RCRA treatment standards are the gray-shaded rows. Red cells denote the screening out of an option based on a high degree of infeasibility with respect to that criterion. Because of the initial screened-out determination, Options 5-13 were not ranked. Discussion of Options 5-13 is found in Section 4.2 Additional RCRA Treatment Options.
*Cost not included in final score.

1 Introduction

On February 14, 2014, a release of radioactivity occurred at the Waste Isolation Pilot Plant (WIPP), resulting in distribution via airborne transport of radioactivity within the repository and to the surrounding environment in the vicinity of the facility. Subsequently, WIPP personnel gained access to the underground and determined that a waste drum or drums had breached in Panel 7, Room 7 of WIPP. After WIPP declared a potentially inadequate safety analysis (PISA) on the possibility of inadequately remediated nitrate salt-bearing waste contained in waste packages at WIPP (May 1, 2014), LANL took precautionary measures to move all remediated nitrate salt (RNS) waste drums to TA-54, Area G, Dome 375 and began daily temperature measurements.

When definitive photographic evidence became available (May 15, 2014) that the breeched drum was indeed an RNS waste drum processed at LANL (Drum 68660), LANL implemented additional precautions and controls, including overpacking of the 55-gallon RNS waste drums into Standard Waste Boxes (SWBs)[1], as well as moving all unremediated nitrate salt (UNS) containers[2] to a Permacon at TA-54, Area G, in Dome 375. As of August 2015, the UNS waste drums were moved to the general population located in Dome 230. RNS waste drums similar to those at LANL had previously been shipped to WIPP (515 drums,[3] emplaced in the WIPP underground), and to the low level radioactive waste facility in Andrews, Texas managed by Waste Control Specialists, LLC (WCS) (115 drums, subsequently placed in shallow underground storage with temperature monitoring). Thus, LANL, WIPP, and WCS have taken precautions to protect workers, the public, and the environment from further reactions.

In a series of subsequent actions, LANL took the following steps associated with the UNS and RNS waste drums:

- Environmental Protection Agency (EPA) Hazardous Waste Number D002 (corrosivity) was conservatively applied to 26 of the UNS containers due to the presence of free liquids,[4]

[1] On May 18, 2014, there were 57 RNS waste containers at LANL, overpacked into a total of 55 SWBs. Four additional containers were pipe overpack containers. The resulting final number of RNS containers was 61 as of June 30, 2015. An August 27, 2015 update reflected 56 RNS waste containers remained in 54 SWBs. The remaining four pipe overpack containers were each stored in an 85-gallon overpack.
[2] At the time that LANL suspended further processing of UNS waste on May 2, 2014, there were a total of 29 UNS waste drums that had not yet been processed. The movement of these drums to Dome 375 was completed on June 3, 2014. These drums were moved to Dome 230 with the general waste population in August 2015.
[3] Nitrate Salt Bearing Waste Container Inventory March 27, 2015 (ADESH-15-052) and April 24, 2015 (ADESH-15-071).
[4] The waste drums are lined with epoxy to minimize corrosion. LANL took the conservative approach and designated the drums as D002 in July 2014.

- EPA Hazardous Waste Number D001 (ignitability) was applied to all UNS waste containers based on the presence of nitrate salt compounds,

- EPA Hazardous Waste Number D001 (ignitability) was applied to all RNS waste containers. This step was taken based on independent testing using surrogate samples comprised of mixtures of the organic absorbent (Swheat™ kitty litter) and sodium nitrate indicating that the remediated nitrate salts are considered to be oxidizers under Department of Transportation rules; and

- EPA Hazardous Waste Number D003 (reactivity) was not initially applied to the RNS waste containers. The oxidizer basis for applying the D001 EPA Hazardous Waste Number (ignitability) was deemed sufficient to characterize the waste because it was the primary constituent and regulatory basis for the characterization (40 CFR §261.21(a)(4)); a thermal reaction would be the most probable source for a reactivity determination; there were relevant and applicable testing procedures available; the oxidizer characterization was rebuttable by testing under DOT regulations at 49 CFR §173.127; and the waste would be managed with all special requirements for both ignitable and reactive waste.

Drums at LANL continue to be managed and monitoring results are reported to the NMED under the requirements of the LANL Nitrate Salt Bearing Waste Container Isolation Plan (Isolation Plan: LANL, 2014). Drums are currently stored under HEPA filtration and the temperature controls provided by the buildings, with active fire suppression systems. Monitoring of the drums consists of hourly visual inspections, daily temperature measurements of the SWBs containing the RNS waste drums, and periodic sampling and analysis of the headspace gases within these SWBs. This configuration of the RNS and UNS wastes at LANL, and the hazardous waste designators applied to the drums represent the "initial state" for subsequent treatment options being considered in this Options Assessment Report.

This report documents the methodology used to select a method to treat the RNS and UNS waste in a manner that renders them safe and suitable for transport and final disposal in the WIPP repository, under specifications listed in the WIPP Waste Acceptance Criteria (WAC) (DOE/CBFO, 2013). Furthermore, on December 6, 2014, the NMED issued an Administrative Compliance Order (ACO: NMED, 2014) to DOE and LANS[5] for violations to LANL's Hazardous Waste Facility Permit (Permit) connected to the management of nitrate salt wastes. The pertinent portions of the ACO relevant to

[5] As of the writing of this report, negotiations are ongoing and the ACO has not been finalized.

this report are the following compliance actions pending NMED issuance of the ACO actions.[6]

> *130. No later than 60 days after this order becomes final, Respondents shall submit to NMED for review and approval a plan to remediate and/or treat the 57 remediated daughter containers pursuant to all applicable HWMR and Permit requirements.*
>
> *131. No later than 60 days after this order becomes final, Respondents shall submit to NMED for review and approval a plan to remediate and/or treat the 29 un-remediated parent containers pursuant to all applicable HWMR and Permit requirements.*

To comply with these actions, documents are being provided to the NMED to provide the technical and other justification for the proposed treatment plans that LANL proposes. Figure 1-1 is a schematic diagram representing the feeds and information content of the various documents comprising the overall plans. Documentation of LANL's scientific work consists of a series of scientific investigations feeding the summary report of Clark and Funk (2015). This collection of reports provides the technical underpinning for the remaining documents. The Options Assessment Report (this document) provides the rationale for LANL's recommendation of the treatment options for RNS and UNS wastes, including a description of the process used to arrive at the recommendation. Finally, the Remediation Plans for RNS and UNS wastes will establish the recommended path forward for final treatment of the waste streams. These plans translate the Options Assessment Report recommendation and the LANL facility-based requirements to resume safe operations (the Resumption Plan) into an actionable plan for treatment to render the nitrate salt wastes safe for transportation and final disposal in the WIPP repository. The scientific studies, the Options Assessment Report, and the Remediation Plans collectively serve to satisfy the ACO deliverables previously cited.

The remainder of this Options Assessment Report consists of a brief summary of the scientific findings relevant to the future treatment of UNS and RNS waste and a discussion of assumptions. The report describes the potential treatment options that were considered for RNS and UNS wastes including the methodology used to arrive at the recommended treatment options. The methodology was an expert-based process in which a cross-disciplinary team of LANL professionals established a set of evaluation criteria and ranked the various proposed options. Finally, the results of this process are presented, and specific recommendations for remediation of RNS and UNS wastes are summarized.

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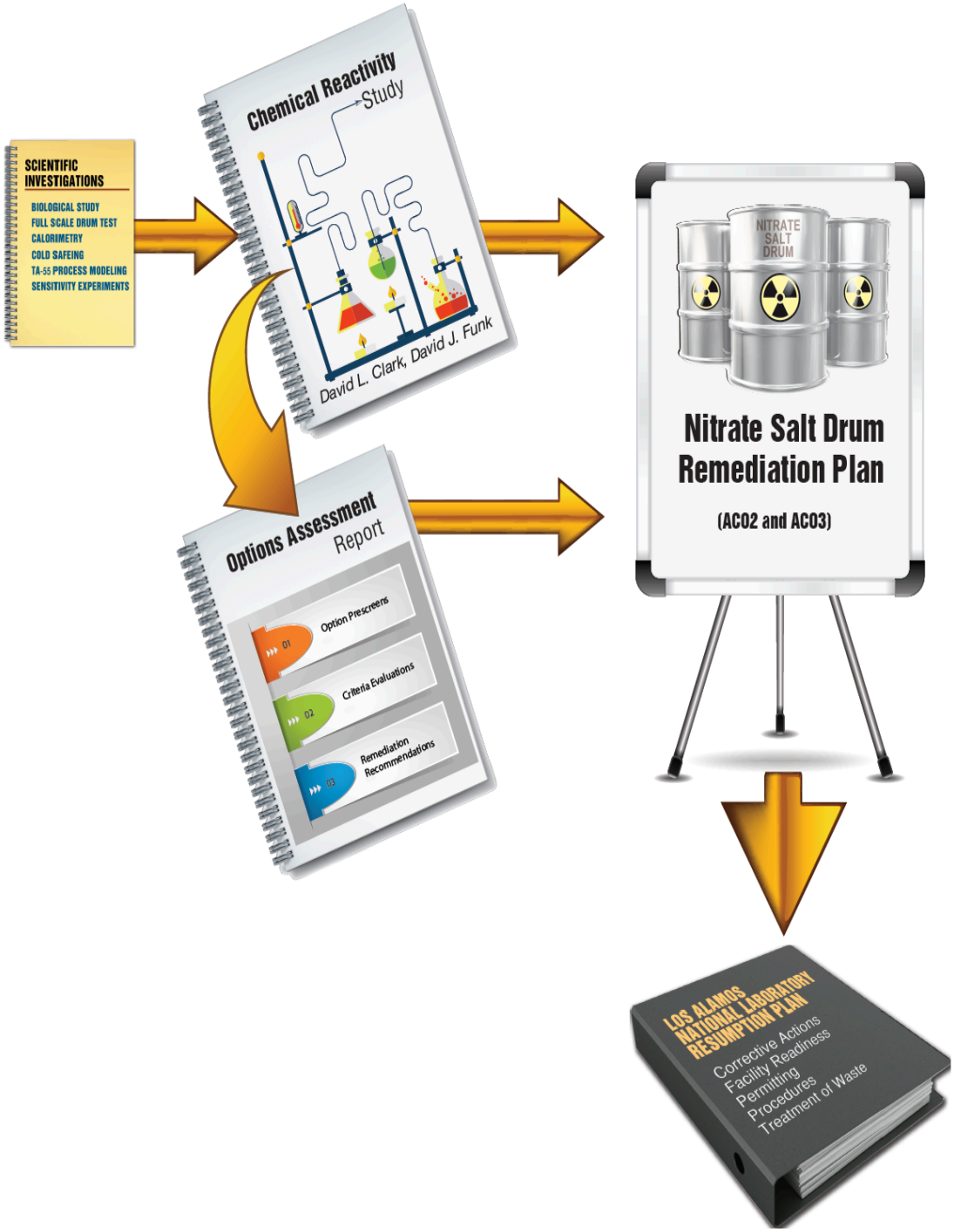
[6] From NMED, 2014. HWMR refers to the Hazardous Waste Management Regulations, 20.4.1 NMAC, and "Permit" refers to the LANL Treatment, Storage, and Disposal Facility (TSDF) Permit, EPA I.D. Number NM0890010515-TSDF.

Figure 1-1 Schematic diagram describes the documentation elements associated with the Administrative Compliance Order deliverables for treatment of nitrate salt waste



2 Summary of Scientific Findings on RNS Waste

This section provides a brief summary of the findings of LANL scientists with respect to the energetic reaction that occurred in RNS waste drum 68660 in the WIPP repository, leading to the breach of that drum. It is provided to set the stage for subsequent evaluation of treatment options. This description is derived from the report of Clark and Funk (2015): refer to that report for details.

This section is divided into two parts, a summary of the technical understanding of the chemical reactivity in the RNS waste drums, followed by the remediation strategy recommended in the Clark and Funk study on the basis of this understanding.

2.1 Chemical Reactivity of RNS Waste

Experimental and modeling studies performed at LANL indicate that mixtures of metal nitrate salts (oxidizer) with Swheat™ organic kitty litter (fuel) create the potential for exothermic chemical reactions. The use of Swheat™ absorbent in the processing of nitrate salt wastes can be pinpointed as the critical processing decision that led to the failure of Drum 68660 in the WIPP repository, regardless of the details of the thermal processes that enabled the drum to achieve temperatures sufficient to initiate the chemical reactions. Evaluation of the characteristics of the failed drum, coupled with extensive chemical testing indicate that, in addition to the nitrate salt/Swheat™ organic kitty litter mixture, an additional trigger mechanism (or mechanisms) is likely required to raise the internal drum temperature high enough to initiate the nitrate salt/Swheat™ organic kitty litter reaction.

A combination of chemical conditions were identified that may lower the temperature for reaction, including initial high acid concentration of free liquids; significant quantities (> 1 gal) of neutralized, absorbed free liquids; the presence of reactive or catalytic metals like magnesium, iron, or lead; the presence of bismuth containing glovebox gloves; and the presence of natural biological activity. Complex surrogate nitrate salt mixtures prepared to simulate wastes, particularly those containing iron and magnesium, can generate NO_x gases that partially nitrate the organic Swheat™ kitty litter and form a more energetic fuel, i.e., triethylaminenitrate (TEAN). These complex surrogate salt mixtures display exothermic behavior at temperatures *as low* as 60 °C (140 °F) which is still well above the ambient temperature conditions experienced by a drum.[7]

Neutralization of free liquids and sorption onto Swheat™ establishes conditions (moisture with near-neutral pH) that will support natural biological activity. Spontaneous self-heating generated by low-level chemical reactions and/or the

[7] The lower bound is dependent upon total mass. The lower bound is a complicated thermal transfer problem and dependent upon volume and configuration.

respiration of bacteria, molds, and microorganisms is potentially important in the early stages and may be sufficient to raise the temperature as high as 60 °C (140 °F), where the other exothermic chemical reactions can take place. Additional studies are being conducted to evaluate the role biological activity may have played in initiating the event. Planning for these studies is ongoing, and is anticipated to require long-duration experiments due to the nature of the evolution of biological processes under these conditions.

From the combined results of literature studies, modeling, and experiments amassed to date, one can arrive at a plausible scenario in which a production of heat, either from low-level chemical reactions or the growth of natural microbes, in concert with mixed metal nitrate salts, bismuth lined glovebox gloves and/or lead nitrates when combined with the Swheat™ organic kitty litter, generated a stepwise series of exothermic reactions that heated and pressurized the drum resulting in the venting of high-temperature gases and radioactive material into the room.

It is likely that a specific set of conditions is required to trigger the suite of reactions that has to date led to thermal runaway in just one drum, to the best of the technical experts' knowledge. However, the complexity of the mixtures, ambiguity in procedures such as those used for neutralization, the heterogeneity of the drum contents, and the difficulty of sampling leads to an irreducible level of uncertainty that mandates the exercise of caution in managing RNS wastes. Even though drums being monitored at LANL have not exhibited any observable thermal excursions, analyses of samples of the headspace gases within the SWBs containing RNS waste are consistent with the presence of oxidation reactions or microbial activity (Leibman et al., 2015). There is evidence that the UNS drums are outgassing H_2. For the RNS drums, the headspace gases are being monitored and sampled. Thus, the organic-oxidizer combination is inherently a thermally sensitive mixture actively exhibiting the RCRA characteristic of ignitability (D001). Finally, recent studies with the most reactive surrogates developed to study the hazards indicate some sensitivity to electrostatic discharge (ESD), which mandates additional study to ensure that waste handling and processing procedures appropriately account for this possibility.

This situation requires that the RNS waste stream continue to be monitored and that safety precautions be taken during continued storage and ultimately during treatment. By contrast, the UNS waste stream does not possess these same hazards (Funk, 2014), but the fact that the waste is a RCRA characteristic ignitable (D001) due to it being an oxidizer requires that the UNS waste will undergo normal WIPP certification process which includes treatment prior to transportation and disposal at WIPP.

2.2 Clark and Funk Recommendations on Cooling and Treatment of RNS Waste

On the basis of the scientific understanding gained from their study, Clark and Funk (2015) provided a technical recommendation for rendering the RNS waste safe for subsequent treatment. Their recommended two-step process is:

1. *Cool the RNS waste drums.* Cooling the waste is a safety measure to be performed in advance of removing the waste from its current configuration in order to sample and subsequently process the solids. Cooling drums to -10 °C or lower will slow down both chemical and biological reactions. Drums can then be warmed back to +10 °C, a value that is 50 °C below the onset temperature of exothermic reactions, consistent with chemical industry safety guidelines for process operating conditions for exothermic reactions.[8,9]

The UNS sampling must appropriately bound the waste in the RNS drums. Currently planned strategies for the RNS waste treatment plans indicate that the treatment success demonstration will involve testing the "treated" surrogate waste rather than the RNS waste to avoid the Safety Basis complication (i.e., difficult or unsafe to sample radioactive waste on-site). This includes chemical constituents and physical properties (e.g. particle size and surface area, which would have strong effects on ignitability and burn rate) and to ensure the mixture is not ignitable or corrosive after treatment without affecting the Safety Basis. The treatment plan demonstrates that the physical properties impacting D001/D002 characteristics are modified by stabilization process – such that measuring and testing UNS waste is sufficient to define the characterization and treatment testing. The treatment plan ensures species, characteristics and/or properties are measured during and after processing to ensure stabilization of the waste and debris prior to WIPP certification and disposal. If the validation sample comes back with a negative result, then further remediation is necessary.

2. *Mix the RNS waste into an inorganic matrix of natural mineral zeolite like clinoptilolite.* Adding zeolite to the RNS and UNS waste containers is a potential process to remove the RCRA hazardous waste characteristic (D001, ignitability) from the waste in the containers that prevents them from meeting the WIPP WAC. Determining the capability of the zeolite to meet this condition and the quantity of zeolite used will need to be determined through treatment studies which will subject surrogate waste samples to a variety of EPA-specified tests for ignitability and oxidizer potential (SW-846, EPA, 2007). If for some reason, natural zeolites are found to be undesirable, then grout is an acceptable alternative with the important caveat that following water addition to make grout, the wetted nitrate salt/Swheat™ organic kitty litter mixture should be processed directly into concrete.[10]

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[8] Center for Chemical Process Safety "Guidelines for Chemical Reactivity Evaluation and Application to Process Design," AIChE, New York, NY, 1995.

[9] Pressure is very important to achieving thermal runaway. A filter block may occur due to ice particle buildup. If the filter is moist at the time of cooling, ice can block the filter decreasing the amount of gas flow capacity. This safety measure must also be investigated.

[10] Results of oxidizing solids testing EMRTC Report FR 10-13 conclusively demonstrates that either zeolites (36 wt.%) or grout (55 wt.%) in proper ratios deactivate D001 characteristics per EPA SW-846, Method 1040.

3 Assessment Assumptions

This section establishes the underpinning assumptions that the team formed to perform the evaluation used in its deliberations on treatment options for the RNS and UNS waste streams. These are the "boundary conditions" that are important to consider when assessing the viability of different options.

All options require continued management of waste in its current configuration or in a configuration that ensures safety during storage. Studies continue to be conducted to understand the factors that led to the breach of drum 68660 in the WIPP repository. Continued safe management of the waste will consist of control of the environmental conditions around the drums, such as temperature, and the continued application of engineering controls under an approved Container Isolation Plan. Temperature control is also a necessary precursor to denesting[11] and handling of the waste (Clark and Funk, 2015). Processing of the waste under an approved Permit modification will enable it to be removed from the Isolation Plan. This assumption applies equally to all proposed treatment options, and impacts all treatment options equally.

Surrogate wastes developed from UNS sampling will be representative of the RNS waste stream. Development of an effective treatment option for the unique RNS waste stream requires that surrogates of the waste be used for product testing to ensure that the ignitibility characteristic has been mitigated in the final waste form.

Only the RNS waste contains a combination of fuel and oxidizer such that a significant energetic reaction can occur. While latent chemical reactions may exist in the UNS, they are not sufficient to cause a large release of heat. Some of the drums that have a decomposition of the salts are endothermic and release gas but not heat. Due to the obstacles in sampling the contents of the RNS drums at the present time, surrogate mixture compositions and samples of Swheat™/salt mixtures starting with UNS waste will be developed that are bounding and represent samples of the actual RNS drum compositions. Surrogate wastes developed from UNS sampling will be representative of the RNS waste stream.

To ensure that estimates of the contents of the drums are appropriately bounded by these mixtures and to demonstrate RCRA treatment success, confirmatory sampling and analysis of UNS and RNS wastes must be performed during the treatment process. This assumption applies equally to all proposed treatment options, and impacts all treatment options equally.

The selected treatment option will be conducted only after re-establishment of facility readiness, implementation of required corrective actions, and regulatory approval of modifications to the LANL Permit. This assumption applies equally to all proposed treatment options, but some options may make it easier or more difficult to fulfill the

[11] Denesting is the removal of the waste drums from the overpack for sampling and then stabilization.

requirements. Several criteria used to evaluate options allow for discrimination between options on the basis of relative ease to obtain these approvals.

Waste will be treated in a manner that leads to safe onsite storage of the treated waste, followed by shipment to and disposal in the WIPP repository. This assumption applies equally to all proposed treatment options, but some options may be technically straightforward, technically challenging, or even infeasible.

Several criteria used to evaluate options allow for discrimination between options or screening out of some options on the basis of the ease or difficulty of producing a waste form that meets the WIPP WAC (DOE/CBFO, 2013).

To ensure the remediation plan is adequate and to address the similarity between the RCRA characteristic of ignitability (D001) and reactivity (D003)[12] for the RNS waste, LANL will demonstrate that neither the ignitability nor the reactivity characteristic are present after the selected treatment process for UNS and RNS waste.

A modification of the LANL Hazardous Waste Facility Permit would be required in order to commence with treatment of the nitrate salt wastes. The options being considered must result in deactivation to remove the EPA Hazardous Waste Numbers of D001, ignitability, and D002, corrosivity, for the nitrate salt waste. This will need to be demonstrated, with a technical basis and data, in the permit modification request (application to NMED) for the process to ensure the remediation plan is adequate. LANL will also conservatively demonstrate that the reactivity characteristic is removed with the selected treatment process for UNS and RNS waste as discussed above.

The permit modifications will also take into account the definition of related waste streams and their corresponding characteristics to ensure the permit properly describes the wastes generated, stored, and treated at LANL.

[12] The UNS waste must meet 40 CFR 261.23 criterion for evaluation of the characteristic of reactivity to ensure remediation plan is adequate: *(6) It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.*

4 Treatment Methods Evaluated

This section describes the full suite of potential treatment options considered for both the RNS and UNS wastes. After the breeched drum in WIPP was revealed to be a RNS waste drum generated at LANL, staff began a process to develop a series of options based on current waste management practices and considering the availability of LANL facilities to conduct the work. From this initial work, four RCRA stabilization options were identified involving zeolite addition, zeolite addition with cementation, and wet or dry cementation. Later, when the Core Remediation Team was established (see Section 5.1), the team decided to expand the list and subject the options to a screening process to ensure the broadest possible consideration of options. To do this, a range of general or industry-practice-based technologies recommended in the RCRA treatment standards (40 CFR Part 268) were included that appeared to be applicable. Nine additional options were added as a result. A fifth stabilization option of combined technologies, filtration and dissolution with cementation of the nitrate salt waste (Option 14), was evaluated as a treatment option after the initial meeting of the remediation team.

By nature of the way these options were developed, the five RCRA stabilization options are more developed than the other nine RCRA treatment standards. The five stabilization options are presented in summary form in Section 4.1 (and in greater detail in Appendix 1), after which the nine other treatment options are described in Section 4.2. Table 4-1 is a summary of all of the treatment options considered, and indicates whether the option is applicable to the RNS waste, the UNS waste, debris, or any combination. It should be noted that the "best" option for each stream might be different.

4.1 RCRA Stabilization Options

The five RCRA stabilization options are described in summary form below, and a more complete presentation is provided in Appendix 1. Four of the RCRA stabilization options were proposed by LANL staff in the initial months after the WIPP release, and took into account scientific and technical considerations as well as facility and waste specific issues, given that the work is to be performed at LANL. Salt Dissolution With Cementation/ Stabilization was later added to the option investigation process. Once the preliminary studies of surrogate samples conclude, the five RCRA stabilization options will be revisited to ensure each option is viable. Note that if one of these processes is implemented, additional optimization would take place, and the details might change. However, the descriptions represent the basis that the Core Team used in its evaluation.

A comparison of the process steps for the five stabilization options is presented schematically in Figure 4-1. As stated in the assumptions section (Section 3) and as pointed out by Clark and Funk (2015), the RNS waste must be under temperature control during handling until steps are taken to mitigate the potential for reaction. This is indicated in the figure with the light blue frame labeled "Temperature Control." These controls can be removed once the possibility of runaway reactions is eliminated. Also indicated on Figure 4-1 are the estimated number of daughter drums and the estimated duration required to

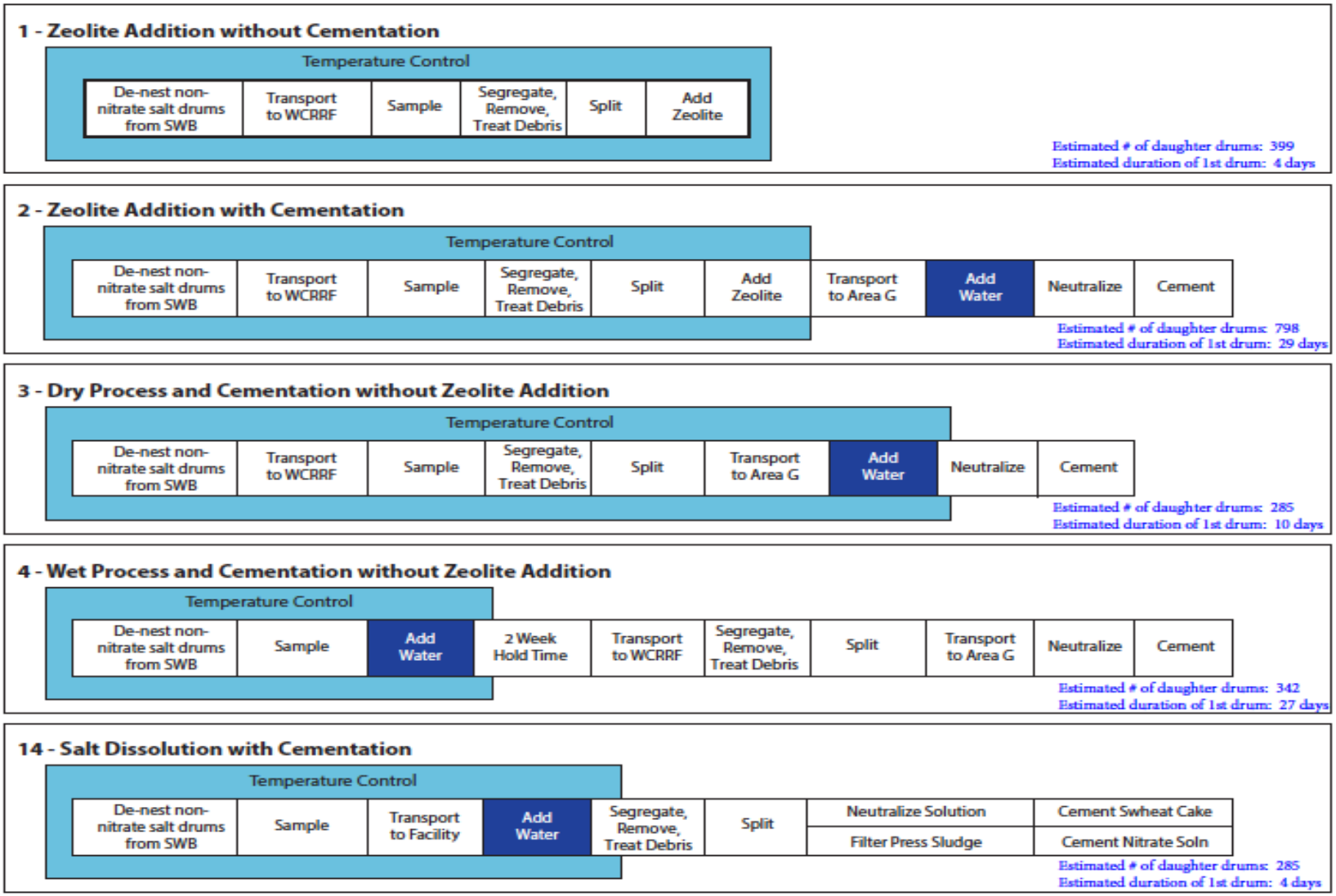
generate the first drum (an indication of complexity and duration of the process). For details, see Appendix 1.

Table 4-1. Summary of potential treatment options considered

Option	Description	Applicability		EPA Technology Code*
		RNS	UNS	
RCRA Stabilization Options				
1. Stabilization Using Zeolite	Mix waste into inorganic natural mineral to eliminate ignitability potential of the waste	X	X	STABL /RHETL
2. Stabilization Using Zeolite With Cementation	Option 1, followed by production of cement waste form	X	X	STABL /RHETL
3. Stabilization Using Dry-Process Cementation	Production of cement waste form with water added only at the time of cementation	X	X	STABL
4. Stabilization Using Wet-Process Cementation	Initial water addition to eliminate potential thermal runaway reactions, followed by production of cement waste form	X		STABL/WTTRx
14. Salt Dissolution With Cementation/ Stabilization	Water addition followed by filtration and cementation process of Swheat™ cake and nitrate salt solution	X		WTRRx/STABL/ RHETL
Other RCRA Recommended Options				
5. Incineration	Burning of waste in a radiological incinerator	X		INCIN
6. Thermal Oxidation of Organics	Treatment of waste in air to oxidize without flame	X		RTHRM
7. Biodegradation	Biological breakdown of organics or non-metallic inorganics under aerobic or anaerobic conditions	X		BIODG
8. Chemical or Electrolytic Oxidation	Breakdown of organics through the addition of oxidation reagents	X		CHOxD
9. Chemical Reduction	Breakdown of nitrate constituents through the addition of reducing reagents	X	X	CHRED
10. Vitrification	Incorporation of waste into a glass waste form	X	X	HLVIT
11. Alternate Macro-Encapsulation	Coating of the waste with an organic polymer to reduce surface exposure	X	X	MACRO
12. Neutralization	Reagent addition to neutralize the pH	X	X	NEUTR
13. Controlled Reaction or Leaching	Removal of soluble salts by leaching with water	X	X	

* EPA Technology Code derived from 40 CFR 268.42.

Figure 4-1. Summary of stabilization treatment options



Note for Option 14: The number of daughter drums is dependent upon the repulp options chosen. The no repulp option would produce 4 daughter drums of cemented waste and another partial drum of debris (plastic bags and liner material) from the orignal drum. If the repulp option is chosen, 6 drums per waste would be generated, resulting in 342 drums.

Option 1. Stabilization Using Zeolite

Waste is processed by removing debris and processing it separately. Following removal of the debris, an inorganic matrix of natural mineral zeolite such as clinoptilolite is added to the RNS. The resulting mixture will not be corrosive, ignitable, self-heating, or an oxidizer. The quantity of zeolite used would be determined through treatability studies using surrogate mixtures of waste, and confirmed once the waste is sampled. To do this, surrogates of the waste would be used for product testing to ensure that the corrosivity and ignitibility characteristics have been mitigated in the final waste form by subjecting treated waste samples to a variety of EPA-specified tests for corrosivity, ignitability, and oxidizer potential. Surrogate samples of Swheat™/salt mixtures would be prepared based on the analysis of the UNS waste representative of the actual RNS drum compositions. Corrosivity will be addressed through absorption of the liquid medium by the zeolite addition. The zeolite will also reduce the potential for thermal runaway and render the mixture safe by creating a thermal barrier. Zeolite, being a desiccant, separates the waste components, reduces the potential for chemical kinetics and acts as a physical and thermal barrier against reactions.[13] The debris separated from the original RNS waste stream is not expected to have the D001 designation because the percent of residual reactive material is small and will be confirmed by visual inspection to determine the degree of contamination of the debris.

For RNS waste, the drums will be processed at temperatures below ambient in order to reduce chemical reaction risk during denesting and slow chemical kinetics potential, and to allow for safe and efficient denesting and handling. Denesting would occur at Area G, and the waste would be transported to the Waste Characterization, Reduction and Repackaging Facility (WCRRF) for processing. For UNS waste, similar processing would be conducted, but temperature control is not required because the nitrate salts without organic absorbent do not pose a safety hazard from oxidation reactions involving contents within the drum (Funk, 2014). The zeolite remains in the mixture and ultimately reaches physical and chemical equilibrium. Cooling does not affect the amount of water the zeolite absorbs.

Option 2. Stabilization Using Zeolite With Cementation

Waste is processed identically to Option 1 up to and including zeolite addition, at which point the ignitability and corrosivity characteristics of the waste is mitigated. The material is then further treated through a process that includes water addition, additional neutralization as needed, and cementation to produce monoliths that would be suitable for transportation and disposal. Waste transport occurs from Area G to WCRRF for zeolite addition, and, in the process evaluated here, back to Area G for cementation. UNS waste, similar processing will be conducted, but without temperature control. As with Option 1,

[13] Semisolids must pass the paint filter test to be considered non-wastewater and solid. Under environmental temperatures, LANL experimentalists attempted to inflame a zeolite added surrogate mixture with a 1000 °F torch and were unable to ignite the mixture.

surrogate testing would be performed to ensure that the corrosivity and ignitability characteristics are mitigated for the final waste form.

Option 3. Stabilization Using Dry-Process Cementation

Waste is moved to WCRRF and processed by removing debris from the RNS waste and processed separately in smaller quantities suitable for subsequent treatment. Following the removal of the debris, the RNS waste is split into smaller quantities suitable for subsequent treatment. The waste is transported as a dry material to a cementation unit (assumed to be a new facility at Area G) where it is processed through the addition of water, neutralization, and cementation to produce monoliths that would be suitable for transportation and disposal. The addition of water to nitrate salts is an endothermic reaction. Additional cooling will not be necessary to prevent uncontrolled reactions. Thus, the temperature controls are removed at the point at which water is added. As with Option 1, surrogate testing would be performed to ensure that the corrosivity and ignitability characteristics are mitigated for the final waste form.

Option 4. Stabilization Using Wet-Process Cementation

Waste is processed by cementation at Area G, but with water addition early in the process, minimizing the flammability risk for the waste and eliminating the immediate hazard. During the full-scale drum test, it was verified that the wetted Swheat did not ignite. At that point, temperature control is removed. The waste is then transported wet to WCRRF for segregation and splitting followed by transportation of daughter drums back to Area G to a new cementation unit where it is processed by neutralization and cementation to produce monoliths that would be suitable for transportation and disposal. Because the early addition of water is a safeing[14] strategy designed specifically for the RNS waste and thus is unnecessary for UNS waste, this option is only applicable for RNS waste. As with Option 1, surrogate testing would be performed to ensure that the corrosivity and ignitability characteristics are mitigated for the final waste form.

Option 14. Salt Dissolution with Cementation/Stabilization

The salt dissolution with cementation process for RNS waste consists of waste repulped in water. Repulp is the size reduction of a slurry to decrease viscosity. The nitrates (potassium and sodium) are highly soluble. For RNS waste, the drums will be processed at temperatures below ambient in order to reduce chemical reaction risk during denesting and slow chemical kinetics potential, and to allow for safe and efficient denesting and handling. Denesting would occur at Area G. The organic Swheat™ is separated from the mixture by a filtration process. A Swheat™ filter cake product and a salt solution product are recovered in separate drums. The fraction of organics that travel with the dissolved nitrate salts is the fraction of organics that can be dissolved in the water. At this stage of dissolution, TEAN is not found in the filtered cake, but rather in the liquid. Organics once dissolved in water are not combustible. Repulping and filtration of the Swheat™ stream can

[14] Safeing is defined as reducing the probability of a deleterious event to an acceptable level.

achieve improved efficiencies in separation of Swheat™/salt if desired. The Swheat™ is then dissolved using caustic digestion and cemented for final preparation prior to transporting for disposal. The salt solution stream is cemented separately then transported for disposal.

UNS waste can be processed by salt dissolution without Swheat™ processing. Testing would be performed to ensure corrosivity and ignitability characteristics are mitigated for the final waste form. Addition of a base to TEAN will result in triethylamine (TEA) and the nitrate salt of the base. This reduces the chemical reactivity of the system overall. However, the pH of the dissolved nitrate salt must be monitored to ensure a good cement monolith is produced.

4.2 Additional RCRA Treatment Options

These nine recommended RCRA treatment options (40 CFR 268 Appendix 1) are numbered 5-13 since they follow the four RCRA stabilization options. The nine options are described generically below. Some of the options are only applicable to either the RNS or UNS waste, the RNS or UNS waste, but not for all categories of waste. The descriptions below identify those instances.

Option 5. Incineration

The waste is intentionally forced to burn in a radiological incinerator. Treatment is performed in units operated in accordance with the technical operating requirements of 40 CFR Part 264 subpart O, which is, using maximum achievable control technology. Furthermore, this option is not applicable for UNS waste since no organic absorbents are present to oxidize.

Option 6. Thermal Oxidation of Organics

Waste is treated in air under high heat to oxidize fuels without flame. A heating process other than flame incineration is used to treat organic constituents of the waste stream or, secondarily, treat residues from a primary treatment process. This option is not applicable for UNS waste since no organic absorbents are present to oxidize.

Option 7. Biodegradation

Waste is treated via biologic breakdown of organics or non-metallic inorganics (i.e., degradable inorganics that contain the elements of phosphorus, nitrogen, and sulfur) in units operated under either aerobic or anaerobic conditions such that a surrogate compound or indicator parameter has been substantially reduced in concentration in the residuals. Salt tolerant bacteria may be cultivated to eat the organic material. But facilities for this treatment would need to be built. This option is not applicable for UNS waste since no organic absorbents are present to biodegrade.

Option 8. Chemical or Electrolytic Oxidation

The waste is treated to eliminate the organics via chemical or electrolytic oxidation utilizing the following oxidation reagents (or waste reagents) or combinations of reagents: 1) hypochlorite (e.g., bleach), 2) chlorine, 3) chlorine dioxide, 4) ozone or UV light assisted ozone, 5) peroxides, 6) persulfates, 7) perchlorates, 8) permanganates; and/or (9) other oxidizing reagents of equivalent efficiency. Chemical oxidation specifically includes what is commonly referred to as alkaline chlorination. This option is not applicable for UNS waste since no organic absorbents are present to oxidize.

Option 9. Chemical Reduction

The waste is treated to chemically reduce the nitrate constituents utilizing the following reducing reagents (or waste reagents) or combinations of reagents: 1) sulfur dioxide, 2) sodium, potassium, or alkali salts or sulfites, bisulfites, metabisulfites, and polyethylene glycols (e.g., NaPEG and KPEG), 3) sodium hydrosulfide, 4) ferrous salts; and/or 5) other reducing reagents. Nitrates are reduced to N_2 by contacting nitrates with metal to convert nitrates to nitrites. Nitrites are reacted with amide to produce N_2 and $C0_2$. This would be performed in small controlled batches and may concentrate TRU waste. The waste could be effectively reduced.

Option 10. Vitrification

Waste is incorporated into a glass waste form by mixing the waste into molten glass in a melter, after which the mixture is poured and allowed to solidify and cool.

Option 11. Alternate Macro-encapsulation

The surface of the waste is coated with an organic polymer (e.g., resins and plastics) or an inert inorganic matrix to substantially reduce surface exposure to potential leaching media.

Option 12. Neutralization

The waste is neutralized to a pH between 2 and 12.5 by adding acids, bases, or water. Such a treatment is likely to be part of a cementation primary treatment process or if free liquids are encountered during treatment.

Option 13. Controlled Reaction or Leaching of Reactive Inorganic Chemicals with Water

Controlled reactions are conducted with water for highly reactive inorganic or organic chemicals with precautionary controls for protection of workers from potential violent reactions as well as precautionary controls for potential emissions of toxic/ignitable levels of gases released during the reaction. Soluble salts are removed by these reactions. This technology is similar to Option 14, but lacks the subsequent stabilization/solidification steps, which deactivate characteristics D001 and D002.

5 Assessment Methodology

This section outlines the methodology employed to assess the various treatment options for the UNS waste and RNS previously developed in Section 4. First, the scope and makeup of the evaluation team are presented, followed by a general overview of the methodology and the definition of the criteria used for evaluating the options.

5.1 Core Remediation Team

This section describes the scope, activities, and composition of the Nitrate Salt TRU Waste Remediation Team, referred to as the "Core Team" in this document. The Core Team is responsible for developing and executing plans to ensure the safety of the RNS and UNS wastes.

This includes a series of high-level steps.

- Conduct an options analysis (Options Assessment Report) leading to a recommended path or paths to remediation of the nitrate salt TRU containers.

- Ensure that the selected remediation option(s) are comprehensively reviewed, vetted, and documented, including development of a regulatory permitting strategy and schedule to begin nitrate salt remediation.

- Ensure that the approved remediation plan is properly reflected in process flow sheets and operating procedures that account for all regulatory, safety basis, permitting, and waste acceptability issues (future activities and work products of the Core Team).

To ensure that the appropriate expertise was engaged in the process, the Core Team was comprised of staff from many relevant disciplines/organizations.

- Energetic chemistry
- Actinide chemistry
- Waste form expertise
- ADEP operations expertise
- TA-55 waste expertise
- Facility Operations Directorate representative
- Regulatory compliance
- ES&H
- Safety basis
- Representative(s) from LANL Carlsbad Office's Difficult Waste Team

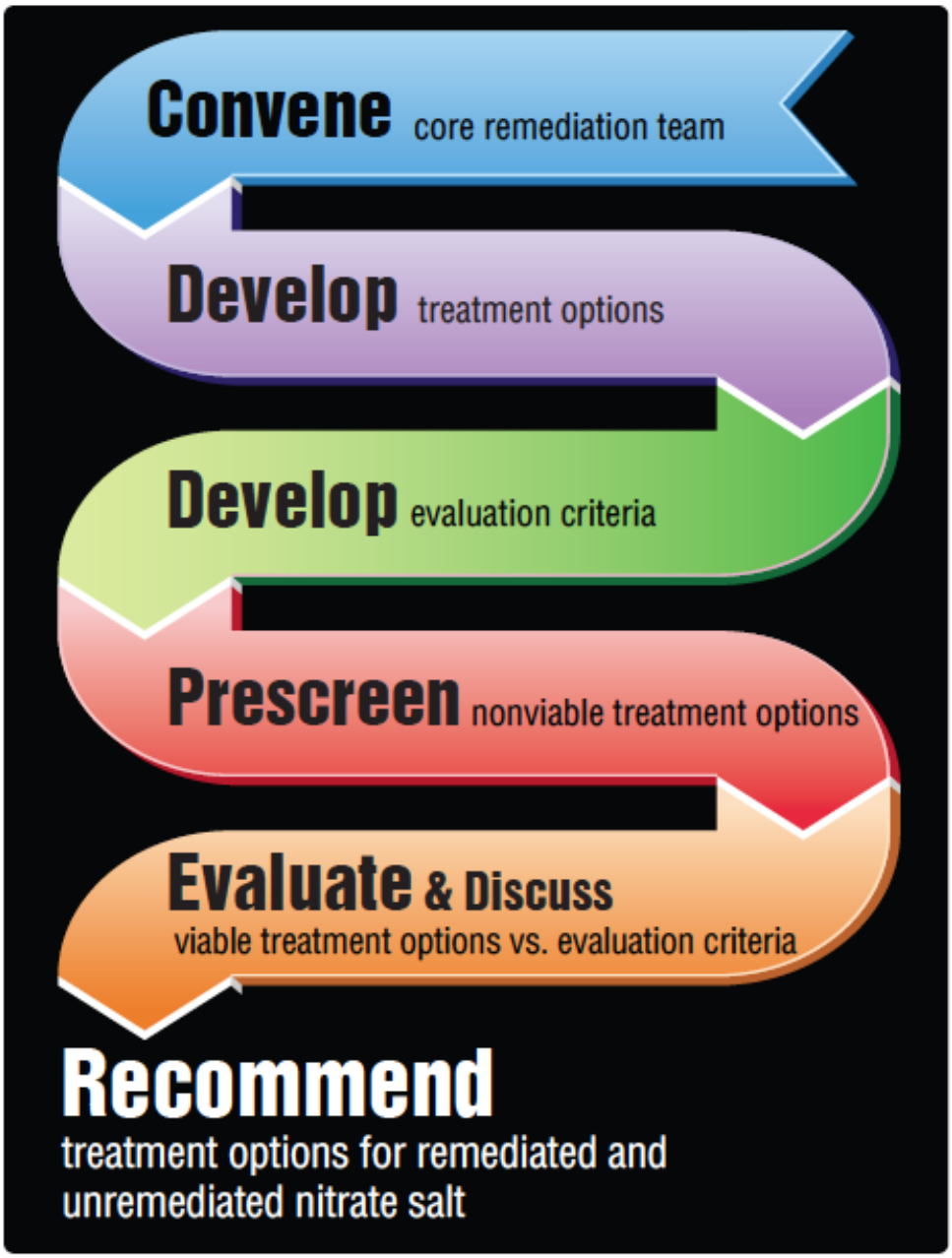
A list of participants in the Core Team as part of this Options Assessment activity is provided in Appendix 2.

5.2 Evaluation Process

The Clark and Funk (2015) report provided a recommendation for a remediation strategy based on the scientific studies and accompanying safety considerations. While from the perspective of science, LANL believes this recommendation to be valid, it was decided to form and engage the Core Team to factor in a broader set of considerations. The Core Team held a series of meetings and performed offline work to develop and run an expert-based process for evaluating and selecting preferred treatment options for the UNS and RNS waste streams and debris streams. An overall map of activities is diagrammed in Figure 5-1, and additional details are provided below.

The first step was to develop a comprehensive list of potential treatment options for consideration. These options were described previously in Section 4. Next, a list of evaluation criteria (see Section 5.3 below) was developed collectively by the Core Team to comprehensively evaluate options against a diverse set of criteria. Then, an initial pre-screening meeting was conducted to cull the list on the basis of a decision of infeasibility of certain potential options with respect to one or more of the criteria. The Core Team discussion was documented to provide the rationale for the screening decisions. The remaining potential options were then evaluated in another meeting of the Core Team. At that meeting, an appropriate member of the Core Team was selected to lead a group discussion for a given criterion. Each option still under consideration was ranked against the criterion in a relative fashion, and numerical scores were then established by consensus. After ranking all criteria, a complete matrix of scores was determined. The final results were tabulated and the discussion and rationale for the scores was documented.

Figure 5-1. Schematic of the process steps used by the Core Team



5.3 Evaluation Criteria

This section provides a list of the criteria that were applied to assess the various treatment options. These criteria were applied in separate evaluations to the February 2014 original number of containers: 57 RNS daughter containers and the 29 UNS parent containers. Since the process required a numerical score to be applied for each treatment option against each criterion, the basis for awarding a particular integer score from 1 to 5 was also defined. A summary of these criteria and scoring range is provided in Table 5-1; full definitions used by the Core Team in its deliberations are provided in Appendix 3.

Table 5-1 List of criteria used to evaluate the potential treatment options

Criterion	**Definition of Minimum Score of 1***	**Definition of Maximum Score of 5**
Robust to Waste Stream Variability	Extremely difficult to develop a robust process	Highly likely to be a robust process
Ease of Permitting (Permitting Difficulties)**	Extremely difficult to permit	Simple permitting process
Safety Basis Challenges	Extremely complex safety basis challenges	Straightforward safety basis approval process
Extent of Testing Required	Very onerous testing required	Straightforward testing required
Reduction of Toxicity, Mobility, Corrosivity, and Ignitability	Marginally effective waste form and/or difficult to package	Highly effective waste form and straightforward to package
Reduction of Volume	Large volume and/or large number of daughters generated	Low volume with low numbers of daughters generated
Short Term and Long Term Effectiveness	Effectiveness of the final waste form is questionable or indeterminate	Highly effective final waste form
WCS Implications	Extremely difficult to implement for WCS drums	Straightforward to implement for WCS drums
Scalability and Complexity	Extremely difficult to implement for drum remediation	Straightforward to implement for drum remediation
Facilities Challenges	Extremely difficult to implement due to Authorization Basis scope	Highly likely to implement under current LANL Authorization Basis status.
Schedule	Extremely time consuming	Expedited schedule is achievable
Cost***	Extremely expensive	Cost-effective option

*If a treatment option was judged by the Core Team to be infeasible based on any of the criteria, it was eliminated in the initial screening and not considered further. A minimum score of 1 applied to an option that is not screened out is a very unfavorable score, but by definition is not a score that on its own rules the option out.

** A more precise definition of the scores for ease of permitting is provided in the text description of this criterion (see Appendix 3).

*** Cost was not a primary evaluation criterion used to evaluate potential options; it is provided for information purposes and could have been used as a final discriminator in the event of ties. The evaluation process did not lead to any ties: therefore, the cost scores are for information only and did not factor into the final recommendations.

6 Assessment Results

This section presents the results of the evaluation of the Core Team of the fourteen potential treatment options against the evaluation criteria, leading to the recommendation of treatment options for the RNS and UNS waste streams. As discussed in Section 5.2, the evaluation occurred in two steps: a prescreening step and a full evaluation of options not screened out in the first step. The results of the evaluation, including a narrative capturing the discussions within the Core Team, are provided in the next two subsections, and discussed in Section 6.3.

6.1 Screening Results

The results of the screening exercise indicate that each of the five stabilization treatment options (Options 1 through 4, and Option 14) were determined to be suitable for full evaluation, whereas the other RCRA treatment options were screened out in the initial evaluation. This section provides the rationale for the elimination of options 5 through 13, capturing the discussion of the Core Team leading to the screening decision.

Option 5. Incineration

In theory, this method is attractive from a volume standpoint in the sense that it minimizes the mass and volume of the final waste product by destroying both the nitrate and starch components in a system with engineered controls for deflagration. The result should be highly radioactive metal oxide wastes, assuming that all of the nitrates that do not react with the cellulose decompose to a non-oxidizing solid. Experience suggests that this operation would be very difficult to permit and is complicated by the presence of transuranics. The incineration of the RNS drums may concentrate the TRU waste and the heating of TEAN may have dangerous consequences. Previously, a radiological incinerator at LANL was constructed but proved to be very difficult to permit, and DOE decided not to go forward with the incinerator after approximately a year of experimental testing. Thus, the Core Team believed that the risk of failure to achieve the necessary safety basis and regulatory approvals is unacceptably high.

Based on Safety Basis Challenges and Ease of Permitting, this treatment option is removed from further consideration for RNS waste and debris, and, as discussed in Section 4.2, is not applicable for UNS waste.

Option 6. Thermal Oxidation of Organics

In the context of the current RNS waste stream, lab experiments conducted by LANL prove that heating would unavoidably result in the onset of thermal runaway and further work needs to be done to ensure 60 °C is the bounding condition. However, this option may therefore be considered "inadvertent incineration," which is not acceptable from either a

safety or regulatory basis.[15] Removal of organic materials may concentrate TRU waste and the heating of TEAN may have dangerous consequences. Both dry and wet thermal oxidation techniques were considered. Current wet thermal oxidation techniques involve the use of superheated steam that would require complex additional facilities and procedures.

Based on Safety Basis Challenges and Ease of Permitting, this treatment option is removed from further consideration for RNS waste and RNS debris, and is not applicable for UNS waste (see Section 4.2).

Option 7. Biodegradation

One hypothesis concerning the initial heating of the waste drums holds that biological metabolism of the organic kitty litter is heating the drums, and that for drum 68660, this heat generation was sufficient to trigger other exothermic reactions leading to thermal runaway. According to this hypothesis, adding competent biological organisms, including salt resistant bacteria, to the dry waste could precipitate thermal runaway. Alternatively, wetting the waste sufficiently to afford a heat sink for the biological activity and adequately reduce the high ionic strength of the medium would only be a preliminary step, as the waste would need to be further treated to make it acceptable under the WIPP WAC. This would require extensive drying and dilution after a long incubation period. Finally, any nitrated starch in the barrels would likely be untouched, effectively concentrating a compound of greater hazard than the original organic absorbent. This option is not acceptable due to complicated accretion of risk and is time and cost prohibitive.

Based on Safety Basis Challenges this treatment option is removed from further consideration for RNS waste, and is not applicable for UNS waste or debris (see Section 4.2).

Option 8. Chemical or Electrolytic Oxidation

The fundamental instability of the remediated nitrate salt waste stems from the mixture of fuel with oxidants. One redeeming outcome of the method used is the fact that the average drum is probably fuel rich, although knowledge of the exact contents of the drums is limited. Addition of oxidizing compounds will bring the material closer to oxidative stoichiometry, increasing the potential hazard. Electrochemical oxidation suffers from the low solubility of starch in aqueous solution and the necessary dilution of the waste into a large volume of aqueous solvent. This treatment process could result in thermal runaway. Also, the waste stream already contains oxidizing material. The goal of this treatment is to remove the oxidative properties, not to enhance the waste.

Based on Safety Basis Challenges this treatment option is removed from further consideration for RNS waste, and is not applicable for UNS waste (see Section 4.2).

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[15] UNS waste drums do not require the same remediation as the RNS waste drums. Reactions may occur between the salts but heat is not generated to cause an additional reaction.

Option 9. Chemical Reduction

For RNS waste, the fundamental instability stems from the mixture of fuel with oxidants. It is not clear that adding more fuel will improve the situation; moreover, it is likely to evolve heat and thermally traumatize the material. None of the reducing agents listed are effective against nitroesters. Thus the expected result for RNS waste is a radiological contaminated energetic fuel with no disposal path. This treatment process could result in thermal runaway. For UNS waste, chemical processing of this sort would present severe safety basis challenges associated with the act of deliberately adding fuel to the nitrates. Heating the UNS waste would create an oxidizing environment. This operation falls outside of existing facility safety basis (engineered operation to control the chemical reduction in an efficient and safe manner). This reaction is highly exothermic and could result in uncontrolled release of material. Containment of reaction requires special facilities. Facilities for this treatment would need to be built. This option would be time and cost prohibitive.

Thus, based on Safety Basis Challenges and Short Term and Long Term Effectiveness this treatment option is removed from further consideration for both RNS and UNS.

Option 10. Vitrification

Vitrified waste forms are highly durable and of uniform consistency. If the process is well controlled, all organic constituents in the RNS waste will be destroyed. However, this treatment process is equivalent to, if not more violent than, incineration. The level of process control required is intensive, and thus vitrification is generally applied only to large waste streams in facilities resembling a chemical plant. Furthermore, for disposal in salt at WIPP, a waste form with the durability of glass is not required. Vitrification technology may not be locally available. Mobile units could be relocated but could be cost prohibitive to permit efficiently.

Based on Scalability and Complexity and Schedule this treatment option is removed from further consideration for both RNS and UNS waste.

Option 11. Alternate Macro-Encapsulation

The fundamental instability of the RNS waste stems from the mixture of fuel with oxidants. Coating the oxidizing nitrate salt particles in an organic polymer would improve intimate mixing between fuel and oxidizer, potentially sensitizing the waste. Furthermore, for either RNS or UNS waste, the virtue of reduced susceptibility to leaching is of minimal benefit in the WIPP repository, a dry repository in bedded salt, with no groundwater intrusion and minimal natural fluids. Per EPA stabilization/solidification documents, this is not recommended for TRU waste.

Based on Short Term and Long Term Effectiveness, this treatment option is removed from further consideration for both RNS and UNS waste.

Option 12. Neutralization

Both the starch and nitrostarch in RNS waste could be destroyed by adequate addition of alkaline media (e.g. sodium hydroxide solution). Experiments with these protocols were conducted by LANL as a pre-treatment for cementation. The relative merits of these protocols are relevant in regard to cementation. However, while acid- or base-catalyzed hydrolysis could be used to degrade the nitrostarch component of the RNS waste, it would be difficult to monitor the progress and ensure complete destruction. Furthermore, this treatment would do nothing to address the oxidizer characteristic associated with the nitrate salts in either the RNS or UNS waste. Thus, neutralization on its own will be insufficient to treat the waste, and must be combined with solidification or absorbent addition to be considered an adequate treatment process to remove the D001 characteristic. Neutralization will not remove the highly soluble nitrate salts.

Neutralization treatment option, as a stand-alone treatment, is not considered for either RNS or UNS waste or debris. This discussion was based on reduction of toxicity and mobility. However, neutralization may be a step within another treatment option such as cementation.

Option 13. Controlled Reaction or Leaching of Reactive Inorganic Chemicals with Water

None of the ingredients in the RNS waste are water reactive. Nitrate salts in either the RNS or UNS waste could be removed by liquid/solid extraction. However, for the RNS waste, this would have no effect on nitrated starch material, and the resulting waste would potentially be a radiological contaminated energetic fuel with no disposal path. For UNS waste, the leaching on its own would result in an aqueous waste stream that would need to be combined with a solidification option such as cementation to be considered an adequate treatment process.

Based on Short Term and Long Term Effectiveness, this treatment option is removed from further consideration for RNS waste, and on its own is not considered further for UNS waste, but may be a step within another treatment option such as cementation.

The results of the screening exercise are presented in summary form in the bottom portion of Table 6-1.

Table 6-1. Summary of results of the evaluation of treatment options

EVALUATION CRITERIA / POTENTIAL TREATMENT OPTIONS	Robust to waste stream variability	Ease of permitting (permitting difficulties)	Safety basis challenges	Extent of testing required	Reduction of toxicity and mobility	Reduction in volume	Short-term and long-term effectiveness	WCS implications	Scalability and complexity	Facilities challenges	Schedule	Cost (not a primary evaluation criterion) *	SCORE
1 Stabilization Using Zeolite (remediated)	5	3	4	5	4	2	4	2	4	4	4	4	41
Stabilization Using Zeolite (unremediated)	5	3	4	5	4	2	5	N/A	5	5	5	5	43
2 Stabilization Using Zeolite With Cementation (remediated)	5	2	3	3	4	1	4	1	2	2	1	1	28
Stabilization Using Zeolite With Cementation (unremediated)	5	2	3	3	4	1	5	N/A	3	3	2	1	31
3 Stabilization Using Dry-Process Cementation (remediated)	5	2	2	3	4	3	4	1	3	2	2	2	31
Stabilization Using Dry-Process Cementation (unremediated)	5	3	2	3	4	3	5	N/A	4	3	3	3	35
4 Stabilization Using Wet-Process Cementation (remediated)	3	4	1	3	4	2	4	1	1	1	1	2	25
14 Salt Dissolution With Cementation/Stabilization (remediated)	3	3	1	3	4	2	4	1	2	1	2	2	26
5 Incineration													
6 Thermal Oxidation of Organics													
7 Biodegradation													
8 Chemical or Electrolytic Oxidation													
9 Chemical Reduction													
10 Vitrification													
11 Alternate Macro-Encapsulation													
12 Neutralization													
13 Controlled Reaction or Leaching of Reactive Inorganic Chemicals With Water													

Note: Stabilization Options 1-4 and 14 are discussed in Section 4.1 RCRA Stabilization Options. Options developed from RCRA treatment standards are the gray-shaded rows. Red cells denote the screening out of an option based on a high degree of infeasibility with respect to that criterion. Because of the initial screened-out determination, Options 5-13 were not ranked. Discussion of Options 5-13 is found in Section 4.2 Additional RCRA Treatment Options.
*Cost not included in final score.

6.2 Full Evaluation of Remaining Options

Based on the screening out of options 5 through 13 and the judgment that Options 1 through 4 and 14 were feasible, the Core Team performed a full evaluation of the latter group, which are the five RCRA stabilization options described in Section 4.1. The most effective way to compare the options was to discuss the relative merits of each option for each criterion, and then present the results by criterion. Typically, the group discussion focused on the more problematic RNS waste stream including debris, and after scores were established, the UNS scores were determined by reference to the RNS score. For example, for Scalability and Complexity, the UNS score is one point higher than the corresponding RNS score because temperature control is not required for UNS waste). This logic is also captured in the discussion below.

Criterion 1: Robust to Waste Stream Variability

The committee carefully examined the initial five options and compared the testing results and input from an explosives and reactive material Subject Matter Expert (SME) on the stability of the zeolite waste form produced from Option 1 (Stabilization Using Zeolite). Further discussion examined the data obtained from testing completed by a cementation SME for the cement waste form produced by the options employing cementation. In addition, there was discussion of the equipment and training requirements to correctly execute and consistently produce the waste forms from all options. The variability of the waste from drum to drum, and within a drum, was also assessed to evaluate the applicability of the treatment strategy suitable across the expected range of compositions.

After consideration of the test data, the procedural steps required, the equipment complexity, and waste stream variability, it was the consensus of the committee that the first three options were highly likely to develop a robust process (score of 5) for both the RNS and UNS. All options involve deactivating D001/D002 for waste and debris, and for these options there was little doubt that a robust formulation could be devised to accomplish this objective of rendering the waste unreactive. Option 4 (Stabilization Using Wet-Process Cementation) was ranked a 3 for RNS waste due to the additional complexity of the two-week hold time after water addition, opening the possibility that low-level reactivity could vary across the drum population and complicate the process. Option 14 (Salt Dissolution With Cementation/Stabilization) also ranked a 3 due to the resulting two end streams and the requirement that the dissolved solids must meet the pH requirement for waste and steel corrosion.

Criterion 2: Ease of Permitting (Permitting Difficulty)

Under the assumption that a modification of the LANL Hazardous Waste Facility Permit would be required (see Section 3), the evaluation approach of the Core Team was to examine the degree of complexity for each stabilization treatment option required by standard RCRA permitting factors. Option 4 produces a score of 4 while the other options produce a score of 3. The basis for the higher score was that the permitting difficulty for simpler cementation based processes would be easier due to the common use of the cementation process in the waste management industry.

Upon discussion by the review committee, the RCRA permitting process and schedule, including the NMED's review and approval, would be similar for each treatment option. The original documentation proposing the five treatment options (Appendix 1) captured this by suggesting that a possible permitting mechanism for all the options would be a Temporary Authorization by the NMED with a follow-up Class 2 or 3 Permit Modification Request. Therefore, the potential extent and complexity of the technical discussion needed to be included in each permit modification submittal was estimated for each treatment option and focused on as the determining evaluation criterion rather than simply the permit modification class as originally proposed in the definition of the Ease of Permitting criterion.

Option 1 (Stabilization Using Zeolite) has the advantage of being similar to the process that was previously used to prepare TRU waste containers for WIPP certification. Additionally, the treatment option would be limited to a single permitted treatment storage and disposal facility (WCRRF) at LANL. However, a permit submittal would need to present a strong technical discussion regarding the use of zeolite to inert the ignitable waste including the determination of appropriate types of zeolite, final volumetric ratios with the waste, sampling results, and any other factors determined to be relevant. Based on these complications and technical requirements, the zeolite treatment option was assigned a score of 3 for the Ease of Permitting evaluation criteria. The process required for both RNS and UNS waste appeared similar and the evaluation score of 3 was applied to both types of waste and includes debris.

Option 2 (Stabilization Using Zeolite With Cementation) combines the zeolite process with a second cementation step. Cementation adds the complication of water addition and treatment by neutralization to prepare the waste for solidification with the cement. However, cementation is also a commonly employed treatment procedure for these types of waste and is similar to the treatment process at TA-55 which is already approved in the LANL permit (this is also true of Options 3 and 4). The combined steps for two processes will require a larger amount of technical description in the permit modification request involving both the WCRRF permitted unit and a new location for cementation at TA-54 Area G. The two sites and additional operational changes will also influence other parts of the LANL permit for the two facilities, including potential changes to operational factors such as inspections, training, waste management operations, and emergency procedures. Therefore, the treatment option was assigned a lower score of 2 due to the increased

potential for complexity in the permit modification request. The value was applied for both RNS and UNS waste.

Option 3 (Stabilization Using Dry-Process Cementation) uses the same two waste management sites but limits waste processing at WCRRF to segregation to prepare the waste for subsequent remediation at a new TA-54 Area G cementation location that would require a permit modification. However, many of the same potential operational factors that would need to be described for changes to the permit would be similar. Therefore, the treatment option for the remediated waste stream was assigned the same score of 2 for the potential permitting complexity. However, the absence of the organic component in the UNS waste was considered to be a less complex technical process, and the Ease of Permitting score was raised to 3 for that waste stream.

Option 4 (Stabilization Using Wet-Process Cementation) and Option 14 (Salt Dissolution With Cementation/Stabilization) would also use the same two waste management sites and potential operational factors, implying increased operational changes associated with the permit. However, as stated above, cementation treatment alone in Option 4 is a simpler process and has been previously approved. Option 14 is slightly more complex than Option 4 due to the generation and treatment of two discrete waste streams with associated facilities but similar in the cementation processes. The early addition of water would minimize the worker safety concerns and waste management procedures related to the oxidizer capability in the early stages of the process, a beneficial factor for permitting by potentially mitigating the degree of operational change descriptions needed to modify the permit. The need for temperature control of the waste is limited to the earliest stages of the waste treatment process, making potential permit conditions at WCRRF less complex. As a result, options 4 and 14 were assigned evaluation criteria values of 4 and 3, respectively, for the remediated waste stream regarding permitting difficulty.

Criterion 3: Safety Basis Challenges

This criterion includes the facility features needed for radiation protection, as well as the degree of procedure development needed to ensure that requirements for worker safety are met. If a treatment option can use or build from the existing safety basis analysis, the challenges will be reduced. Conversely, if facilities not previously used to treat waste are envisioned, or if different processes are developed that are complex or require new controls, safety basis challenges are more severe.[16] On that basis, Option 1 (Stabilization Using Zeolite) was judged to be the option with the simplest safety basis path forward because the operations (transport, processing at WCRRF) are those that were already used to process nitrate salts at LANL.

Comparing the remaining cementation options, Option 2 (Stabilization Using Zeolite With Cementation) and 3 (Stabilization Using Dry-Process Cementation) are identical up to the point at which zeolite is added. After that point, wastes are transported to TA-54 Permacon

[16] There is no impact to the safety basis when the drums are cooled, unless cooling is considered a treatment. The controls considered are temperature and handling.

231 for cementation. Because the mixing with zeolite removes the ignitability and corrosivity hazards, the subsequent movement to TA-54 presents fewer safety basis challenges, making Option 2 (Stabilization Using Zeolite With Cementation) somewhat less onerous (from a safety basis perspective) than Option 3 (Stabilization Using Dry-Process Cementation). It is believed that there is also a clear separation between these options and Option 4 (Stabilization Using Wet-Process Cementation) and Option 14 (Salt Dissolution With Cementation/Stabilization), which has the challenges of the other two cementation options, but also includes movements and handling of waste to which water has been added. These new additional steps led to the determination that Option 4 (Stabilization Using Wet-process Cementation) and Option 14 (Salt Dissolution With Cementation/ Stabilization) present the most difficult safety basis challenges of the five options and were given a score of 1.

In summary, for RNS waste, the team perceives a distinct difference in the five options, resulting in the assignment of scores of 4, 3, 2, 1, and 1 to Options 1, 2, 3, 4, and 14 respectively, for the safety basis criterion. For UNS waste, the team believed that essentially the same challenges exist, so the same scores were assigned for the first three options. Option 4 is not applicable for UNS waste or debris.

Criterion 4: Extent of Testing

Extent of testing refers to the amount and complexity of sampling and analysis required to implement the treatment process. The new characterization of the TRU nitrate salt bearing waste stream with the D001 EPA hazardous waste number for ignitability (based on the presence of oxidizers) requires that the final treated product or appropriate surrogates must demonstrate that the oxidizer capability has been negated by testing to SW-846 Test Method 1030, Ignitability of Solids, Test Method 1040, Oxidizing Solids, Test method 1050 Test Methods to Determine Substances Likely to Spontaneously Combust and DOT methods. Since any treatment strategy would require such testing, there are no scoping differences that would contribute to the overall score. Likewise, gas and solids sampling of the barrels was not included as it is common to all processes. The evaluation specifically compared the amount of testing that would be required during the remediation operation, and post-processing.

For any cementation operation (all Options except Option 1, Stabilization Using Zeolite), achieving the proper pH for the mixture is critical to making a viable grout, making pH testing mandatory during remediation to ensure proper pH. In addition, cemented mixtures are known to dewater during storage, which adds an additional requirement[17] for tests to ensure that the solid matrix was stable and did not lose water. By comparison, the Core Team believes that no pH testing was necessary or beneficial in the case of Option 1 (Stabilization Using Zeolite), and that post-treatment dewatering may not be necessary when the prescribed selection of the appropriate zeolite ratio is used.

[17] The WIPP WAC (DOE/CBFO, 2013) requires that, due to corrosivity concerns, the waste packages contain no free liquids.

Based on these considerations, Option 1 (Stabilization Using Zeolite) received a score of 5 for both RNS and UNS waste since they require no tests other than those requisite for waste acceptance. All of the remaining options involve cementation, requiring pH testing during the remediation operation followed by surveillance for dewatering after they had set. For this reason, these options all received a score of 3 for both RNS and UNS waste.

Criterion 5: Reduction of Toxicity, Mobility, Corrosivity, and Ignitability

The design and operating permit for the WIPP facility is the primary consideration for the applicability of the criteria for mobility of contaminants.[18] In a bedded salt repository, the waste form is of secondary importance to the long-term performance of the repository. The waste form for all options is a solid waste confined by the waste containers. Even if the waste form dewaters over time, the amount of liquid liberated would be insufficient to facilitate transport of radionuclides through the salt bed to the accessible environment. The self-sealing of the salt will limit the availability and transport of water into and through the repository, and correspondingly minimize the potential release of TRU nuclides from the repository. In the undisturbed repository scenarios considered by the WIPP repository program, no significant release of actinides from the WIPP is predicted.[19] The nature of the WIPP salt bed would prevent mobility of contaminants. All five options meet the WIPP WAC, are an effective waste form and fairly straightforward to package as long as the corrosivity and ignitability characteristics of the content are removed to mitigate the safety hazard. Therefore, this criterion was determined to not be a discriminator among treatment options, so a uniform score of 4 was applied to each option.

Criterion 6: Reduction of Volume

The number of daughter drums generated by each option was the primary criterion used for ranking each option with respect to this criterion. The estimated number of drums generated for the five options are 399, 798, 285, 342, and 285 respectively (Table 6-2). Based on the fact that all five options increase the number of drums of waste to be disposed, the maximum number for these options was capped at 3: Option 3 (Stabilization Using Dry-process Cementation) received this score. Scaling the remaining scores to the relative number of drums generated, Option 1 (Stabilization Using Zeolite) received a score of 2, Option 2 (Stabilization Using Zeolite With Cementation) scored a 1, Option 4 (Stabilization Using Wet-Process Cementation) scored a 2, and Option 14 (Salt Dissolution With Cementation/Stabilization) scored a 2. The corresponding scores for UNS waste, where applicable, were assigned the same values.

[18] WIP WAC prohibits free liquid. Therefore, WIPP is not permitted to accept wastes with observable liquid that is more than 1 percent by volume of the outermost container at the time of radiography or visual examination.

[19] Title 40 CFR Part 191 Subparts B and C Compliance Recertification Application 2014 for the Waste Isolation Pilot Plant Appendix SOTERM-2014 Actinide Chemistry Source Term, Appendix PA-2014, Section 7.

Criterion 7: Short Term and Long Term Effectiveness

Regarding the effectiveness of the examined remediation options to produce an acceptable final waste form, RNS and UNS mixed with zeolite or in a concrete monolith are equally acceptable if a sufficiently robust cemented waste form is developed that will not dewater. The scoring of Criterion 4, Extent of Testing Required, covers the development and testing of a cement waste form containing RNS or UNS. Should testing fail to reveal a cemented monolith waste form that will not undergo dewatering then Option 1 (Stabilization Using Zeolite) is the superior remediation option. However, assuming that testing confirms the suitability of either type of waste form, there is no reason to favor one over the other with respect to effectiveness.

Further, it is recognized that treatment to an acceptable final waste form for the UNS waste can be accomplished with greater certainty than for the RNS waste. Mixing of the UNS with either zeolite or grout to remove the ignitability characteristic assigned to oxidizers is straightforward and has already been thoroughly examined by Walsh (2010). The conservative zeolite or grout treatment ratios will be sufficient to account for future liquid production and will, therefore, remove the potential for the corrosivity characteristic. If enough zeolite is used, dewatering will not occur. Therefore, on the basis of this increased certainty for UNS waste, scores are assigned one point higher for UNS waste than for RNS waste. Thus, all five options received a score of 4 for RNS waste, and the three options applicable to UNS waste received a score of 5.

Criterion 8: WCS Implications

This criterion, which addresses the relative ease with which a treatment process could be implemented for nitrate salt waste in storage at WCS, applies only to the RNS waste. The Core Team discussed two general approaches to treatment of WCS waste: On-site treatment at WCS, and transport of waste to LANL where treatment would be conducted using LANL facilities. The team did not discuss burying the drums at WCS. If the waste were to be treated at LANL, the untreated waste residing at WCS does not meet certification of compliance for transport. The RNS waste is considered ignitable; therefore, transporting the RNS waste without treatment requires an exception by NRC. The team evaluated the options under the assumption of the need for WCS to construct and operate an on-site capability to process the waste due to the difficulty in transporting ignitable waste. There was agreement that this would be a difficult process and that relatively low scores should be given to any of the options. Comparing Option 1 (Stabilization Using Zeolite) to the three cementation options, deploying a glove box for the single step of zeolite addition was judged to be easier than deploying equipment for multiple steps of a cementation process. On that basis, Option 1 (Stabilization Using Zeolite) was given a score of 2, and each of the cementation options was given a score of 1.

Criterion 9: Scalability and Complexity

In the evaluation of this criterion, issues that were considered were the ability to treat RNS and UNS with the current available facilities at LANL, consideration of whether similar

operations have been performed at LANL or elsewhere in the DOE complex, and the number and complexity of steps required to complete the operation. The availability of engineering controls to meet ALARA in accordance with LANL and DOE requirements were also considered.

Table 6-2, constructed from the descriptions developed in Appendix 1, allows the options to be compared with respect to the number of facilities used, the total number of operational steps, the number of transport movements between facilities, and the complexity in procedure and/or facility changes. This table contains information relevant to this criterion, as well as the next (Facility Challenges).

In summary, Option 1 (Stabilization Using Zeolite) is the most straightforward option to implement due to the smaller number of operational steps, the use of only WCRRF for treatment, and the precedent of having performed these operations in WCRRF in the past (albeit with an inappropriate use of an organic absorbent, non-permitted neutralization and in violation of the BIO). It was given a score of 4 for RNS waste as a result. All of the cementation options involve many more operational steps and drum transport steps. On a relative basis, Option 3 (Stabilization Using Dry-Process Cementation) is the most straightforward of the cementation options and has the lower number of daughter drums generated. Next is Option 2 (Stabilization Using Wet-Process Cementation) has one fewer step than Option 3 (but many more than Option 1) but suffers in this evaluation from the generation of many more daughter drums. One of the most complex, least scalable choices is Option 4 (Stabilization Using Wet-Process Cementation), which involves a large number of operations and transport steps, water addition at TA-54 Permacon 375 (which presents new challenges), and the transport of drums which have had significant water added. Option 14 (Salt Dissolution With Cementation/Stabilization) consists of a filtration process followed by two separate streams, nitrate solution and Swheat™ cake, both requiring cementation. For these reasons, the scores issued to these four options for RNS waste were 4, 2, 3, 1, and 2 respectively.

For UNS waste, the scores applied to the three options are one point higher than the corresponding RNS waste score for that option due to the absence of required temperature control, which makes the operations less complex.

Table 6-2. Statistics and features of the five stabilization treatment options

Option	# of Daughter Drums[1]	Drum Duration (days)[2]	# of Operational Steps[3]	# of Facilities[4]	# of Drum Movements[5]	Other Considerations[6]
1. Stabilization Using Zeolite	399	4	6 w/ debris removal	2	2	• Precedent has been established for this option • Personnel are familiar with this option • Readiness activities should be straightforward compared to cementation operations stood up at TA-54 • WCRRF is authorized for TRU waste treatment
2. Stabilization Using Zeolite With Cementation	798	29	10	3	3	• Additional procedures and training for cementation process (also applies to Options 3 and 4) • New glove box and related utilities and permit modification (also applies to Options 3 and 4)
3. Stabilization Using Dry-Process Cementation	285	10	9	3	3	• Fewer number of daughter drums makes this a more scalable option than the other cementation options
4. Stabilization Using Wet-Process Cementation	342	27	10	3	3	• Water addition would be an additional new operation • Drum movements after water addition is a new operation
14. Salt Dissolution With Cementation/ Stabilization	285	4	10	2[6]	2	• Water addition, filtration with water, and filter press of sludge, and drum movements after water addition are new operations.

[1] Values are for treatment of the RNS drums. Corresponding values for the UNS waste scale by a factor of 29/57, or 0.51. (The number of steps and transportation between facilities accounts for the increase in option time.) The number of daughter drums includes grouted parent and debris drums.
[2] Drum duration refers to the "cycle time" starting from initial handling to a completed waste drum ready for shipment.
[3] Operational steps are represented schematically in Figure 4-1. Values do not include temperature control steps, which apply to all options for RNS waste.
[4] Facilities include WCRRF at TA-50 (all options), TA-54 Permacon 375 (current storage location of RNS waste), and TA-54 Permacon 231 (assumed to be used for cementation operations, if applicable).
[5] Movements include transport from current location to WCRRF, transport to cementation location (applicable for cementation options), and transport of treated daughter drums to final storage location.
[6] Facility location has not been determined.

Criterion 10: Facility Challenges

In the evaluation of this criterion, the issue that was considered was the ability to use available sites and facilities that are currently operating under the LANL approved Authorization Basis (AB) to treat RNS and UNS waste. Evaluation of options consisted of comparing the number of facilities used in each option, the current operational configuration of each facility and what operation(s) are currently authorized to occur in each facility.

In summary, Option 1 (Stabilization Using Zeolite) was judged to present the easiest path from a facility readiness and AB perspective. WCRRF could be used for Option 1 without modification, and is already authorized for TRU waste treatment. In contrast, the three cementation options all employ one additional facility, and require the installation of a glove box in TA-54 Permacon 231, with accompanying new evaluations to obtain AB approval. Thus, the cementation options are all ranked significantly below Option 1 (Stabilization Using Zeolite) for this criterion. Of the four, Option 4 (Wet-Process Cementation) and Option 14 are considered to be the most challenging with respect to facilities because the additional complication of the water addition step in TA-54 Permacon 375 requires introduction of additional new equipment (beyond that of the other cementation options) that would need to be evaluated prior to operations. For these reasons, the four options received scores for RNS waste of 4, 2, 2, 1, and 1 respectively for the facilities challenges criterion.

For UNS waste, the scores applied to the three options are one point higher than the corresponding RNS waste score for that option due to the absence of required temperature control, which makes the facilities challenges somewhat less onerous.

Criterion 11: Schedule

Schedule factors considered in the Core Team deliberations included compliance schedules, staffing requirements, and project and procedure development. Some factors influencing the schedule, such as the time required for permitting approvals, and treatment-process facility design complexity, were not included here because it was agreed that those are covered in other criteria. Additionally, during discussion it was recognized that dominant factors influencing schedule (discounting the preliminary steps before treatment operations) were the number of drums created, and the "cycle time" associated with a drum, from first handling to completion of all steps to make the drum ready for shipment. A lower cycle time results in a decrease in the number of drums generated which require less storage space, potential movement, and processing time. These measures are provided for the four potions in 6-2.

Option 1 (Stabilization Using Zeolite) was determined to rank the highest among the four options due to the modest number of daughter drums created[20] and the short

[20] The value of 399 daughter drums is thought to be an upper-bound estimate because it is based on a 3:1 zeolite/waste ratio, which very likely overestimates the amount of zeolite required to inert the RNS waste.

drum duration. In contrast, all of the cementation options have significantly longer drum durations. Options 2 (Stabilization Using Zeolite With Cementation) and 4 (Stabilization Using Wet-Process cementation) have particularly long drum durations due to the large number of steps required. Option 4 has the unique requirement of a hold time on the drums after initial water addition. Option 14 (Salt Dissolution With Cementation/Stabilization) consists of a two-part process; nitrate solution collected in one drum and Swheat™ cake collected in a second drum. Both drums require cementation processing. With regard to the number of daughter drums generated, the cementation process envisioned requires leaving enough room in the drum for cement addition and mixing after splitting the RNS waste, resulting in a lengthy process of cementation being applied to a large number of daughter drums. Option 2 (Stabilization Using Zeolite With Cementation) would generate a particularly large number of daughter drums, which lowers this option's rating with respect to schedule. Option 3 (Stabilization Using Dry-Process Cementation) and Option 14 (Salt Dissolution With Cementation/Stabilization) are the best of cementation options with respect to schedule due to the relatively small number of daughter drums generated, but it is not as time-efficient as Option 1. In summary, based on these considerations, the four options for RNS wastes received scores of 4, 1, 2, 1, and 2 respectively for the schedule criterion.

For UNS waste, the scores applied to the three options are one point higher than the corresponding RNS waste (and debris) score for that option due to the absence of required temperature control, which should shorten the times required to complete the processing of a waste drum.

Criterion 12: Cost

Cost was not used as a criterion for discriminating between treatment options, and was not included in the summation of scores used to rank the options. The scores and this description are included for information purposes, capturing the discussion conducted at the ranking meeting.

For RNS waste, judgments on the relative costs of the options were based on: 1) the number of facilities employed, and the required changes to these facilities in order to conduct the work, 2) the estimated number of daughter drums generated, which correlates to materials and labor costs; and 3) the cycle time required to remediate a drum, which includes additional costs for operations for items such as surveillance while a drum is being remediated. On these bases, Option 1 (Stabilization Using Zeolite) ranks as the most cost efficient option based on the use of existing facilities at WCRRF and Area G, the need for only a single movement of waste after cold safeing, the relative efficiency in terms of number of daughter drums generated, and the relatively fast cycle time to complete the remediation of each drum. A relatively high score of 4 was assigned for these reasons. On the other end of the spectrum, Option 2 (Stabilization Using Zeolite With Cementation) received a low score of 1 based on the far greater number of labor hours per drum, the large number of daughter drums generated, the more involved facility change process required, and greater shipment costs between

facilities. Option 4 (Stabilization Using Wet-Process Cementation) similarly received a low score of 1 because the lower number of daughter drums compared to Option 2 was judged to be offset by the slow cycle time and corresponding larger labor and surveillance costs. Option 4 (Stabilization Using Dry-Process Cementation) and Option 14 (Salt Dissolution With Cementation/Stabilization) was judged to be intermediate to Options 1 and 4 in these aspects, and thus received a relatively low but intermediate score of 2. Option 14 (Salt Dissolution With Cementation/Stabilization) scored a 2 because of the need for a new nuclear facility and gloveboxes.

Operations for UNS waste are the same as for RNS waste except that temperature control operations are not included. Accordingly, the scores for UNS wastes were set one point higher than the corresponding RNS waste score for Options 1 and 3. (Options 1 and 4 received scores of 5 and 3, respectively). The elimination of temperature control steps for Option 2 was deemed to be inconsequential compared to the costliness of the other operations, so Option 2 received a score of 1 for UNS waste, as it did for RNS waste.

6.3 Discussion of Results

The overall results presented earlier in Table 6-1 indicate that for both RNS and UNS waste, Option 1 (Stabilization Using Zeolite) ranked the highest based on the criteria used in the evaluation. This is seen from the total obtained by adding all of its scores except cost, which was not included in the summation. The four cementation options were significantly lower in total score, and were ranked in the following order for RNS waste: the second-ranked option was Option 3 (Stabilization Using Dry-Process Cementation); the third-ranked option was Option 2 (Stabilization Using Zeolite With Cementation); fourth-ranked option was Option 14 (Salt Dissolution with Cementation/ Stabilization); and the fifth-ranked option was Option 4 (Stabilization Using Wet-Process Cementation). For UNS waste, the order of the rankings was the same: Option 1, Option 3, Option 2, and Option 14 (Option 4 is not applicable for UNS waste). Generally, the positive or negative attributes leading to a higher or lower score for a given criterion held true for either RNS or UNS waste. Therefore, the remainder of this discussion will focus on the RNS waste and RNS debris.

The score for Option 1 (Stabilization Using Zeolite) exceeded that for any cementation option by 10 points or more; for virtually all of the 11 criteria applied to the evaluation, this option scored equal to or higher than any of the cementation options. Exceptions to this conclusion are: 1) for the Ease of Permitting criterion, Option 4 (Stabilization Using Wet-Process Cementation) was deemed to pose fewer obstacles to permitting than simple zeolite addition and 2) for the Reduction in Volume criterion, Option 3 (Stabilization Using Dry-Process Cementation) ranked higher than zeolite addition because of the smaller number of daughter drums generated. These are very isolated instances of a higher score for an option other than Option 1. Therefore, even if one were to apply unequal weightings to the various criteria, the conclusion that Option 1 (Stabilization Using Zeolite) is the preferred option will not change. Therefore, the recommendation to pursue Option 1 is very robust.

The results of the cost criterion, though not used in the analysis, reinforce the results of the overall evaluation in that the treatment option recommended based on non-monetary criteria is also judged to be the most cost effective option. Had cost been included in the evaluation, rather than given a zero weight, the recommendation of Option 1 would have been even stronger.

An important aspect of the analysis was the inclusion of a variety of non-stabilization RCRA standards based treatment options in the pre-screening phase of the evaluation. In effect, each of these options received a failing score on one or more criteria, and thus was screened out. Clearly, this result applies only for the particular nitrate salt waste streams at LANL, and is not a general conclusion. Difficulties in permitting, safety basis, and short-term or long-term effectiveness of the final waste form were typical criteria that led to the elimination of most of these options.

Finally, most of the criteria applied to these treatment options had value in discriminating among options. The exception is Reduction of Toxicity and Mobility, which was found to be an ineffective criterion for this application because those attributes are relatively unimportant for waste disposed at WIPP. Typically, such a criterion would be important for low-level waste disposal or situations in which credit will be taken for a durable waste form resistant to leaching of contaminants. This is not the case for disposal of TRU waste at WIPP: Therefore, this criterion should be eliminated from use for any future analyses of this sort.

7 Conclusion

The evaluation of various processes to judge their suitability for treating the nitrate salt wastes at Los Alamos led to a definitive recommendation that Option 1 Stabilization Using Zeolite be pursued for both the RNS and UNS waste streams and associated debris. This result confirms the previous recommendation of Clark and Funk (2015) to mix the waste with zeolite to mitigate the corrosivity and ignitability characteristics. The Clark and Funk recommendation was based primarily on scientific and technical considerations. The evaluation process reported herein was designed to be comprehensive, in terms of the variety of treatment options considered, and robust, in terms of the use of a diverse set of criteria in the evaluation. The Core Team conducting the evaluation consisted of subject matter experts across a wide range of disciplines, thereby ensuring that appropriate experts in the scientific, operational, safety and regulatory arenas informed the evaluation of the options. These factors, plus the decided advantage of zeolite addition revealed by the evaluation, provide confidence in the recommendation.

The results of the Options Assessment Report were externally peer-reviewed. LANL recognizes that the results of the analysis will be vetted with NMED and that a modification to the LANL operating permit is a necessary step before implementation of this or any treatment option. Likewise, facility readiness and safety basis approvals must be received from the DOE. This report represents LANL's documentation of our process for arriving at the recommended treatment option for RNS and UNS waste for consideration by NMED and DOE.

Finally, these recommendations have been developed based on current information and understanding of the scientific, technical, and regulatory situation at the time of writing of this report. Any significant changes to the state of knowledge in any of these areas should be followed up with a qualitative re-evaluation, or a more thorough quantitative evaluation, as appropriate.

References

Clark, D. L. and D. J. Funk, 2015. Waste Isolation Pilot Plant (WIPP): Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes. LANL Report LA-UR-15-22393.

U.S. Department of Energy Carlsbad Field Office (DOE/CBFO), 2013. TRANSURANIC WASTE ACCEPTANCE CRITERIA FOR THE WASTE ISOLATION PILOT PLANT, Revision 7.4, DOE/WIPP-02-3122, April 22, 2013.

U.S. Environmental Protection Agency (EPA, 2015). Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Revision 6, http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm.

Funk, D. J., 2014. Hazards Associated with Legacy Nitrate Salt Waste Drums under the Container Isolation Plan, LANL Internal Memo, WXDIV-14-1554, December 14, 2014.

Los Alamos National Laboratory (LANL), 2014. Revised LANL Nitrate Salt-Bearing Waste Container Isolation Plan, LANL Document LA-UR-14-23820, May 29, 2014.

Leibman, C., D. Martinez, and J.-M. Sansinena, 2015. Transuranic Remediated Nitrate Salt Waste Headspace Gas Sampling and Characterization. LANL internal report, February 17, 2015.

New Mexico Environment Department (NMED), 2014. Administrative Compliance Order HWB-14-20, December 6, 2014.

Walsh, G., 2010. Results of oxidizing solids testing, EMRTC Report RF 10-13, Energetic Materials Research and Testing Center, New Mexico Institute for Mining and Technology, Socorro, New Mexico, prepared for Washington TRU Solutions, LLC, March 12, 2010.

Appendix 1 Description of Stabilization Treatment Options

This Appendix provides additional details on the four stabilization and salt dissolution treatment options developed in the summer of 2014 in response to the recognition that a nitrate salt waste drum generated at LANL had breached in the WIPP underground (Drum 68660). The team was charged with the goal of developing potential treatment options for RNS and UNS waste using LANL facilities, taking into consideration the current state of the waste and facility readiness. Technical requirements that the team considered included the need to store and handle the waste safely before and during treatment, and the development of treatment options that would yield an acceptable final waste form for disposal at WIPP, with recognition that any proposed option will require acceptance by the regulator in the form of an approved modification of the LANL operating permit.

Below are the assumptions that the team made in order to develop the options.

1. All 60 RNS drums will be processed.

2. All 29 UNS drums will be processed.

3. Existing drums are 75% full on average.

4. Zeolite will be mixed at a ratio of 3:1 (zeolite:nitrate salt/kitty litter) by volume. (Testing most likely will change this assumption).[21]

5. Non-cemented product drums will be filled to 50% to allow for mixing.

6. Cemented drums will contain approximately 25% waste material (absorbed or otherwise), which is estimated to produce approximately 80% cemented material.

7. For RNS waste, the drums will be processed at temperatures below ambient in order to reduce chemical reaction risk during denesting and slow chemical kinetics potential, and to allow for safe and efficient denesting and handling.

8. Final forms will be tested to validate that the D001 EPA Hazardous Waste Number is no longer applicable.

9. Final forms meeting WIPP acceptance criteria will have less than 1% liquid and will not have D002 hazardous waste labeling (corrosivity) because of the

[21] A 3:1 ratio was originally selected as a way to mitigate dose because packaging of waste would have been in a pipe overpack container (POC), which is limited by dose and amount of salt that can be transported. The remediated material is significantly different than the original nitrate salts because it is an efficient mix of oxidizer and fuel. Small-scale testing will be performed to determine the appropriate ratio used to eliminate the hazards.

removal of all liquids and neutralization depending upon the treatment option chosen.

10. Temperature control would be applied to the RNS drums until treatment enables removal of the D001 hazardous waste labeling.

11. A container may be removed from the Isolation Plan upon removal of the D001 hazardous waste labeling.

12. The SWB may be considered secondary containment for corrosive liquids during transportation of a container controlled through the Isolation Plan.

13. The SWB will be considered a regulatory control during loading and shipping while a container is controlled through the Isolation Plan.

14. Remediated nitrate salt drum processing (debris segregation, splits and zeolite addition) may be performed at WCRRF.

15. Visual examination will be conducted at WCRRF for debris drum loading with controls to ensure no additional waste is added prior to cementation.

16. Cementation (neutralization, cement addition and mixing) will be performed in a new facility in Area G.

The following is a description of each stabilization option, and accompanying diagrams that were provided to the Core Team to define the options.

Option 1. Stabilization Using Zeolite

Figure A1-1 is a schematic of this option. Waste is processed by removing debris and mixing it into an inorganic matrix of natural mineral zeolite such as clinoptilolite. The resulting mixture removes the characteristics of ignitability, corrosivity, and the oxidizer potential of the nitrate salts is eliminated. The quantity of zeolite used would be determined through reactivity studies using surrogate mixtures of waste, and confirmed once the waste is sampled. For RNS waste, the drums will be cooled to allow for safe and efficient denesting and handling. Denesting would occur at Area G, and the waste would be transported to the WCRRF for processing. For UNS waste, similar processing will be conducted, but temperature control is not required because the nitrate salts without organic absorbent do not pose a safety hazard for oxidation reactions involving contents within the drum (Funk, 2014). The figure is annotated with markers denoting the operational and regulatory steps that would be performed at various stages of the process.

For RNS waste, based on the assumptions of a 3:1 ratio of zeolite to waste, an assumed average volume in each drum, and 50% fill of the new daughter drums, this option is calculated to produce 399 daughter drums including the original empty drums.

Option 2. Stabilization Using Zeolite

Figure A1-2 is a schematic of this option. Waste is processed identically to Option 1 up to and including zeolite addition, ensuring ignitability and corrosivity characteristics are removed. The waste is now considered non-oxidizing, and removed from temperature control. The material is then further treated through a process that includes water addition, neutralization, and cementation to produce monoliths that would be suitable for transportation and disposal when the D001/D002 characteristics are removed. Waste transport occurs from Area G to WCRRF for zeolite addition, and, in the process evaluated at WCRRF. Then, the containers are transported back to Area G for cementation in a new facility. For UNS waste, similar processing will be conducted, but without temperature control. The figure is annotated with markers denoting the operational and regulatory steps that would be performed at various stages of the process. For UNS waste, a similar process would be conducted, but without temperature control.

For RNS waste, based on the assumptions of a 3:1 ratio of zeolite to waste, and an assumed average volume in each drum before and after cement addition, this option is calculated to produce 798 daughter drums, including the original empty drums and debris drums. The 3:1 ratio was based on possible dose. The actual ratio will be determined by the treatment studies.

Option 3. Stabilization Using Dry-Process Cementation

Figure A1-3 is a schematic of this option. Waste is moved to WCRRF and processed by removing debris and splitting it into smaller quantities suitable for subsequent treatment. The waste is transported as a dry material to a new cementation unit (assumed to be at Area G) where it is processed through the addition of water, neutralization, and cementation to produce monoliths that would be suitable for transportation and disposal. Temperature controls are removed at the point at which water is added. The figure is annotated with markers denoting the operational and regulatory steps that would be performed at various stages of the process. For UNS waste, a similar process would be conducted, but without temperature control.

For RNS waste, based on the assumptions of the average volume in each drum before and after cement addition, this option is calculated to produce 285 daughter drums, including the original empty drums and debris drums.

Option 4. Stabilization Using Wet-Process Cementation

Figure A1-4 is a schematic of this option. Waste is processed by cementation as in Option 3, but with water addition early in the process, rendering the mixture absent of ignitability characteristics. At that point, temperature control is removed. The waste is then transported wet to WCRRF for segregation and splitting followed by transportation of daughter drums back to Area G to a new cementation unit where it is processed by neutralization and cementation to produce monoliths that would be

suitable for transportation and disposal. The figure is annotated with markers denoting the operational and regulatory steps that would be performed at various stages of the process. Because the early addition of water is a safeing strategy designed specifically for the RNS waste and thus is unnecessary for UNS waste, this option is only applicable for RNS waste.

For RNS waste, based on the assumptions of the average volume in each drum before and after cement addition, this option is calculated to produce 342 daughter drums, including the original empty drums and debris drums.

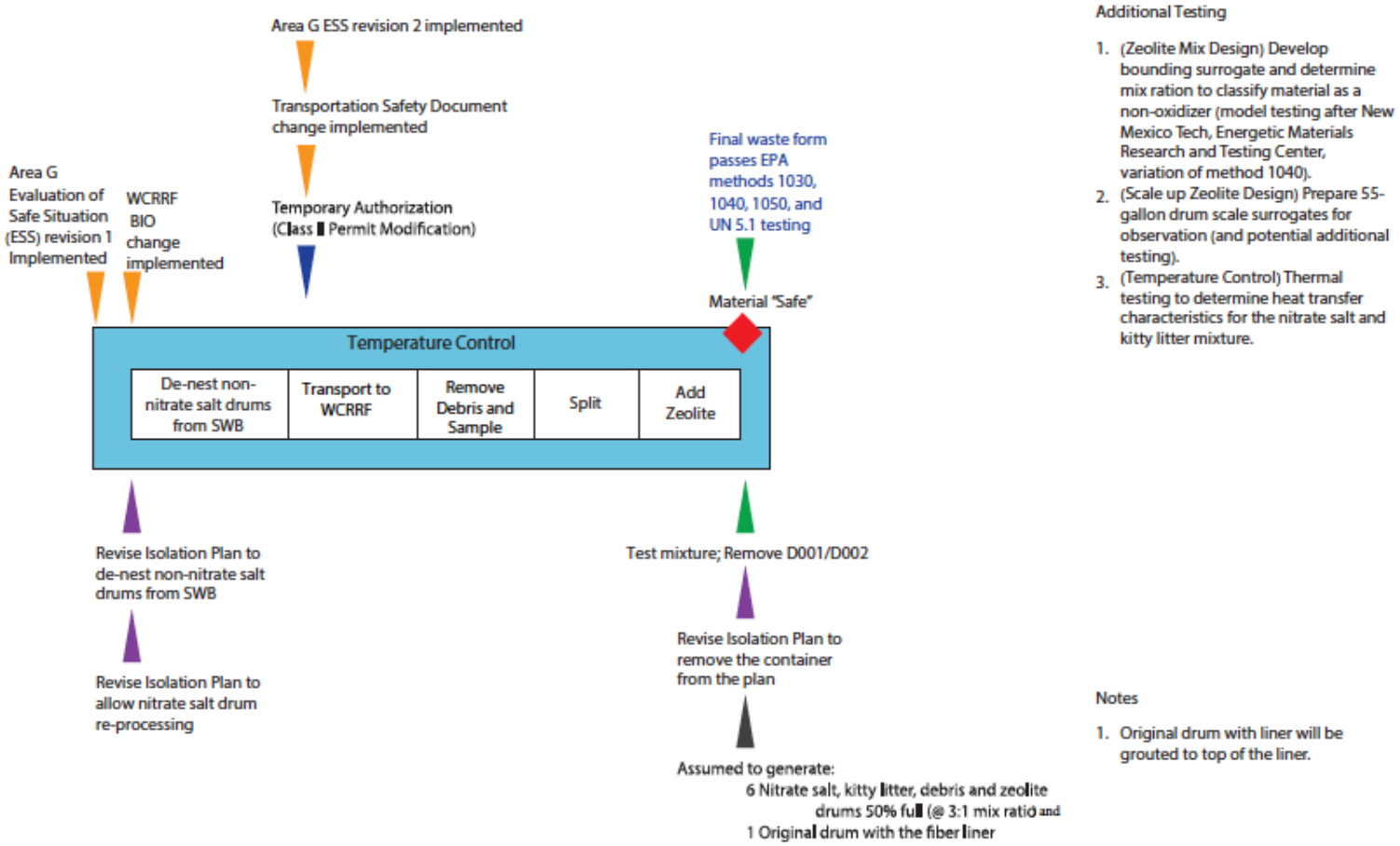
Option 14. Salt Dissolution with Cementation/Stabilization

Figure A1-5 is a schematic of this option. Waste is processed by removing debris, filtering the nitrate salt with water and separately capturing the Swheat™ during the filtration process. Temperature control is removed when the early addition of water occurs. The nitrate solution is neutralized and cemented to produce monoliths that would be suitable for transportation and disposal. The Swheat™ cake is pressed to remove excess water and also cemented for transportation and disposal.

For RNS waste, based on the assumptions of the average volume in each drum before and after cement addition, this option is calculated to produce 285 daughter drums, including the original empty drums and debris drums.

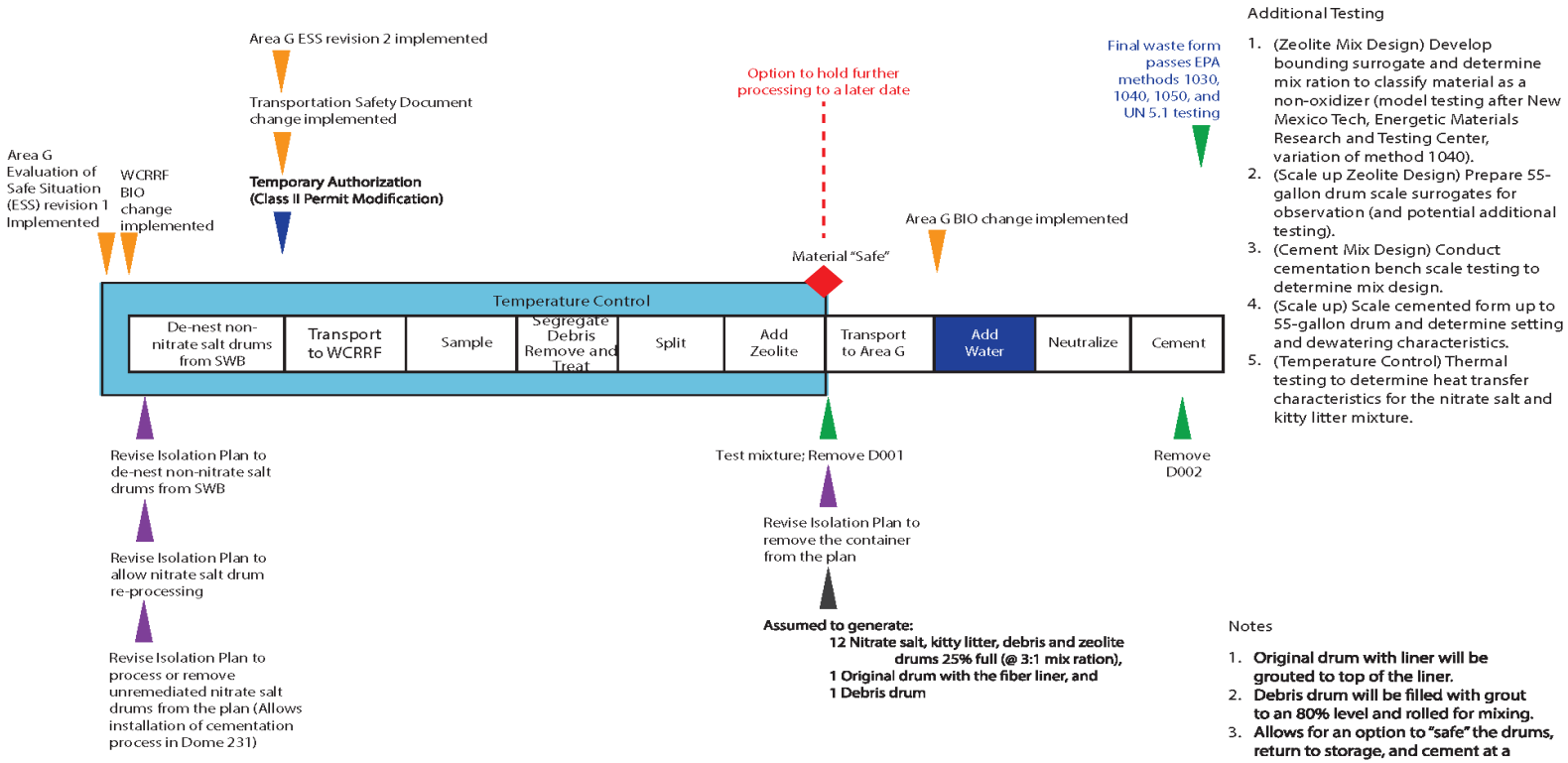
The number of daughter drums is dependent upon the repulp options chosen. The no repulp option would produce 4 daughter drums of cemented waste and another partial drum of debris (plastic bags and liner material) from the original drum. If the repulp option is chosen, 6 drums per waste would be generated, resulting in 342 drums.

Figure A1-1. Schematic of Option 1 (Stabilization Using Zeolite)



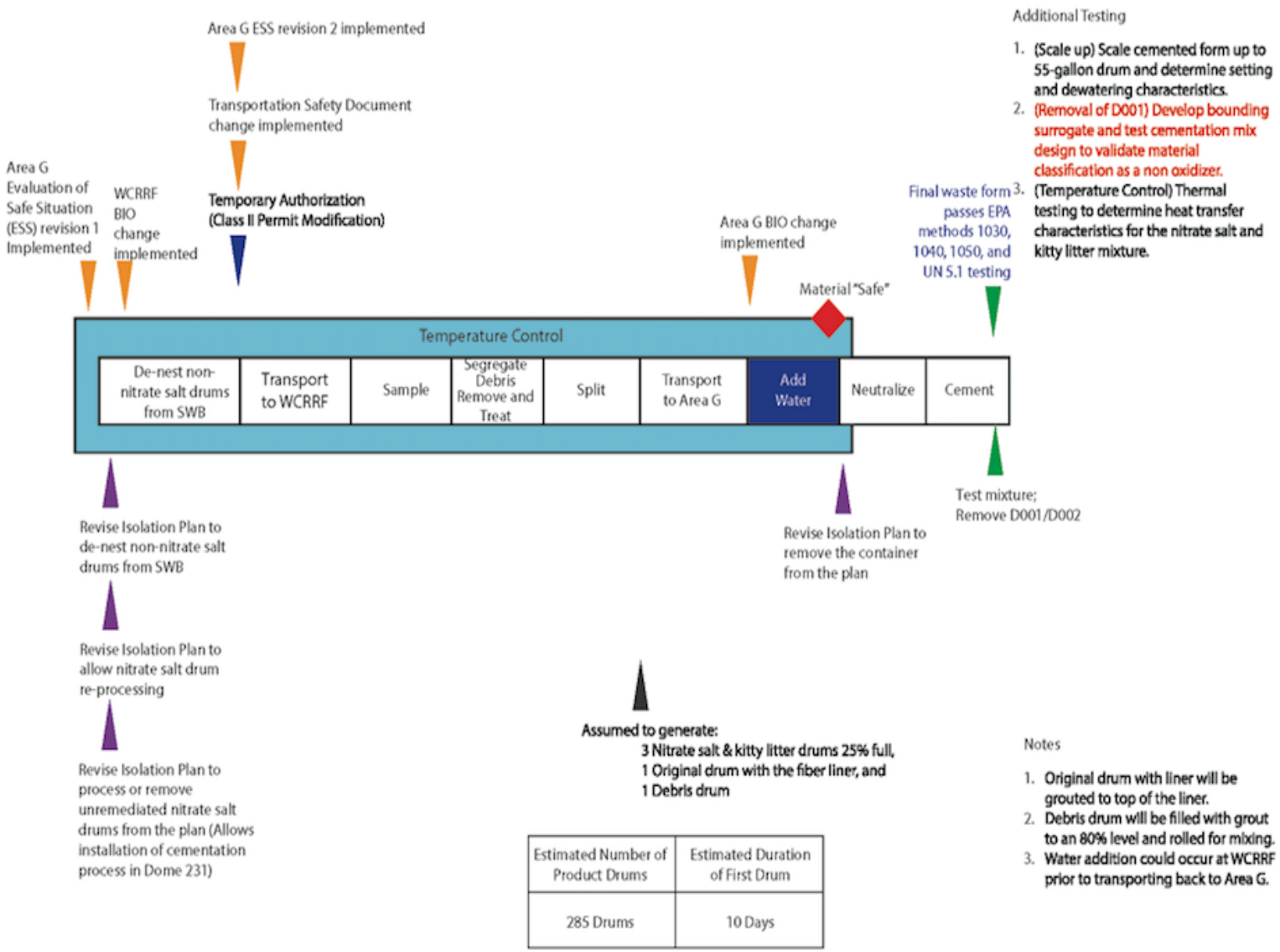
Estimated Number of Product Drums	Estimated Duration of First Drum
399 Drums	4 Days

Figure A1-2. Schematic of Option 2 (Stabilization Using Zeolite With Cementation)



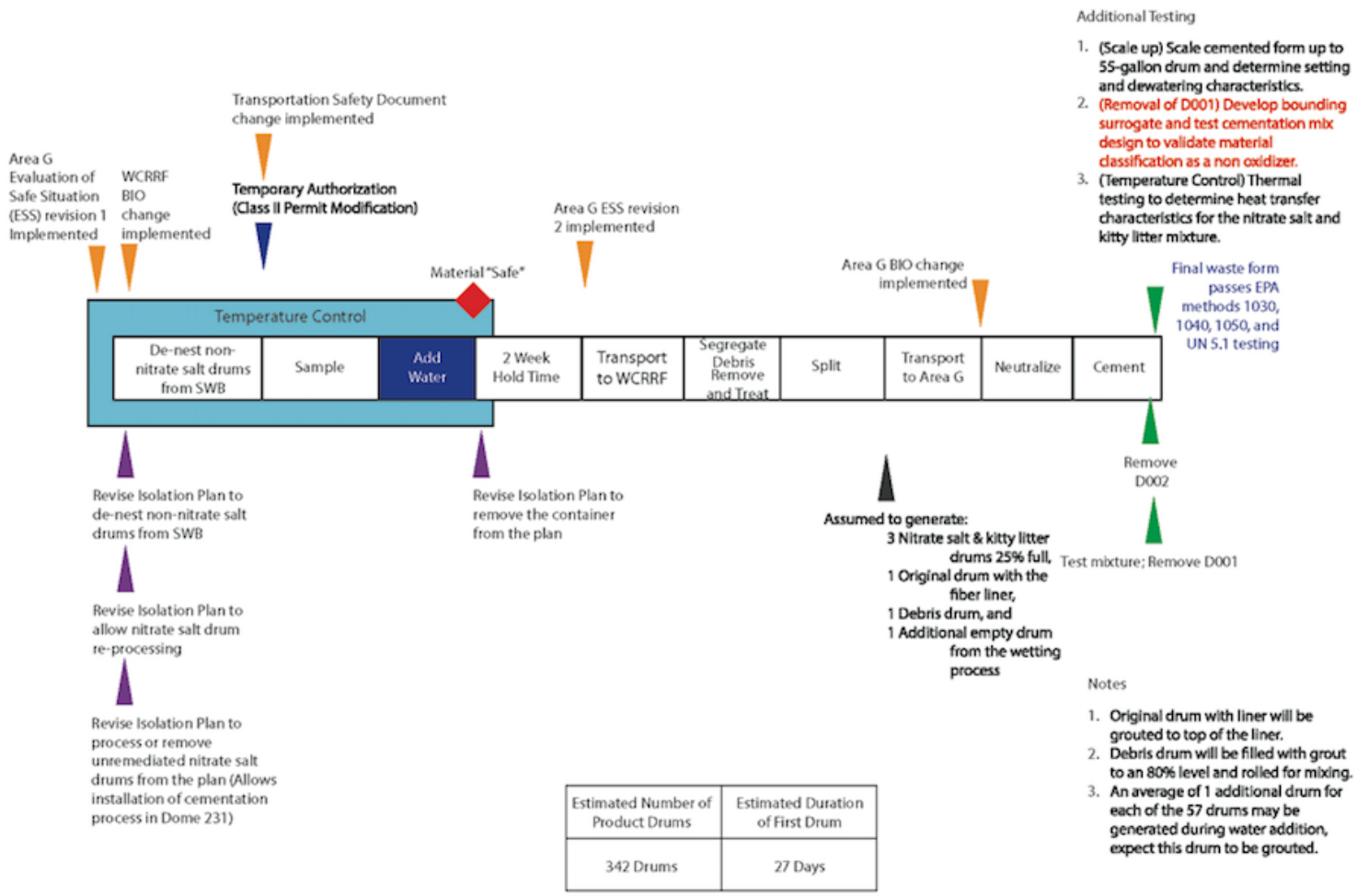
Estimated Number of Product Drums	Estimated Duration of First Drum
798 Drums	29 Days

Figure A1-3. Schematic of Option 3 (Stabilization Using Dry-Process Cementation)



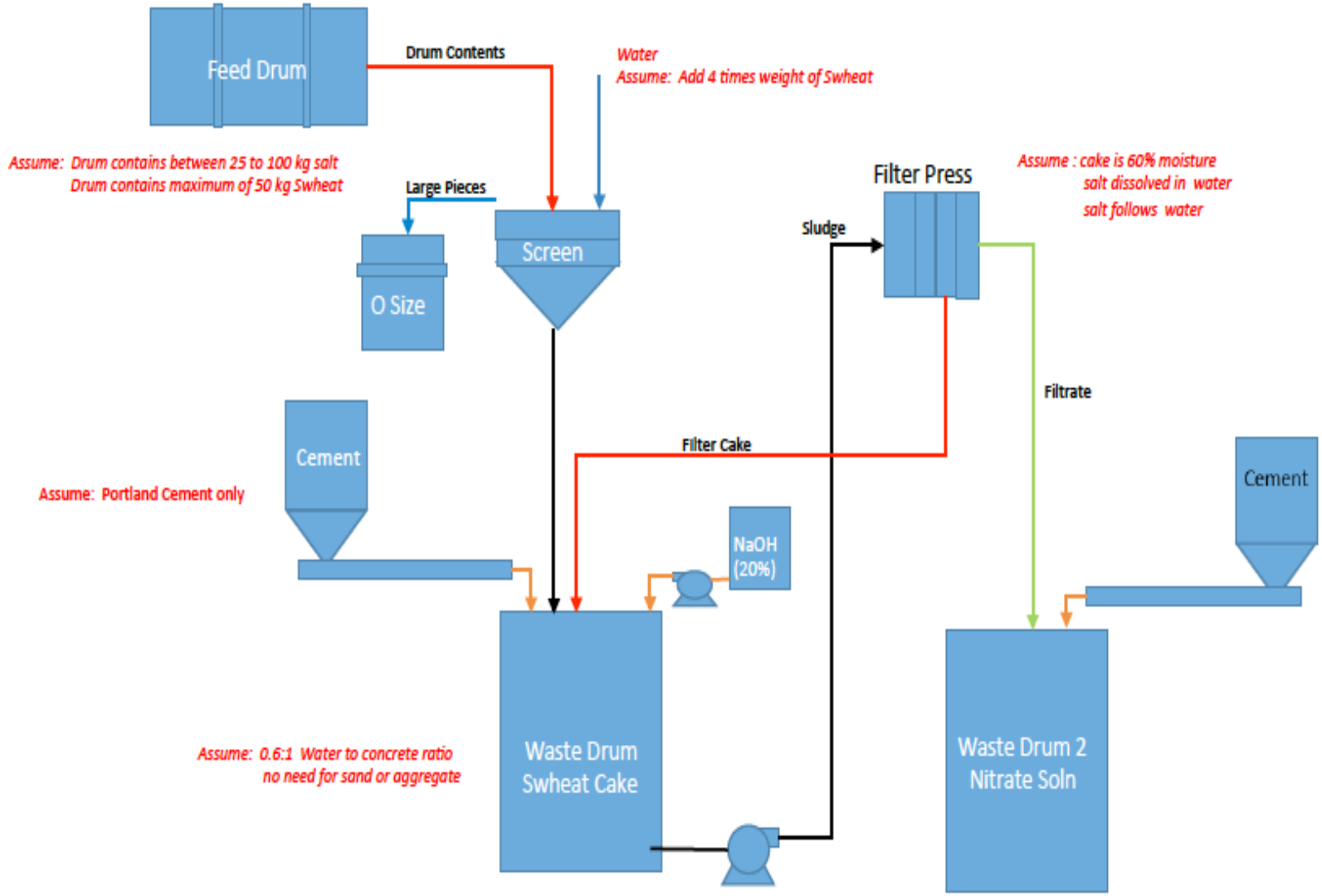
Estimated Number of Product Drums	Estimated Duration of First Drum
285 Drums	10 Days

Figure A1-4. Schematic of Option 4 (Stabilization Using Wet-Process Cementation)



Estimated Number of Product Drums	Estimated Duration of First Drum
342 Drums	27 Days

Figure A1-5. Schematic of Option 14 (Salt Dissolution With Cementation/Stabilization)



Appendix 2. List of Core Team Members and Others Participants in the Options Assessment Activity

Remediation Integrated Team Members

ADEP Management	**Randall Erickson**
Lead	**Bruce A. Robinson**
Technical Input	
Energetic Chemistry	Philip Leonard
Actinide Chemistry	Stephen L. Yarbro
Cementation	Robert M. Wingo
Technical Advisors	David L. Clark
	David J. Funk
Operations	Scotty A. Miller
	James (Steve) S. Clemmons
	Kurt R. Anast
TA-55 Waste Expert	Kapil Goyal
FOD	Rick A. Alexander
	Andrew R. Baumer
	Charles E. Conway
Regulatory	Gian A. Bacigalupa
	John K. Hopkins
ES&H	Robert (Bob) C. Stokes
Safety Basis	Ronald D. Selvage
Quality Assurance	Faris M. Badwan
Carlsbad DWT	Timothy P. Burns
	Christopher J. Chancellor
	Timothy A. Hayes
Benchmarking	Enrique (Kiki) Torres
Project Management	Patrice A. Stevens

Remediation Team Reviewers	
High Explosives Science and Technology	Philip Leonard
Explosive Science and Shock Physics	Gary R. Parker
Environmental Compliance	Paul B. Schumann
Actinide Science and Engineering	D. Kirk Veirs
Environmental Compliance	Luciana Vigil-Holterman

Appendix 3. Evaluation Criteria Descriptions

The following set of definitions of the evaluation criteria presented in Table 5-1 was developed by the Core Team and used in its deliberations on potential treatment options. Instances in which criteria were adjusted or interpreted differently during the evaluation meeting are described in the evaluation write up (Section 6.2).

Criterion 1. Robust to Waste Stream Variability

A ranking of the ability to effectively treat all items potentially in the waste stream. This would include the need for separation, pretreatment or chemical compatibility with each of the items in the waste stream, accounting for potential differences in chemical composition from drum to drum. A procedure must be written that is robust enough to meet all potential waste streams. Note: if a process can be easily adapted to treat both the RNS and UNS waste streams, that benefit should also be factored into this criterion. Range: 1 – extremely difficult to develop a robust process, 5 – highly likely to be a robust process.

Criterion 2. Ease of Permitting (Permitting Difficulties)

The relative ease of obtaining permit approval from NMED, evaluating factors such as the regulator's familiarity with the treatment process, whether the process is used elsewhere at the facility, the overall technical complexity and maturation of the process, the need for associated risk assessments, degree of associated changes to the RCRA permit, and potential for stakeholder opinion. Range: 1 – extremely difficult to permit, 5 – simple permitting process.

A more precise definition of the scoring system used for this criterion was provided to the Core Team.

1 – Class 3 permit modification request with public hearing (Approval process with NMED could take three years or longer because of perceived technical complexity, significant public opposition, and need for extensive negotiations with stakeholders).

2 – Class 3 permit modification request without public hearing (Approval process could take two years).

3 – Class 2 permit modification request (Approval process one year if treatment process is common or less technically significant).

4 – Class 1 permit modification request with NMED approval (Short approval time by NMED without public input if treatment process is relatively simple, similar to previously approved processes, and/or previously coordinated with NMED).

5 – Class 1 permit modification notification without NMED approval or the treatment process can be included in an NMED compliance order without permitting.

NOTE: This range is based on the availability of distinguishing permit mod types in 40 CFR 270.42, Table I. NMED has the option to make any permit modification a higher class based on technical complexity or public interest.

Criterion 3. Safety Basis Challenges

The relative ease of obtaining Safety Basis approval. Factors include facility constraints such as facility features needed for protection from radioactivity. Another factor would be the degree of procedure development needed to ensure that requirements for worker safety are met. Range: 1 – extremely complex safety basis challenges, 5 – straightforward safety basis approval process.

Criterion 4. Extent of Testing Required

A review of the amount and complexity of sampling and analysis required to implement the treatment process. Significant factors will include the need for testing the waste prior to treatment, testing associated with developing operational parameters for the treatment process, operational testing during treatment, and final testing to assure the treatment process is effective. Testing must be sufficient to prove the technical viability of the treatment process. If a process is judged to be technically infeasible, then it will be screened out during the pre-screening phase. 1 – very onerous testing required, Range: 5 – straightforward testing required.

Criterion 5. Reduction of Toxicity, Mobility, Corrosivity, and Ignitability

The ability of the treatment process to provide reductions in toxicity, ignitability, corrosivity, and mobility of the final waste form. This would include factors such as level of ignitability of the final waste form, its ability to prevent releases, and the ability to package the final waste form. Range: 1 – marginally effective waste form and/or difficult to package 5 – highly effective waste form and straightforward to package.

Criterion 6. Reduction of Volume

Reductions in the volume of the final waste form due to the treatment process. This would include the ability to minimize volume of the final waste form including the number of daughter drums generated from the treatment. Range: 1 – large volume and/or large number of daughters generated 5 – low volume with low numbers of daughters generated.

Criterion 7. Short Term And Long Term Effectiveness

A review of the treatment process to evaluate whether the treated waste stream can meet the WIPP WAC, including prevention of future dewatering. Another factor will

be the potential for the treated final waste stream to develop future biological/chemical problems such as degradation of entrained items or chemical compatibility. Range: 1 – effectiveness is questionable or indeterminate, 5 – highly effective.

Criterion 8. WCS Implications

A review of the difficulty of implementing the treatment option for the nitrate salt waste drums at Waste Control Specialists at Andrews, Texas. Evaluation includes the need for transportation of drums to Los Alamos to treat, versus implementing the treatment process on site. Range: 1 – extremely difficult to implement for WCS drums, 5 – straightforward to implement for WCS drums.

Criterion 9. Scalability and Complexity

The ability to treat RNS and UNS waste drums using the available sites and facilities at LANL, including the complexity of the remediation process for either type of drum. This includes the complexity and number of steps required to treat the waste, and whether engineering controls are available to meet ALARA in accordance with DOE and LANL requirements. Range: 1 – extremely difficult to implement for drum remediation 5 – straightforward to implement for drum remediation.

Criterion 10. Facilities Challenges

Ability to use available site and facilities that are currently operating under the LANL approved Authorization Basis (AB) scope. Range: 1 – extremely difficult to implement due to AB scope 5 – highly likely to implement under current LANL AB status.

Criterion 11. Schedule

A review of time constraints, evaluating schedule factors such as treatment process facility design complexity, staffing requirements, project and procedure development, permitting approvals, and compliance schedules. Range: 1 – extremely time consuming, 5 –expedited schedule is achievable.

Criterion 12. Cost (not a primary Evaluation Criterion; can be used as a final discriminator)

A review of financial constraints, evaluating cost factors such as treatment process facility design complexity, required facility modifications, and staffing requirements. Range: 1 – extremely expensive, 5 – cost-effective option.

Appendix 4

The Path to Nitrate Salt Disposition

LA-UR-16-21760
Approved for public release; distribution is unlimited.

Title: The Path to Nitrate Salt Disposition

Author(s): Funk, David John

Intended for: Report

Issued: 2016-03-16

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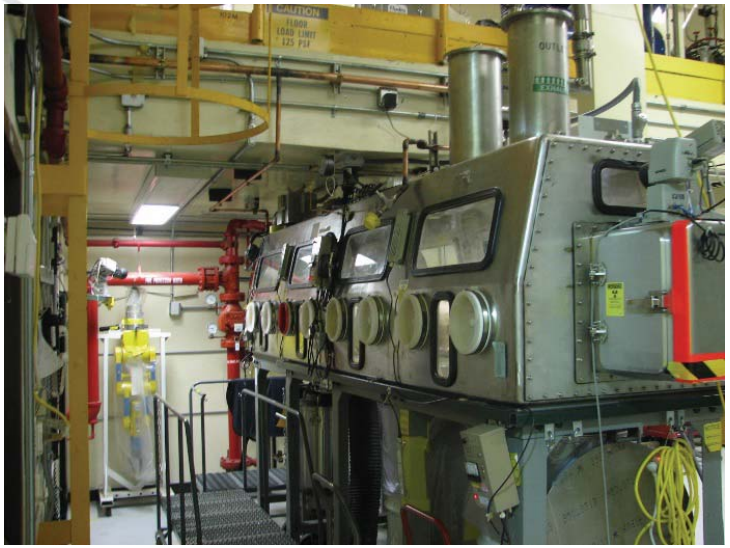
The Path to Nitrate Salt Disposition

Dave Funk

February 22, 2016

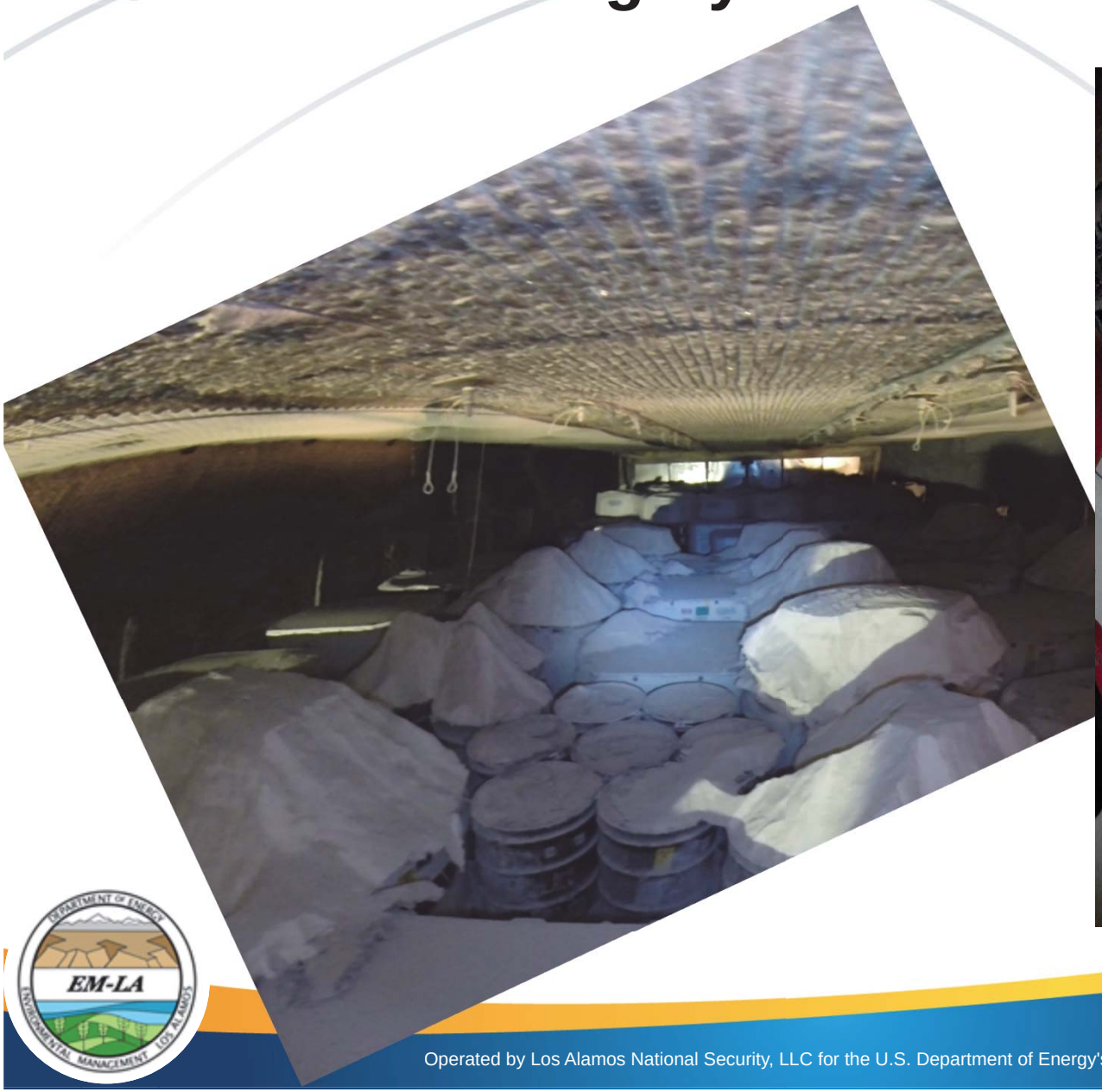


LA-UR-16-21760

Outline

- LANL Nitrate Salt Incident as Thermal Runaway
  - Thermally sensitive surrogates
  - Full scale tests
- Temperature Control for Processing
- Treatment Options and Down Selection
- Assessment of Engineering Options
- Anticipated Control Set for Treatment
- Summary of the Overall Steps for RNS Treatment

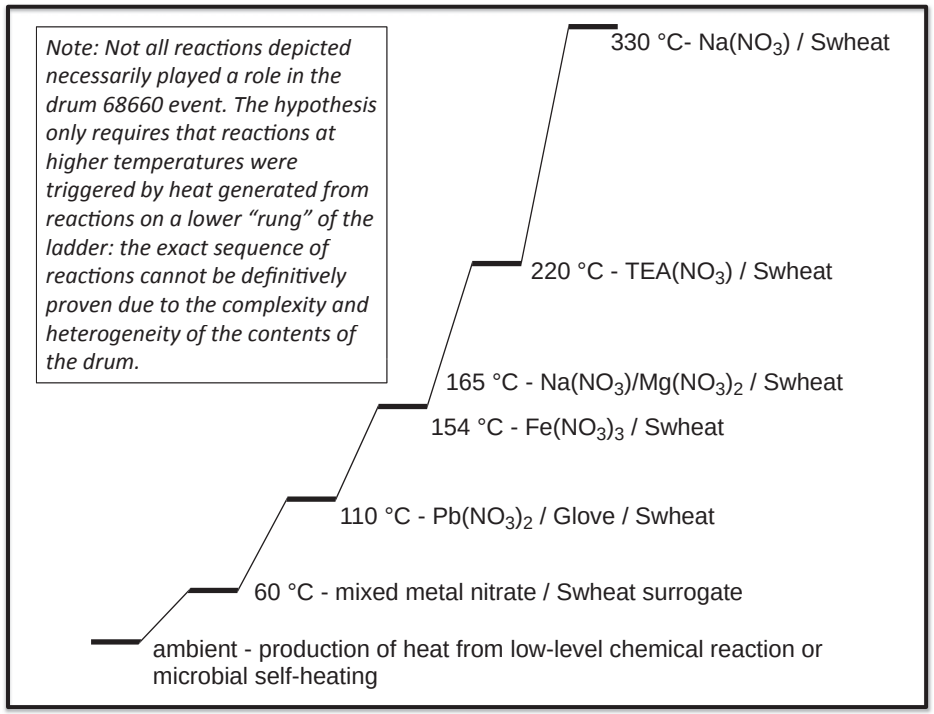
Incident was identified by rad release and imagery from the mine



LA-UR-16-21760

Summary Description of the LANL Nitrate Salt Incident

- The incompatibility of the nitrate salt (oxidizer) and Swheat kitty litter (fuel) mixture, created the potential for thermal runaway that was ultimately realized when Drum 68660 pressurized and breached

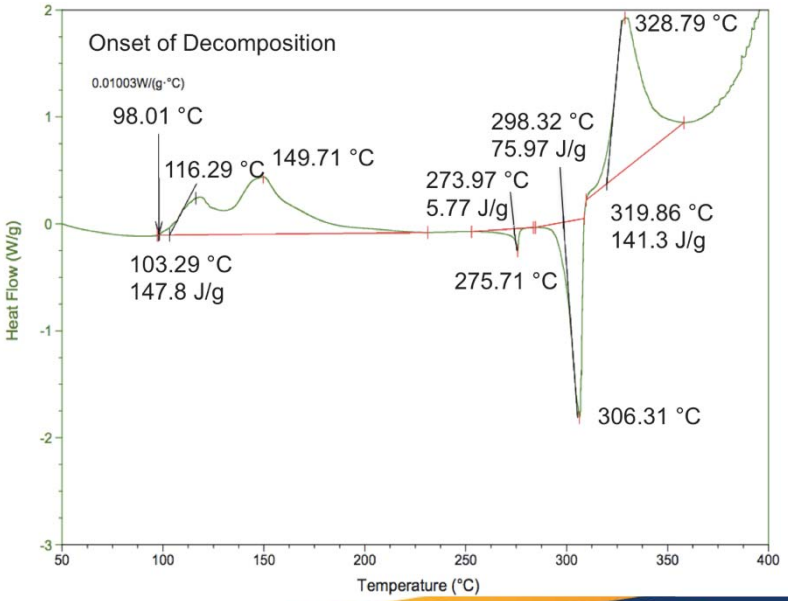


- Production of heat, either from low-level chemical reactions or the growth of natural microbes, in concert with mixed metal nitrate salts, bismuth lined glovebox gloves and/or lead nitrates when combined with the Swheat organic kitty litter, generated a series of exothermic reactions that heated and pressurized the drum resulting in the venting of high-temperature gases and radioactive material into the room.

Our current thinking: the chemical incompatibility lead to thermal runaway through low temperature reactions

Technical studies identified increased nitrate salt/swheat thermal sensitivity with complex mixtures

- $Na(NO_3)$/Swheat – 330 °C
- $HTEA(NO_3)$/Swheat – 220 °C
- $Na(NO_3)/Mg(NO_3)_2$/Swheat – 165 °C
- $Fe(NO_3)_3$/Swheat – 154 °C
- Pb/TEAN/Swheat – 110 °C
  - 1M HNO_3 – no change in decomposition onset
  - 8M and 16 HNO_3 – new exotherm
- Bi-lined glove/Nitrate/Swheat – 110 °C
- Bi-lined glove/TEAN/Sweat
  - 1M HNO_3 – no change in decomposition onset
  - 8M and 16M HNO_3 – new exotherm



LA-UR-16-21760

Thermochemical modeling of processes yielded most sensitive surrogate salt mixtures

- Stream Analyzer (OLI) software used to model the evaporator processes
- The derived mixtures of metal nitrate salts with Swheat show:
  - Low exotherm temperatures ***30-55 °C***
  - Evidence of incompatibilities leading to decomposition and NO, NO_2 evolution, followed by Swheat nitration (as high as 6-7%)
  - Material that exhibits some electrostatic discharge sensitivity
  - Mg, Fe, and Pb appear to be the main contributors to these processes
- Prepare actinide sample through spiking, use of UNS samples

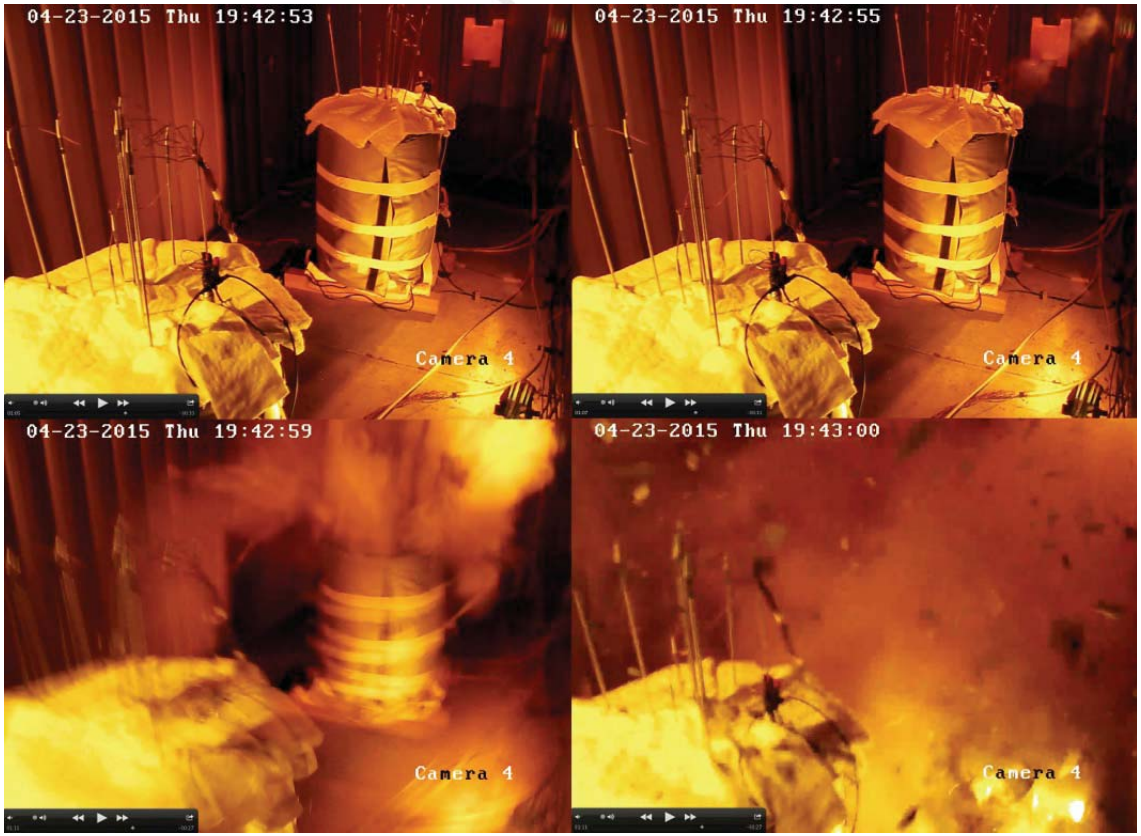
Table 1. Important metal ion concentrations (median values) in evaporator bottoms from Veazey, et al.[2] (in g/l)

	Ion Exchange	Oxalate Filtrate
Ca	61	10.5
Mg	58.7	13.3
K	17.6	4.8
Fe	17.0	7.9
Na	7.4	23.9
Al	4.6	2.3
Cr	3.0	1.94
Ni	1.8	1.205
Pb	0.19	0.056

Veazey, G. W.; Castaneda, A. *Characterization of TA-55 Evaporator Bottoms Waste Stream*; NMT-2:FY 96-13; Los Alamos National Laboratory: Los Alamos, NM, 1996

The full scale drum tests were of significant technical value

- Requested by the AIB to support their investigation
- A goal was to demonstrate that we have an understanding of the mechanisms by which the 68660 breach may have occurred
- Provided valuable insight to guide the storage and processing of existing nitrate salt bearing drums processed with Swheat



The Thermolytic Response of a Surrogate RNS Waste Mixture at the Drum Scale

Gary Parker, Matt Holmes, Eric Heatwole and Peter Dickson
M-6, HE Thermal and Mechanical Response Team

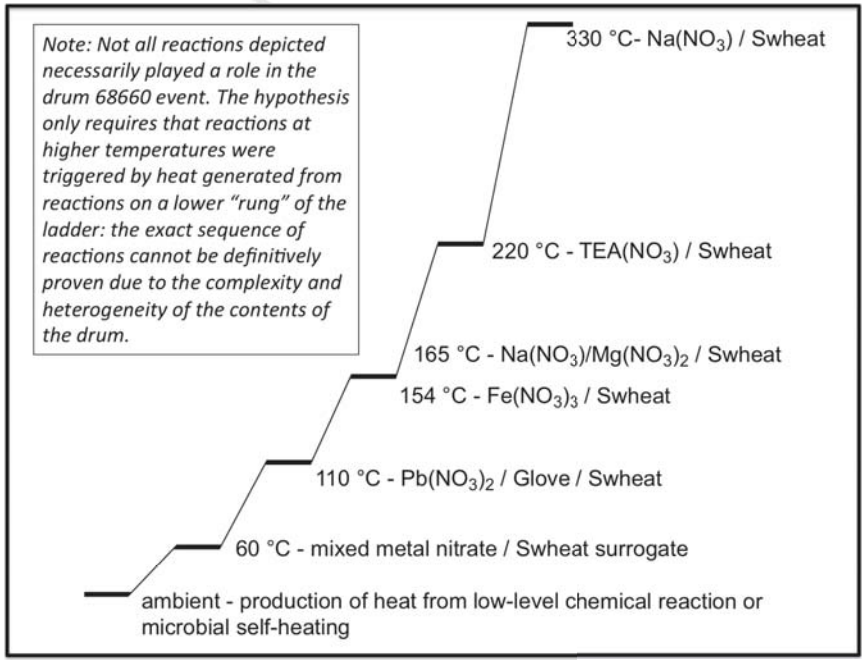
Phil Leonard
M-7, HE Science and Technology

Chris Leibman
C-CDE, Chemical Diagnostics and Engineering

LA-UR-16-21760

Technical Objectives

- Perform long duration, drum-scale tests with a plausible surrogate and physical arrangement.
- Test hypothesized “ladder” of plausible exothermic reactions
- Diagnose the thermal response of the drums; evaluate the effect of compositional inhomogeneity
- Evaluate the effect of pressure
- Perform headspace gas compositional analysis
- Record video and audio

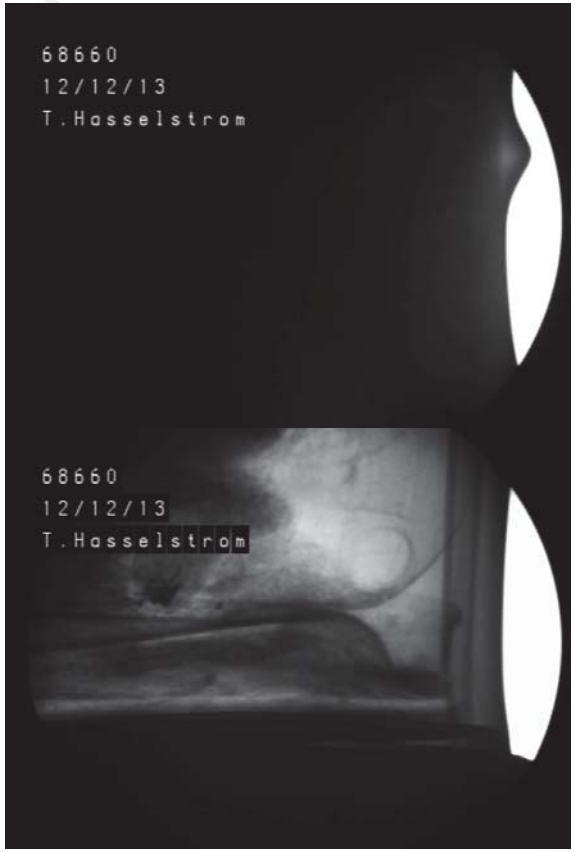


Demonstrate Thermal Runaway from Plausible Initial Conditions

- Be reasonably faithful to the drum contents
  - Variety of nitrate salts
  - Swheat Scoop pet litter
  - No radioactive components
  - Include Pb
  - Liquid neutralized with Kolorsafe Spilfyter
    - Generates triethanolammonium nitrate (TEAN)
- Include known components and prep as expected in WCRRF (layers)
  - Bi-W-La gloves, Spilfyter container

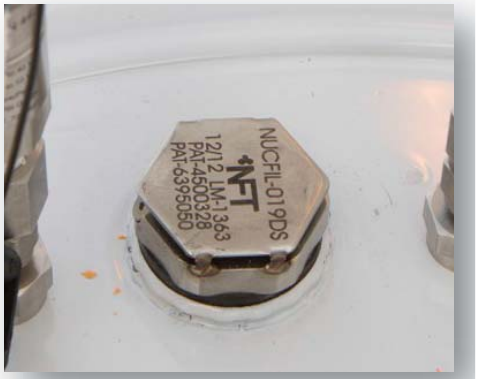
LA-UR-16-21760

Sketch of Contents, Based on RTR



Experiment Variables

1. **Boundary Temperatures: 25˚C and 60˚C**
   - Average temperature at WIPP is ~25˚C.
   - Use elevated boundary temperature of 60˚C as an accelerated rate test.
     - Long term chemical activity or biological activity could have elevated the temperature.
2. **Pressure: Vented and Sealed**
   - Standard drum configuration contains a “nucfil” filter with a carbon frit, designed to allow gas escape and prevent pressurization.
   - #68660 may have become sealed:
     - Permeability of carbon filter is insufficient for high flow-rates.
     - Internal PVC plastic bag liner may have sealed against the outlet.
     - Bags of Magnesium Oxide piled on top covered/sealed filter outlet.
     - Solids/liquids/condensation produced from chemical activity may have clogged the carbon filter.
3. **Chemical Composition: Weisbrod-8 (fixed)**
   - Selected—based on reactivity—to be most likely to result in a violent outcome, yet still a plausible composition for 68660.



Test Matrix

	Drum A	Drum B	Drum C	Drum D
Temperature	25˚C	25˚C	60˚C	60˚C
Pressure	Vented	Sealed	Vented	Sealed

Nominal for WIPP

Accelerated case

- Conditions are bounding with respect to temperature and pressure.
- Surrogate formulation is not bounding, but is plausibly reactive

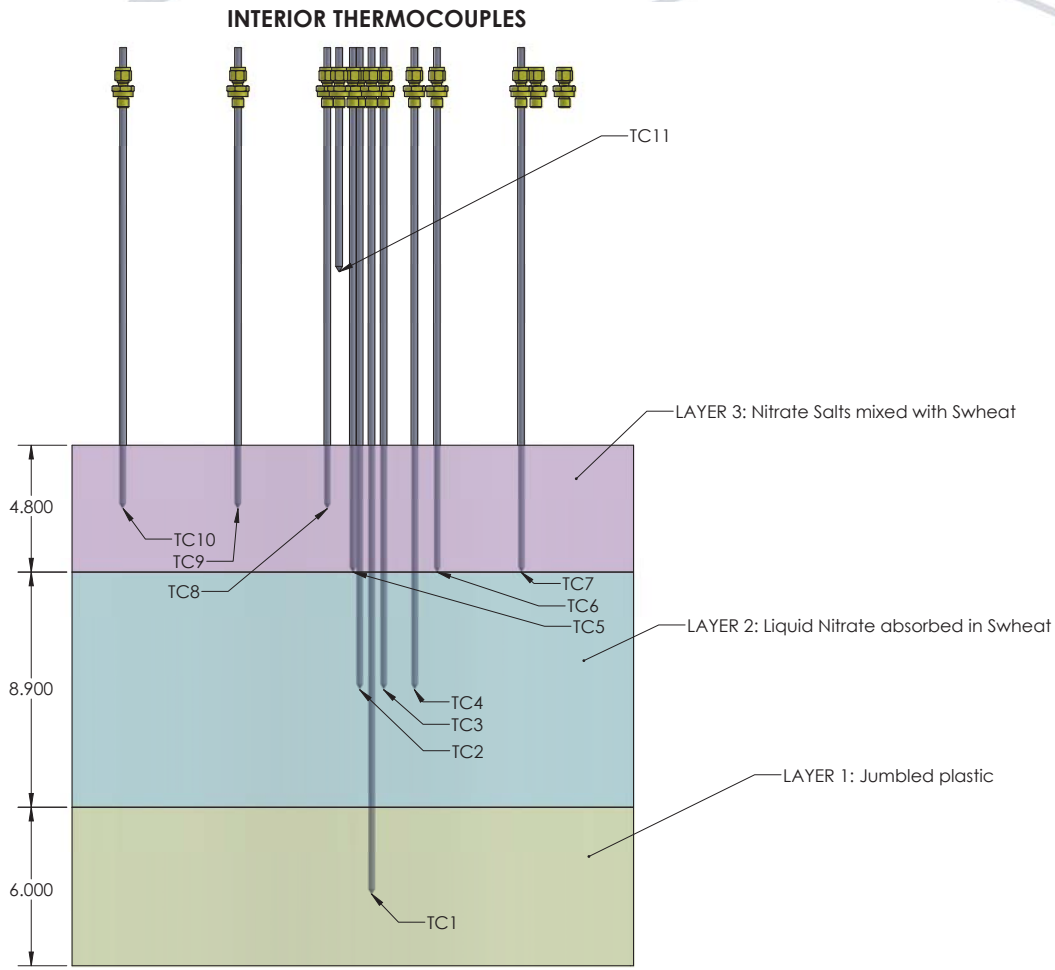
Experiment Layout

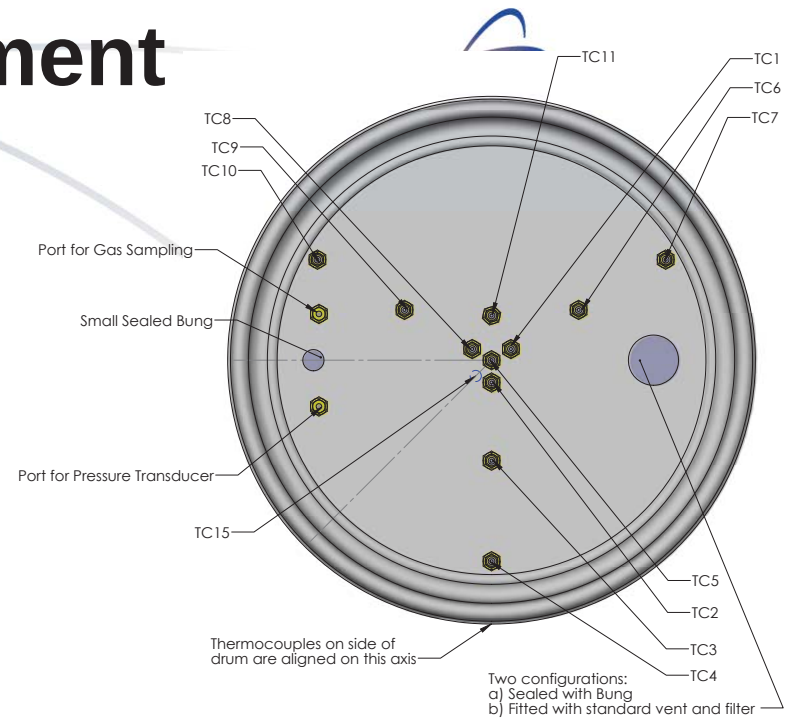
Two transportainers, two drums in each transportainer

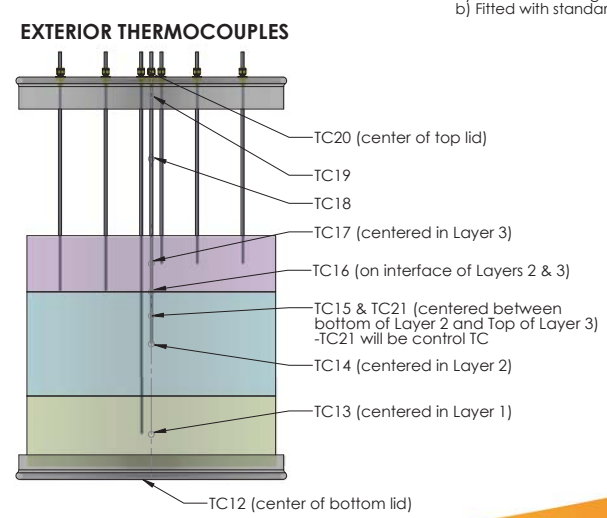
Diagnostics

- Temperature
  - 21 thermocouples on each drum, 2 air temperature TCs
  - Spatially and temporally resolve the thermal response
- Pressure
  - Static transducer to measure the drum pressure as product gases evolve
  - Dynamic transducer to quantify the dynamic response
  - Ambient static pressure gauge monitoring barometric pressure of container
- Video
  - Eight surveillance cameras with constant real-time footage recording.
  - Overview surveillance of transportainers and surrounding environment
- Headspace gas sampling
  - Conducted remotely through a ~30ft tubing run

Thermocouple Arrangement

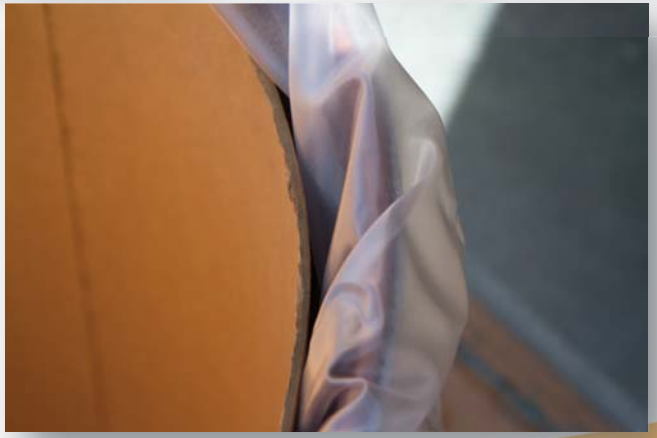






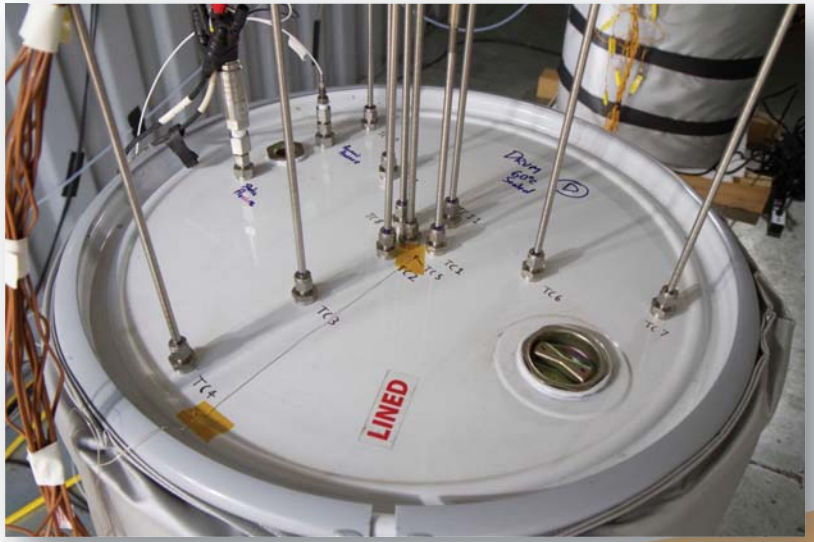
Three layer composition, filling ~60% of barrel
(derived from RTR of 68660)

Drum Preparation Photographs



Drum Preparation Photographs (cont.)

Lid was fastened with a clamping ring that was bolted closed with a specified torque. Lid seal functioned within design spec, withholding pressure of 30 +/- 3 psi.

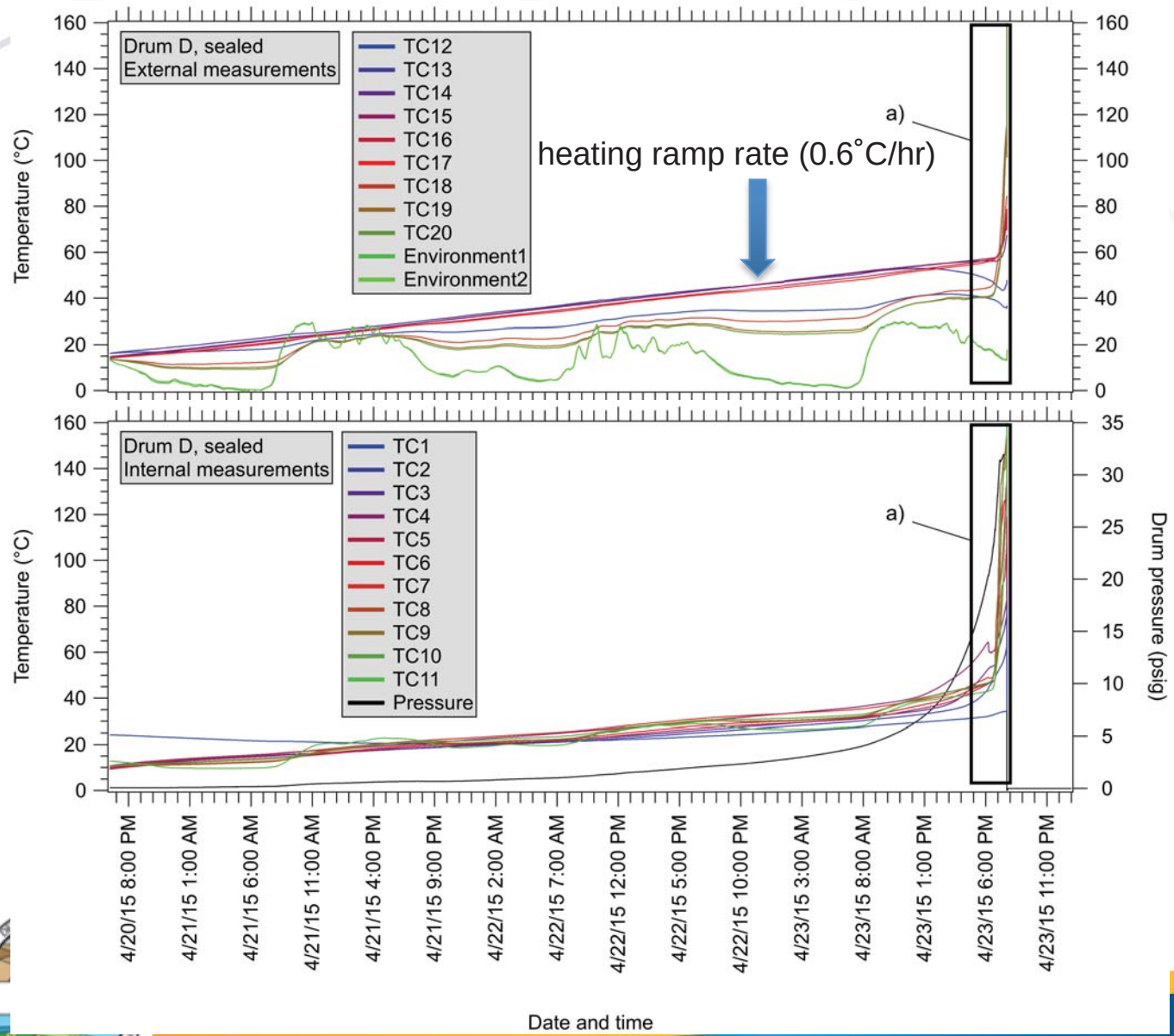


Drum Preparation Photographs (cont.)

Results

Drum D

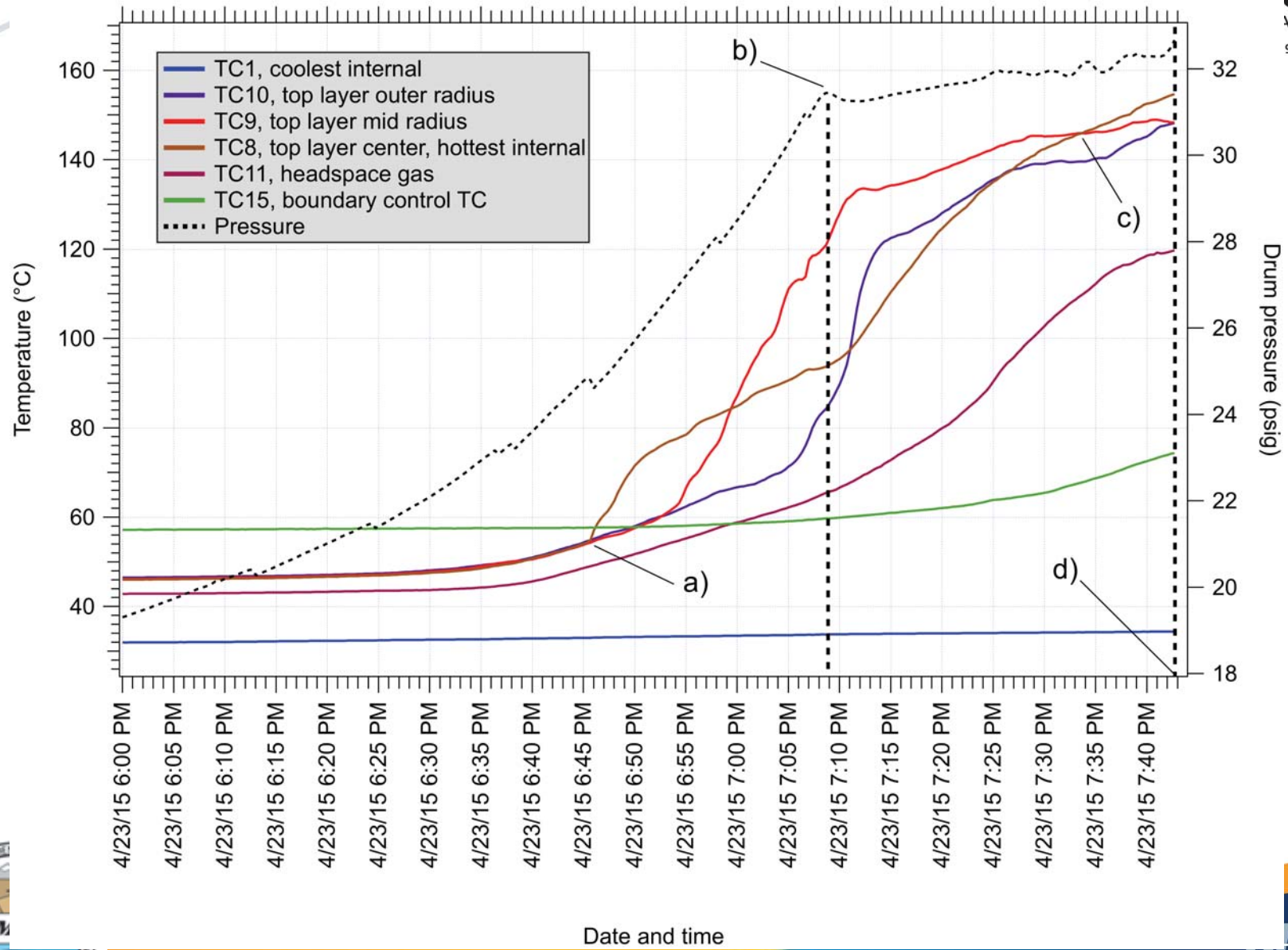
Drum D (60˚C, sealed): Full dataset



This purpose of this test was to explore a drum with a blocked vent and at elevated temperature to jumpstart and accelerate the chemistry.

LA-UR-16-21760

Drum D (60C, sealed)—Detail



LA-UR-16-21760

Drum D: Post-Mortem Images

Drum D: Summary

- Pressure burst occurred in ~3 days (73.3 hrs) in a sealed & heated configuration with the SFWB-8 composition
  - Physical explosion
- Event precursors:
  - Noticeable bulging of lid and base (slowly over the ~3 hrs prior to burst)
  - Considerable fumes (~30 mins prior to burst)
  - Audible indication (~30 mins prior to burst)
- No flame was observed during the burst
- Lid seal failed in a controlled manner at 32 psi, maintaining pressure

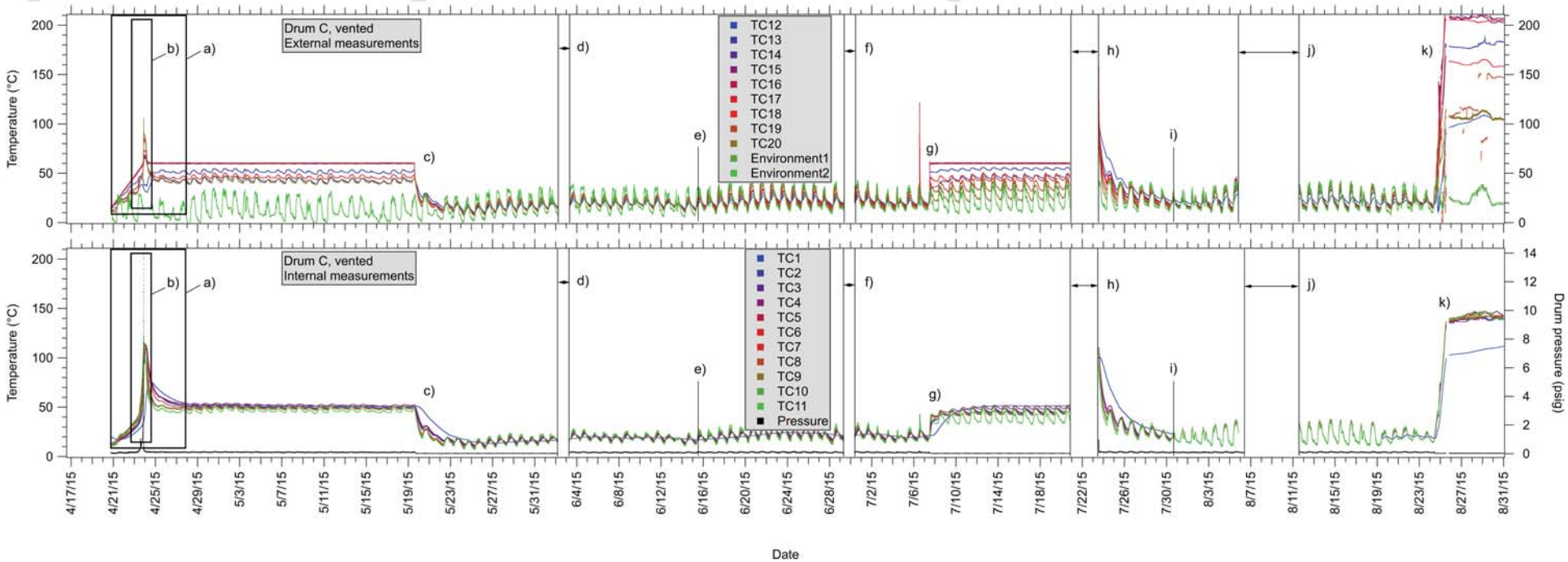
Drum D: Discussion

- When lid seal vented, thermal runaway slowed
  - Evidence for importance of gas-phase reactants on exothermic chemistry
  - Orange vapor is evidence for NO_2 production
- Hottest location in top layer, high headspace gas temp.
  - Also evidence for the importance of gas-phase reactants
- No scorching. Did not get as hot as Drum 68660
  - Did not have MgO sacks weighing down the lid
    - Reaction was quenched when lid blew off, what if it had been held in place?
  - Surrogate might have had more H_20 than drum 68660
    - Heat capacity and latent heat of vaporization

Results

Drum C

Drum C (60°C, vented): Full dataset

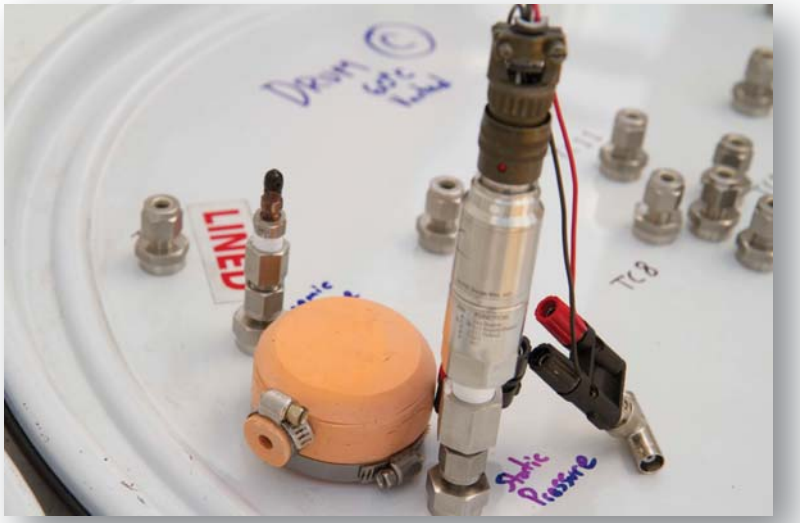


This purpose of this test was to explore a drum with a normal vent, but at elevated temperature to jumpstart and accelerate the chemistry

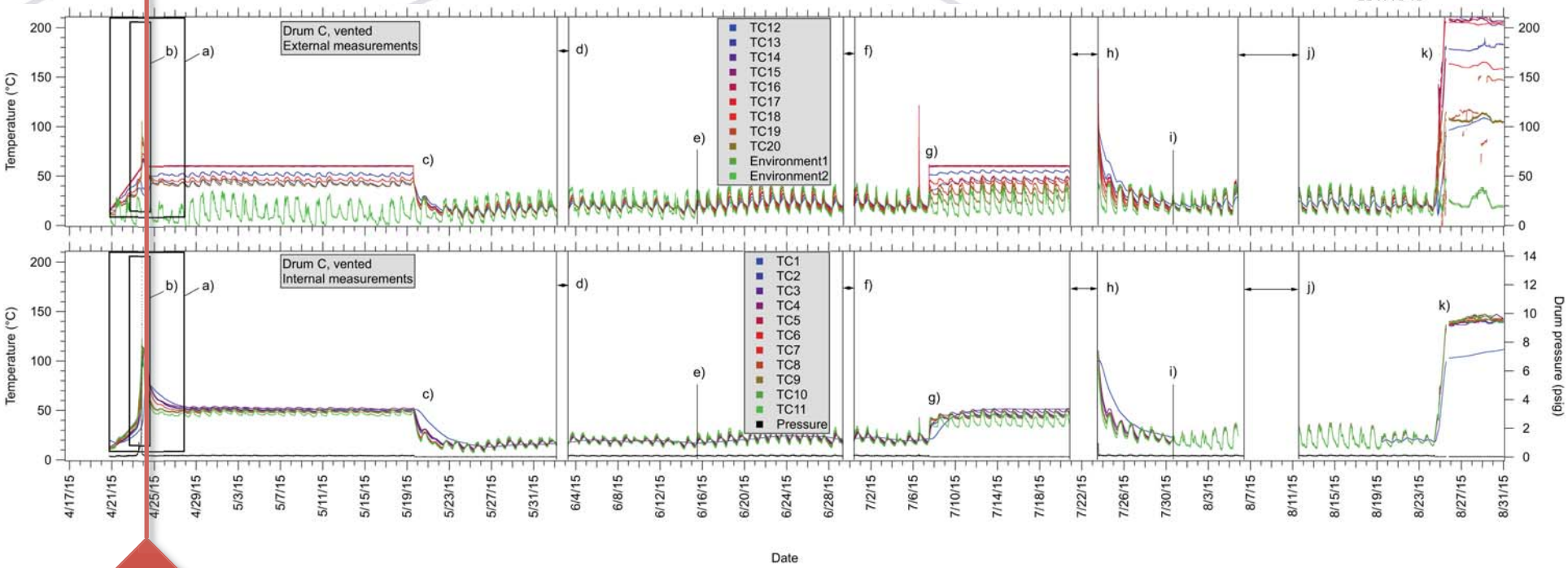
LA-UR-16-21760

Drum C (60°C, vented): Full dataset

Headspace gas sampling cap was fit over the Nucfil filter.



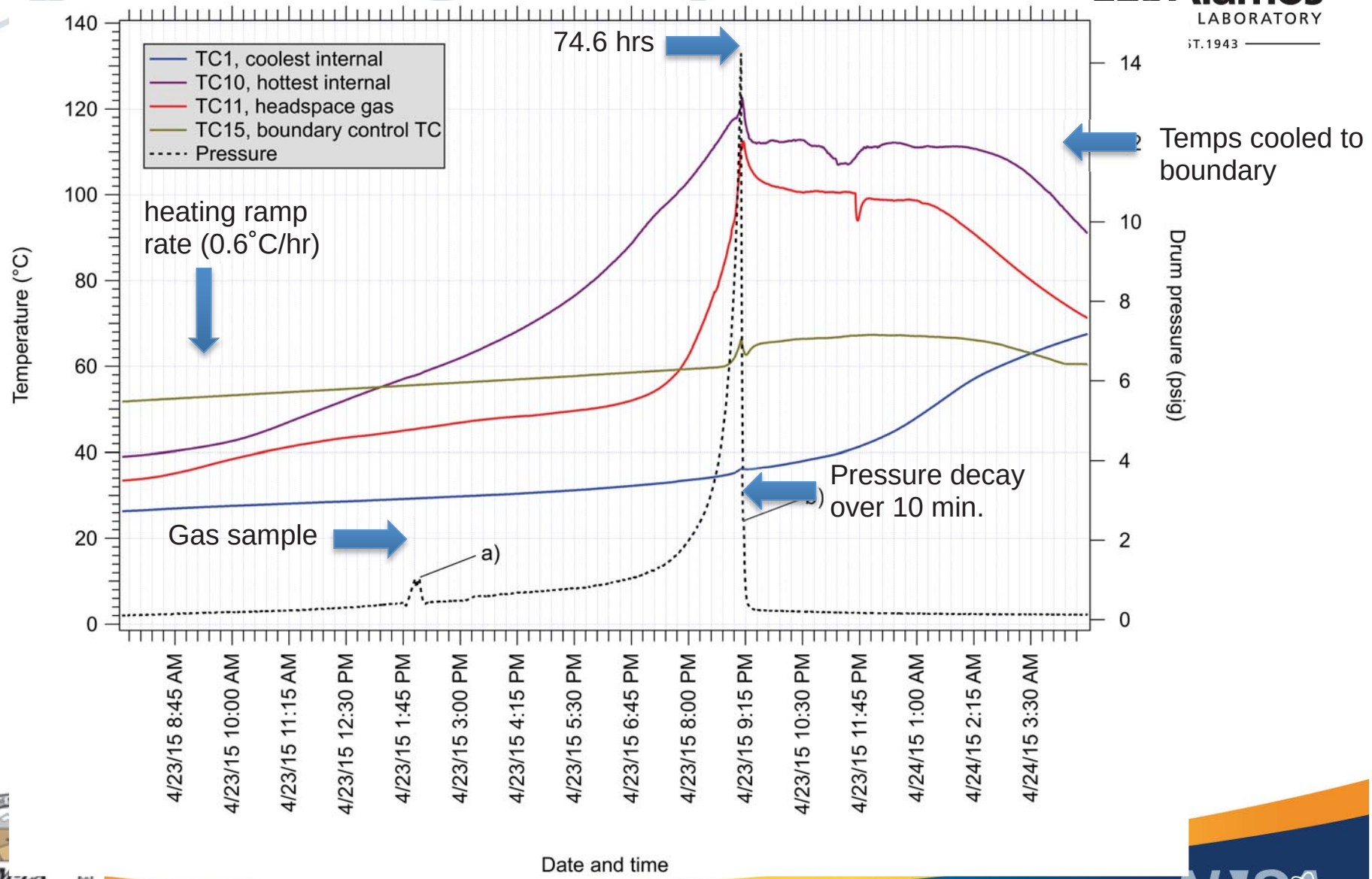
Drum C (60°C, vented): Full dataset



Runaway-quench event

LA-UR-16-21760

Drum C (60˚C, vented) runaway-quench event: detail



LA-UR-16-21760

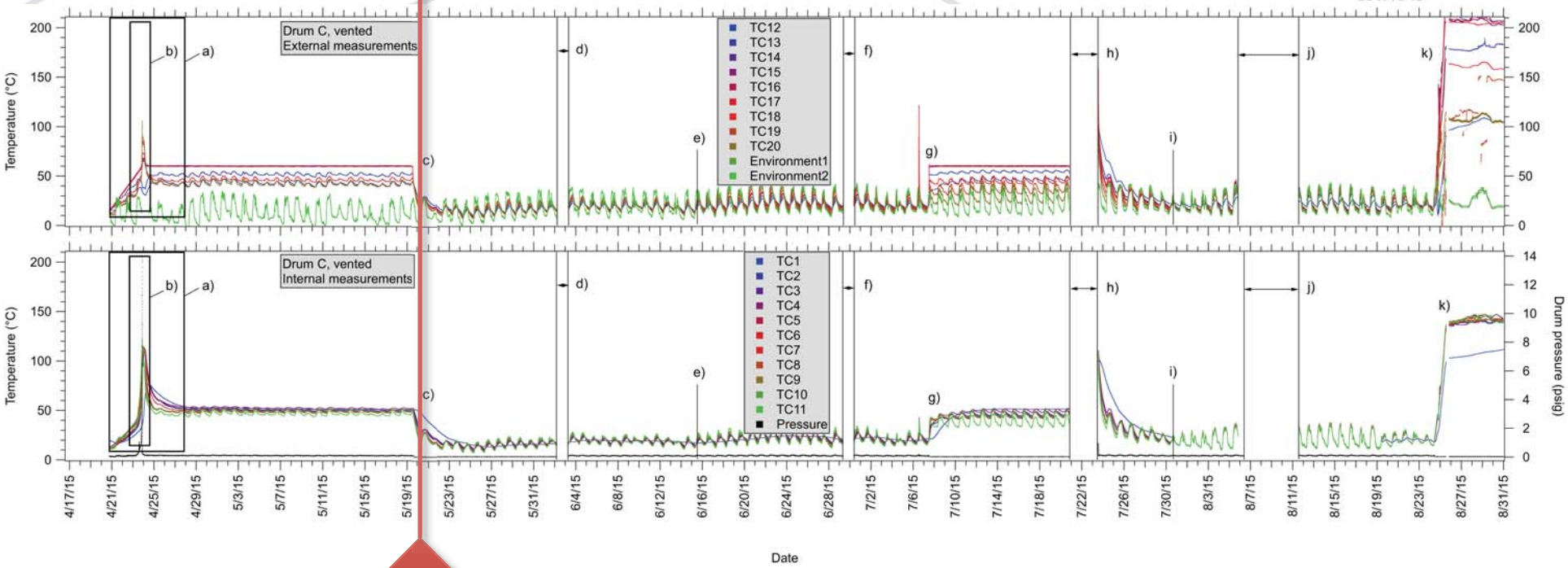
Drum C (60°C, vented): headspace gas analysis

- Flow rate: >2 L/min
- The nitrogen observed is attributed to nitrogen from ambient air. Other gases observed were likely displacing the nitrogen as they were generated within the drum.
- Significant quantities of NO, N_2O and CO_2 were measured.
- Oxygen was not detected in the sample above the reporting limit of 30 mtorr.
- NO_2 cannot be measured directly with the GC/TCD, though pressure balance might indicate very little concentration.

	Gas Partial Pressure (Torr)	% of Drum Headspace	Product Gas Fraction Excluding N_2 (%)
Helium (He)	N.D.		
Hydrogen (H_2)	N.D.		
Oxygen (O_2)	N.D.		
Nitrogen (N_2)	72	50.7	
Nitric oxide (NO)	24.7	17.3	35.2
Carbon monoxide (CO)	3.1	2.1	24.4
Methane (CH_4)	N.D.		
Carbon dioxide (CO_2)	30.2	21.3	43.1
Nitrous oxide (N_2O)	12.2	8.6	17.4
Sum of partial pressures	142.2		
Paroscientific gauge pressure	143.8		
% difference	1.1		

*N.D. < 0.03 Torr

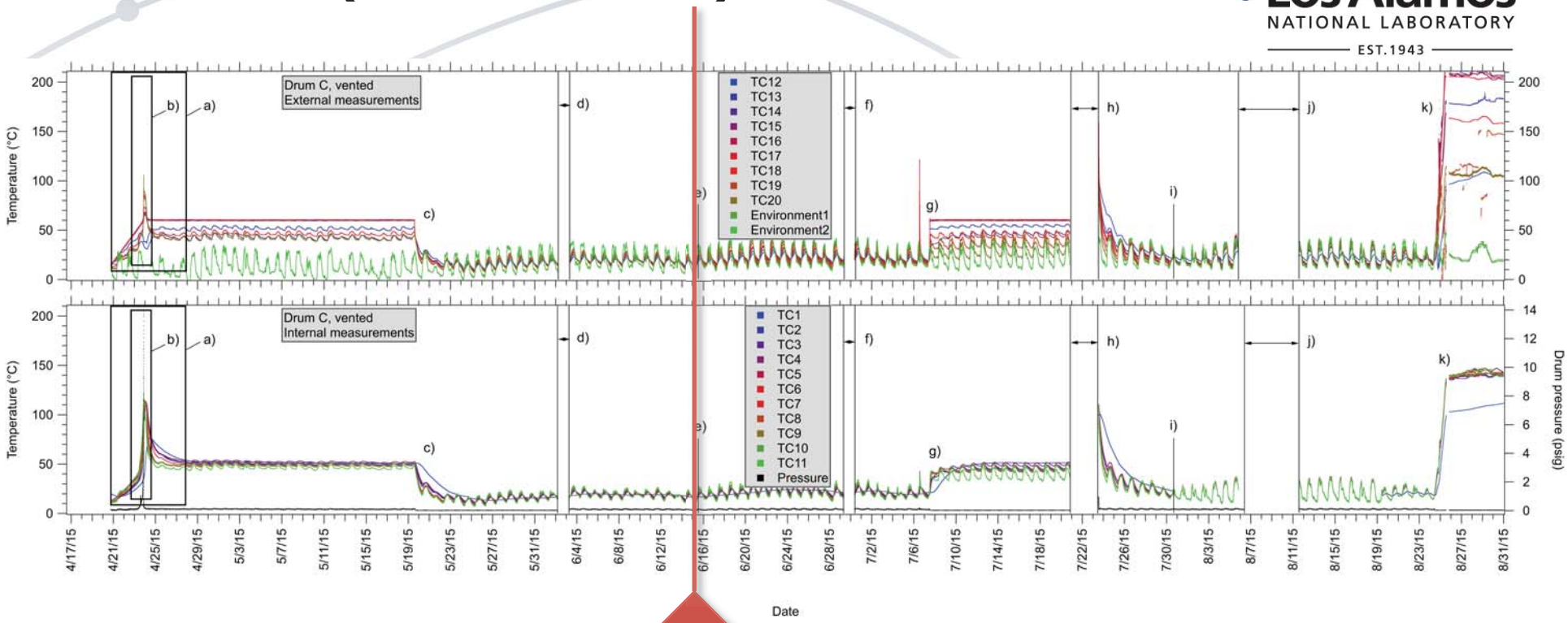
Drum C (60°C, vented): Full dataset



Turned off heater to safely examine gas sampling cap

LA-UR-16-21760

Drum C (60°C, vented): Full dataset



Entered transportainer and examined drum to understand cause of pressure release

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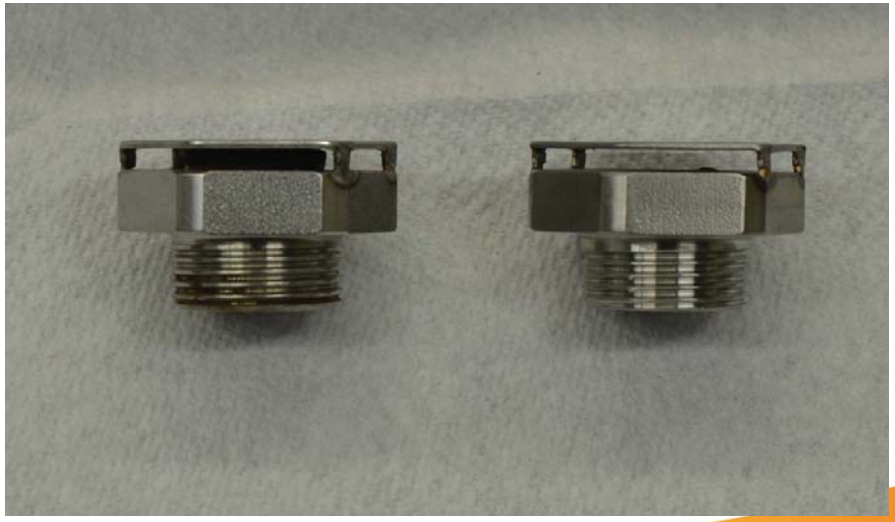
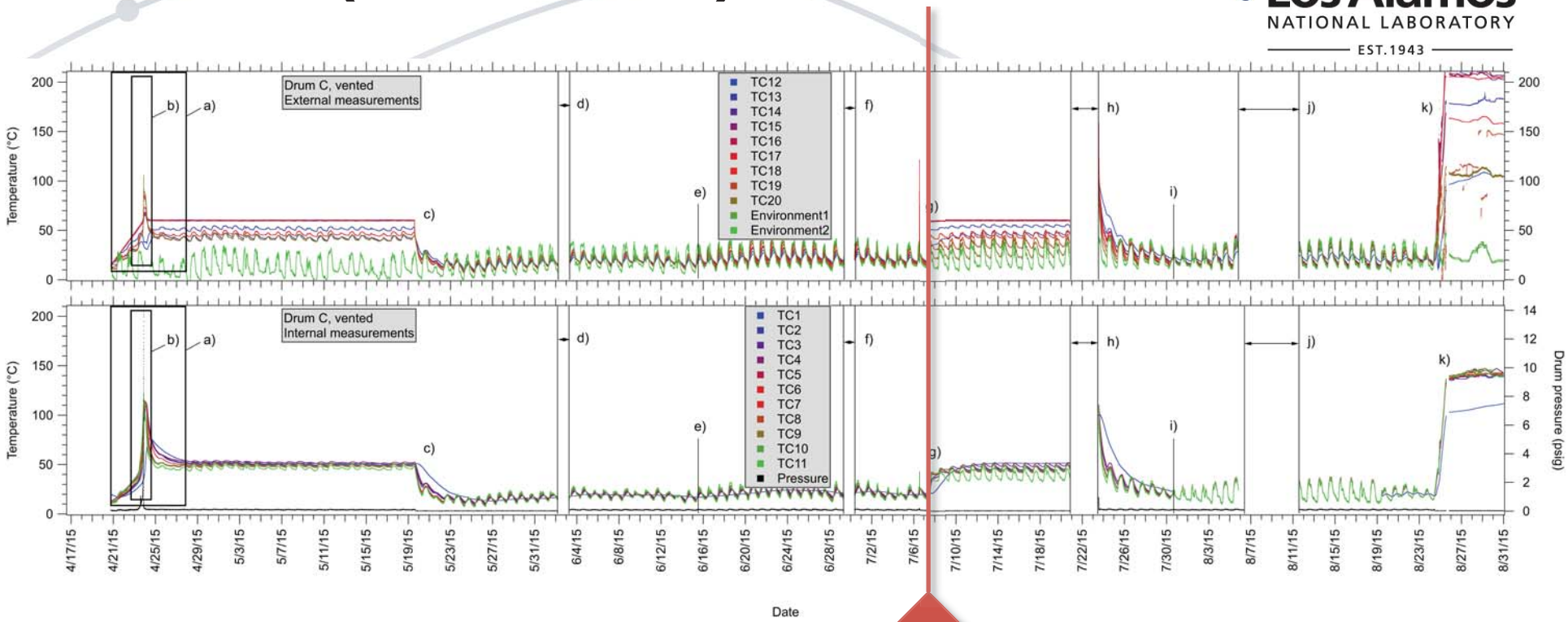
Drum C: Pressure release

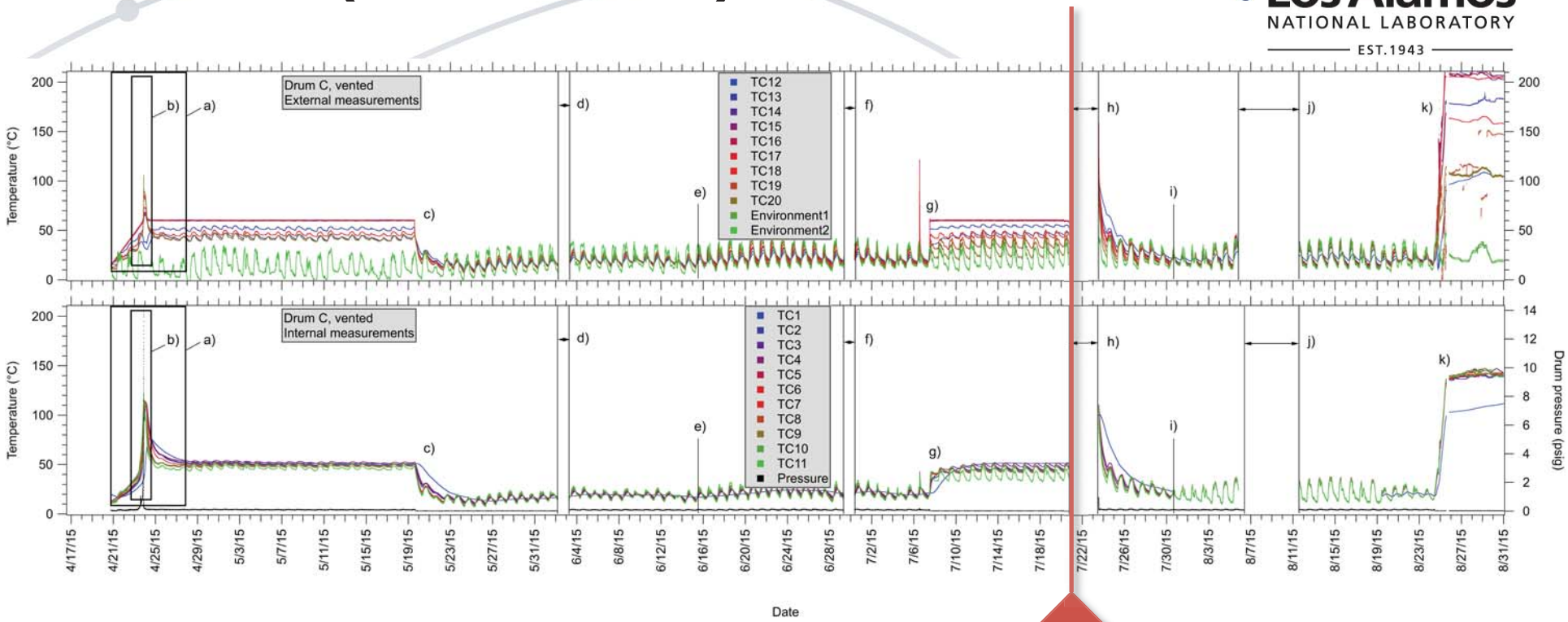


Drum C (60˚C, vented): Full dataset



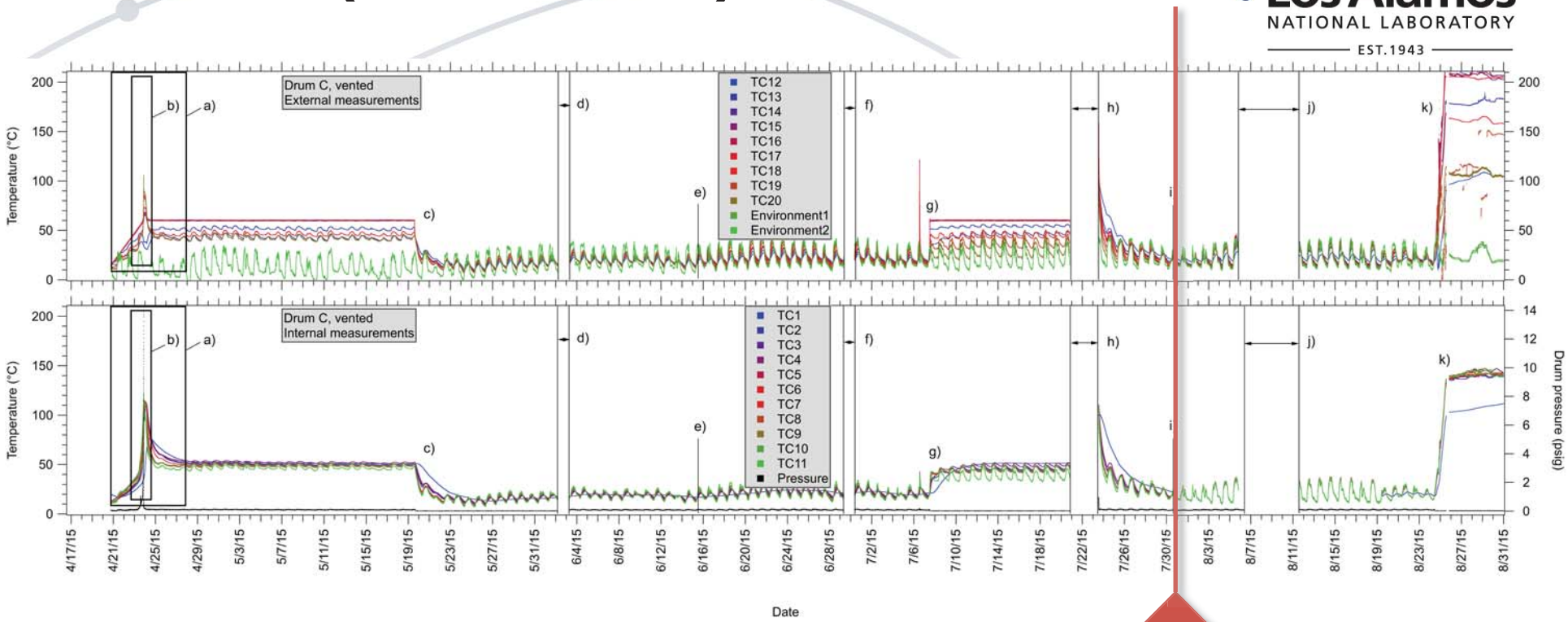
Reheated to 60˚C after replacing Nucfil filter, but not the gas sampling cap. No signs of reactivity.

Drum C (60˚C, vented): Full dataset



Loss of facility power. Controller reset to 160˚C.

Drum C (60°C, vented): Full dataset

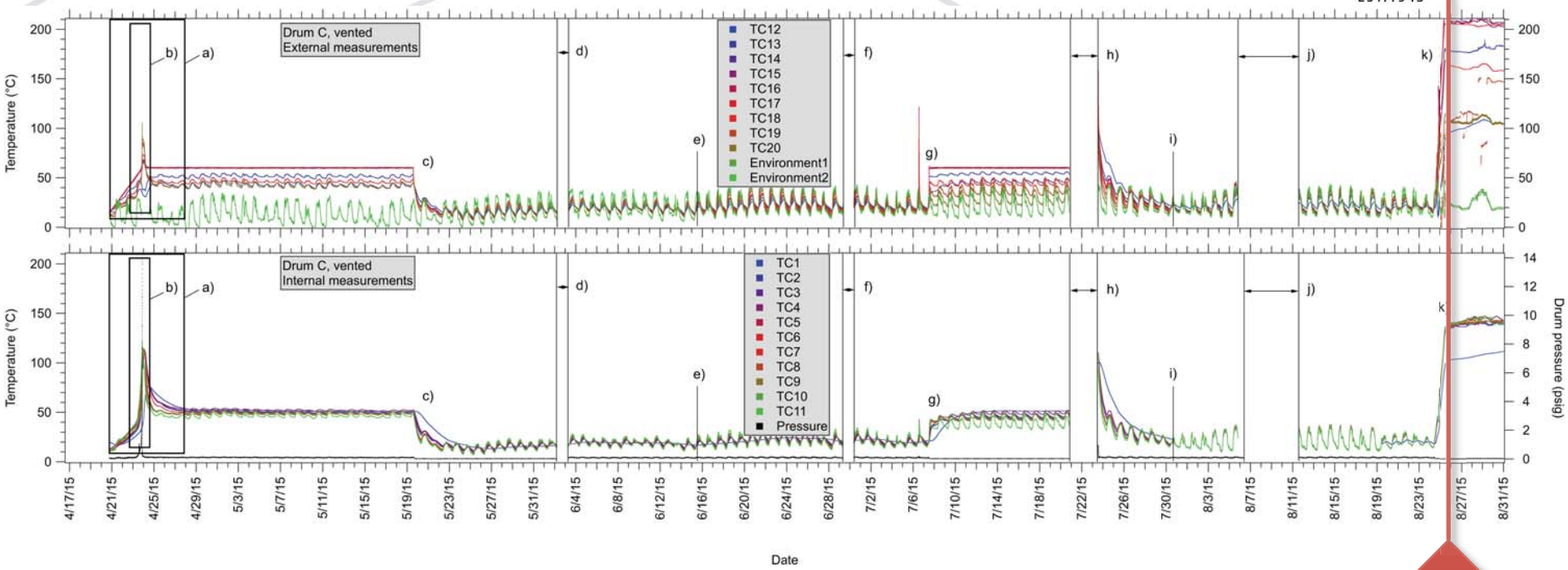


Drum was opened for post-mortem examination

Drum C: Post-mortem

- Lid was corroded
  - Seal had melted
- Bag yellowed
- Condensation present
- Contents were homogeneous, damp and sooty
  - Lighter colored powder is exfoliated cardboard from liner.

Drum C (60˚C, vented): Full dataset



Drum was heated to 200˚C to render the contents safe. No signs of reactivity during this phase of heating.

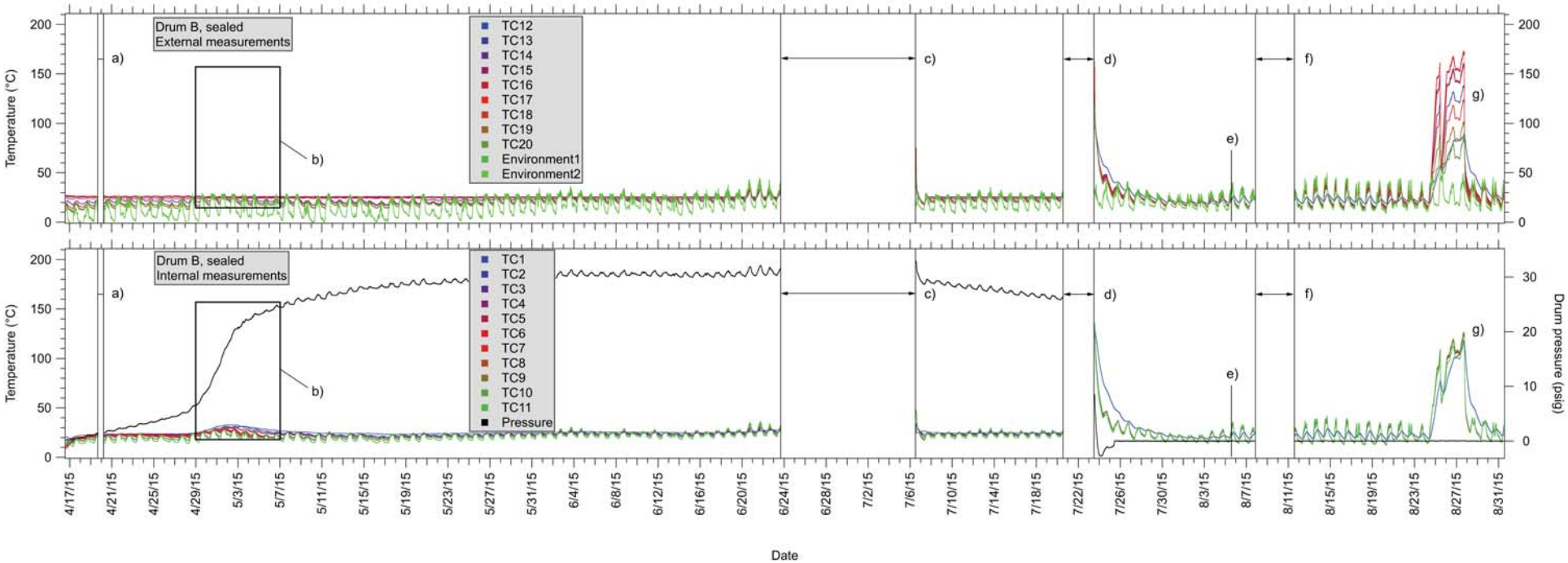
Drum C: Summary

- Pressure appears to be of paramount importance to the thermal runaway
  - Importance of gas-phase reactants attacking the solids
  - When pressure was relieved, runaway was quenched at 115˚C
  - Flow restriction of the vent may be necessary for runaway to occur
- Pressure rise in vented drum due to some combination of:
  - Backpressure from gas sampling fixture
  - Restricted flow through carbon filter
- Hottest location in top layer, high headspace gas temp.
  - Also evidence for the importance of gas-phase reactants

Results

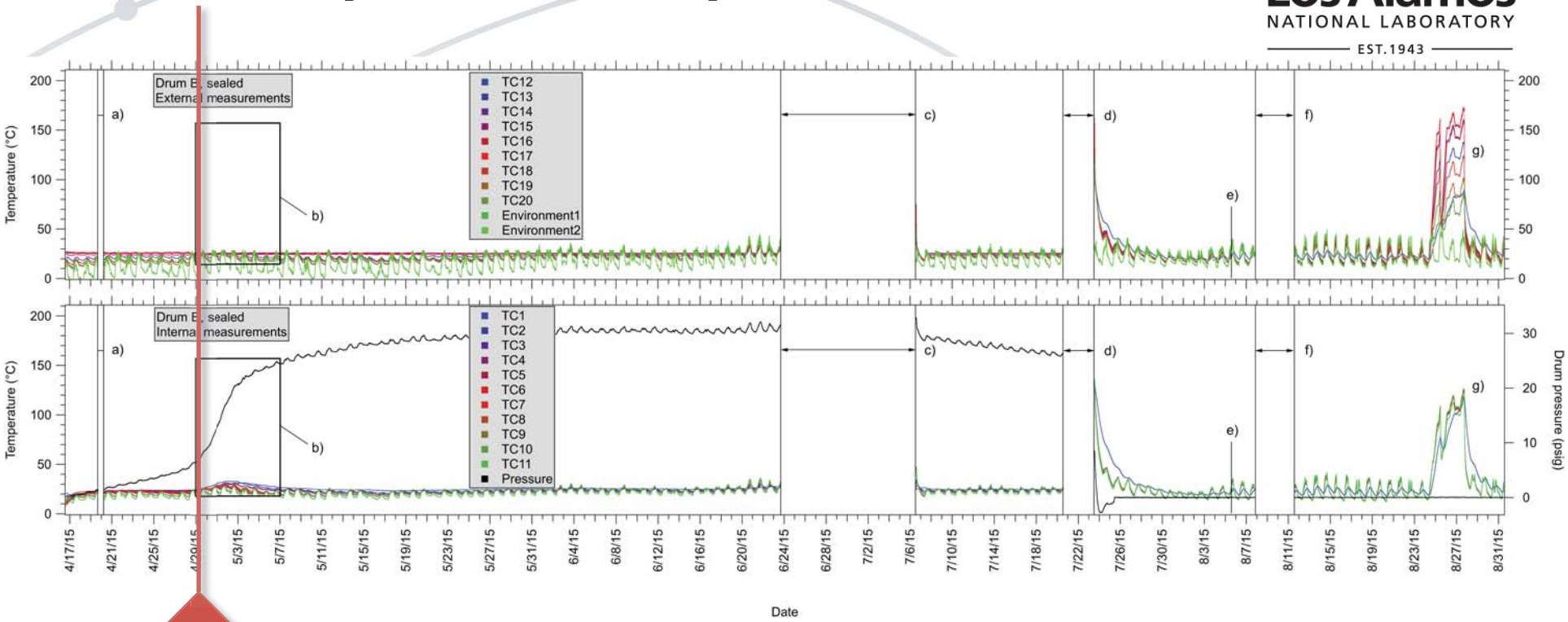
Drum B

Drum B (25°C, sealed): Full dataset



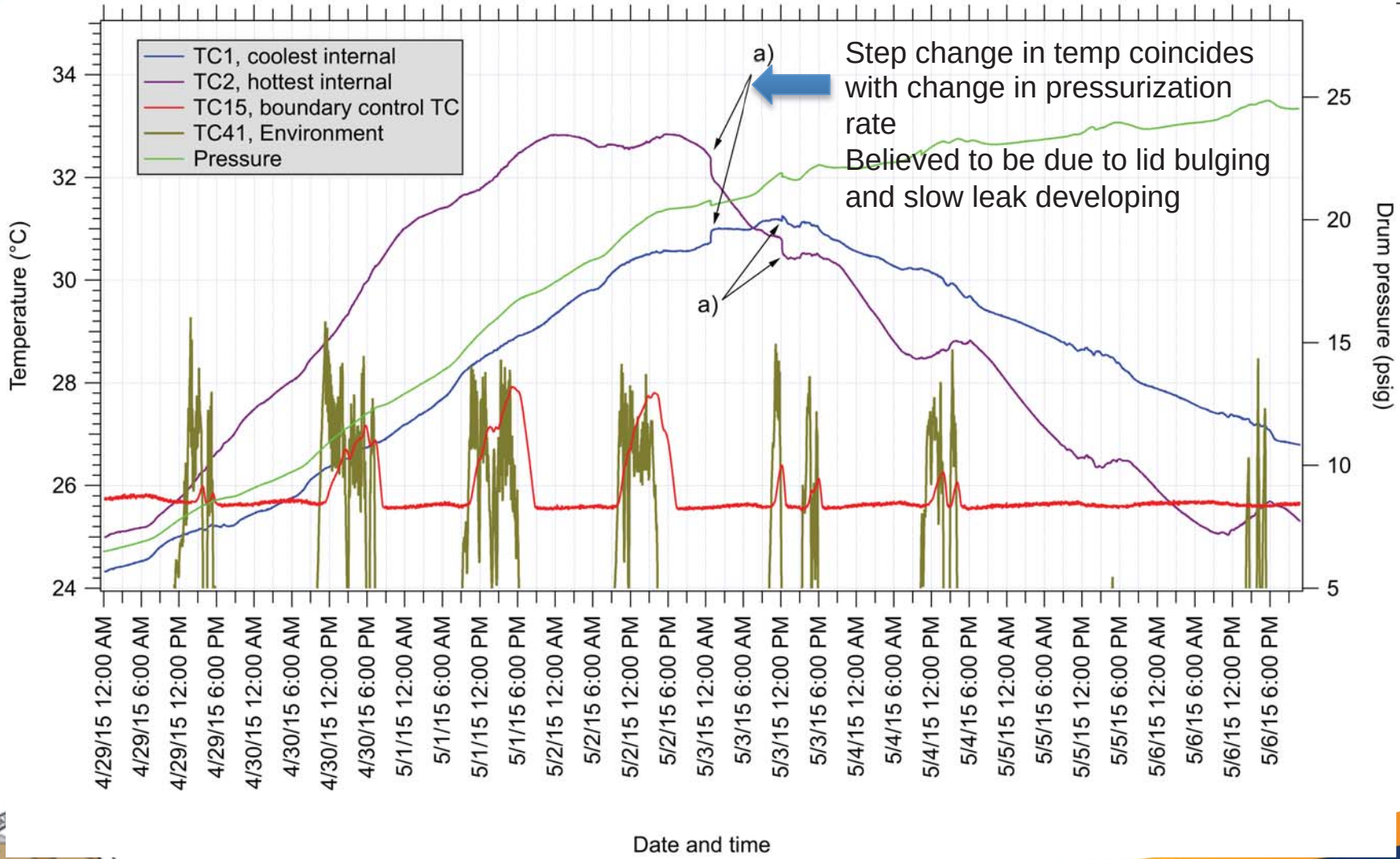
This drum represents a drum stored under normal conditions in the WIPP, but explores the possibility of the vent having become blocked.

Drum B (25°C, sealed): Full dataset

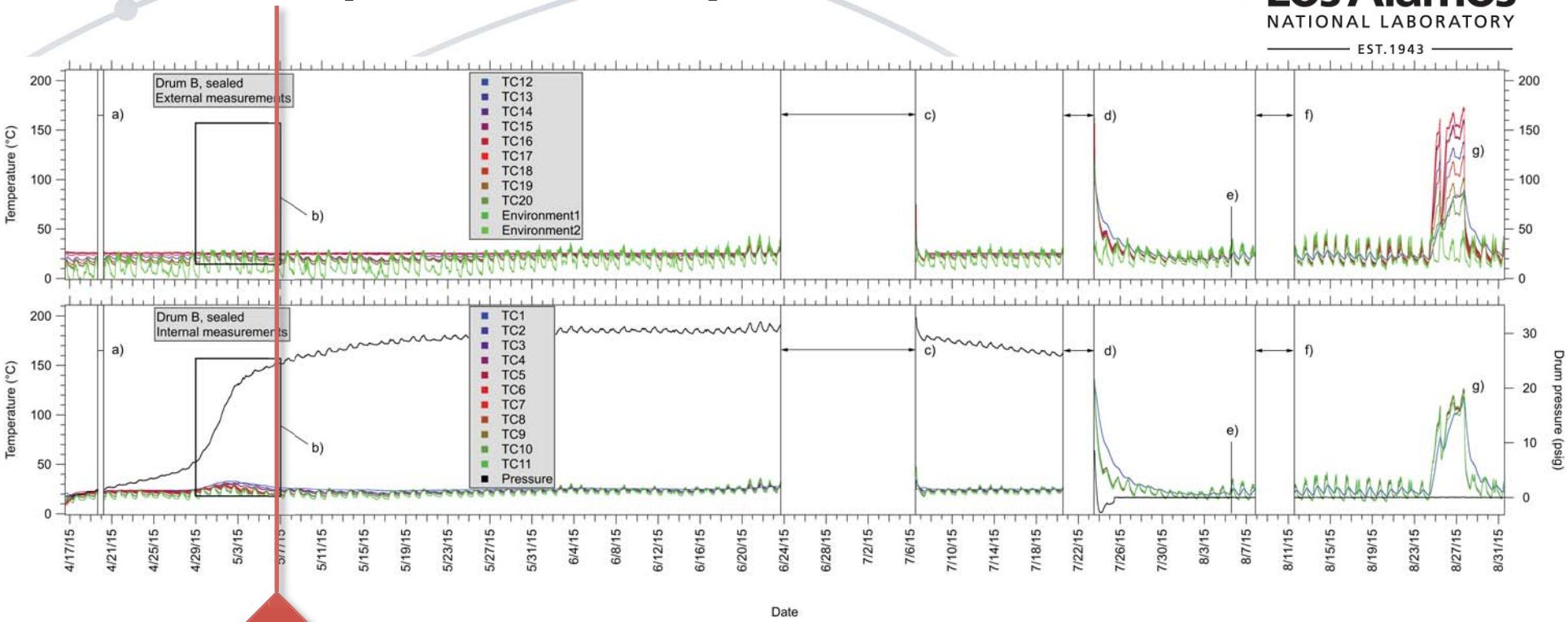


Around day 12, saw the onset of self-heating and increased rate of pressurization, followed by quench. Interestingly, 12 days is the approximate duration that Drum 68660 was emplaced in the WIPP.

Drum B (25°C, sealed): Self-heating detail

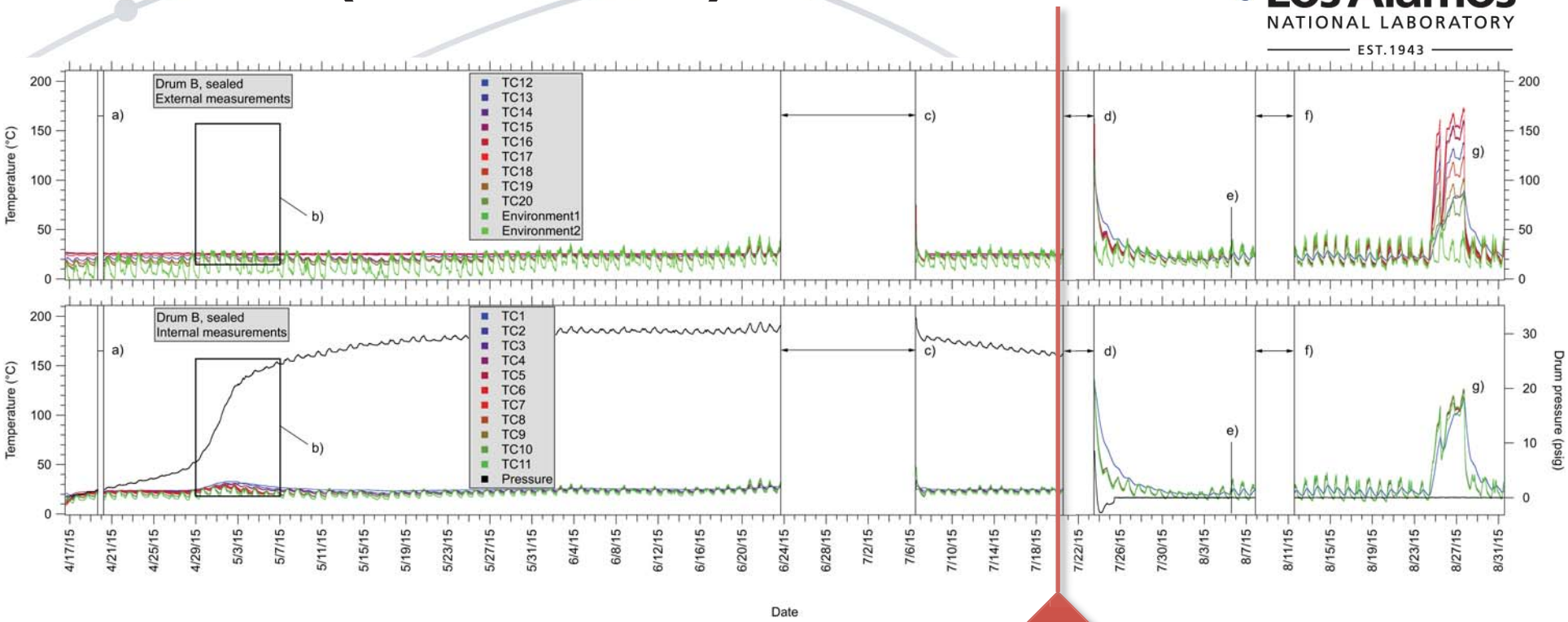


Drum B (25°C, sealed): Full dataset



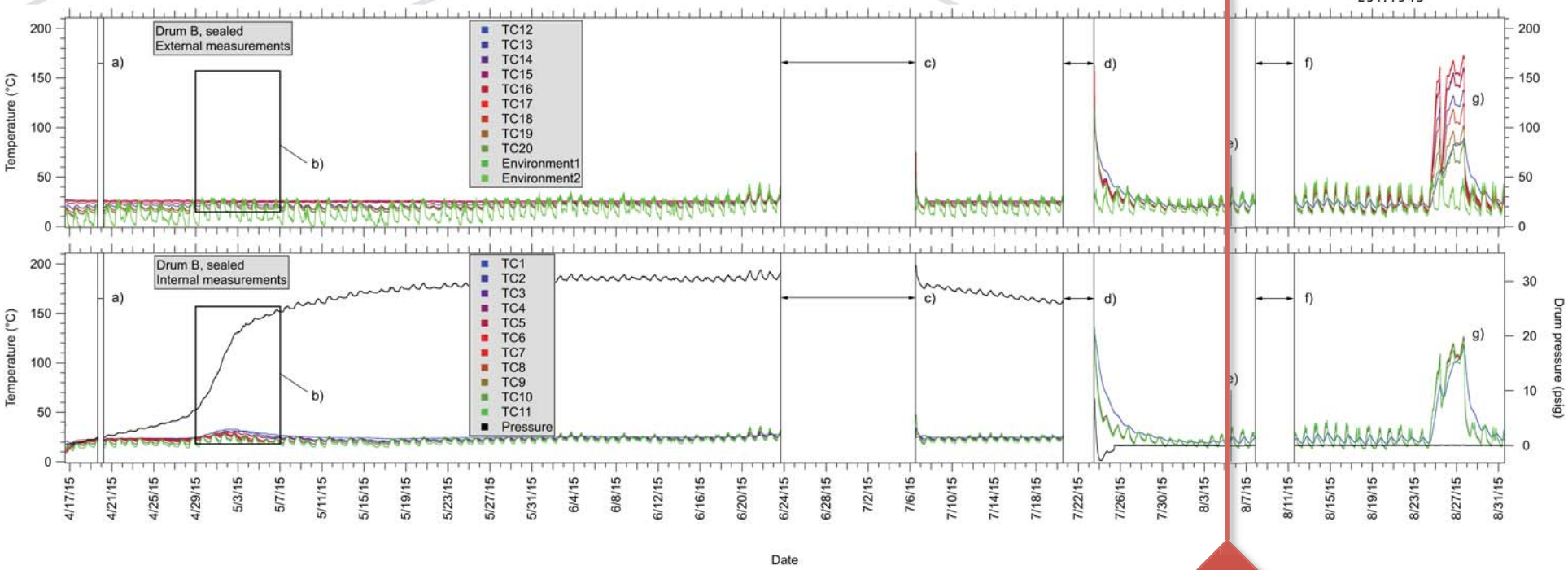
After quench of self-heating, pressure held at ~30 psi with no new signs of heating.

Drum B (25˚C, sealed): Full dataset



Condition was stable for ~75 days, then the facility power was lost and the controller reset to 160˚C.

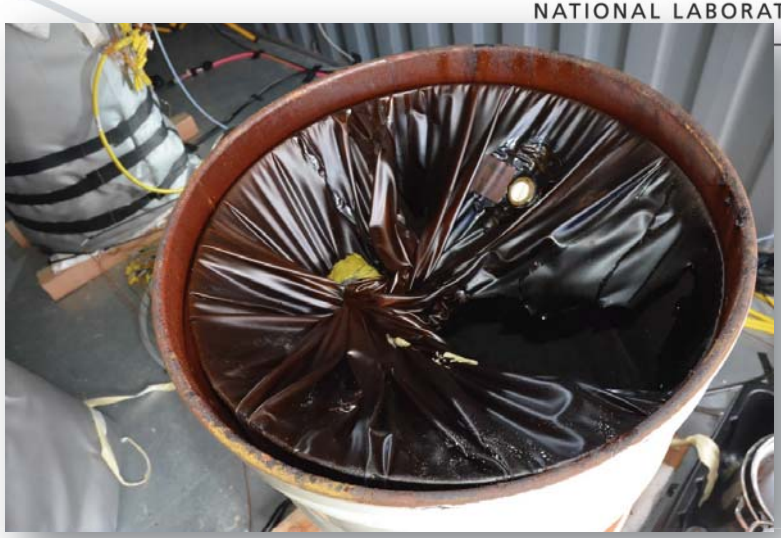
Drum B (25°C, sealed): Full dataset



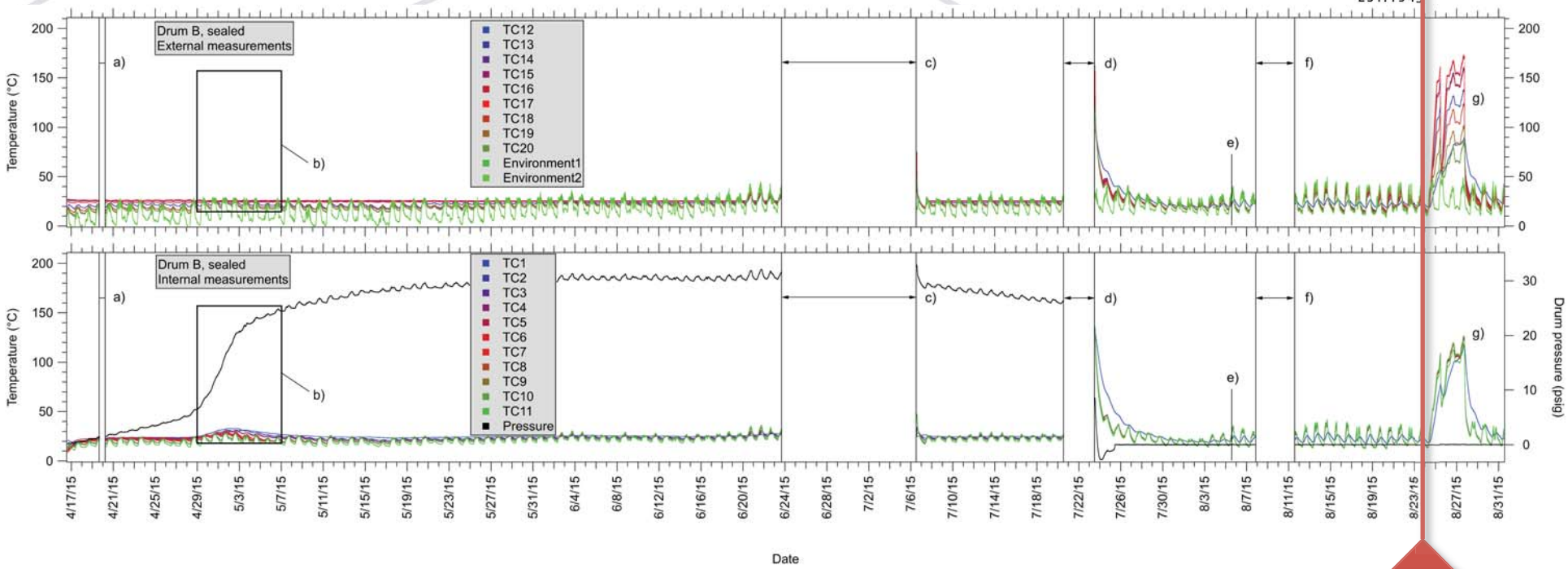
Drum was opened for post-mortem examination.

Drum B (25°C, sealed): Post-mortem

- Lid was bulged
  - Lid seal failed
- Bag reddened and thermally damaged
- Contents were homogenous, dry and sooty
  - Material had slumped



Drum B (25˚C, sealed): Full dataset



Drum was heated to 170˚C to render the contents safe. No signs of reactivity during this phase of heating.

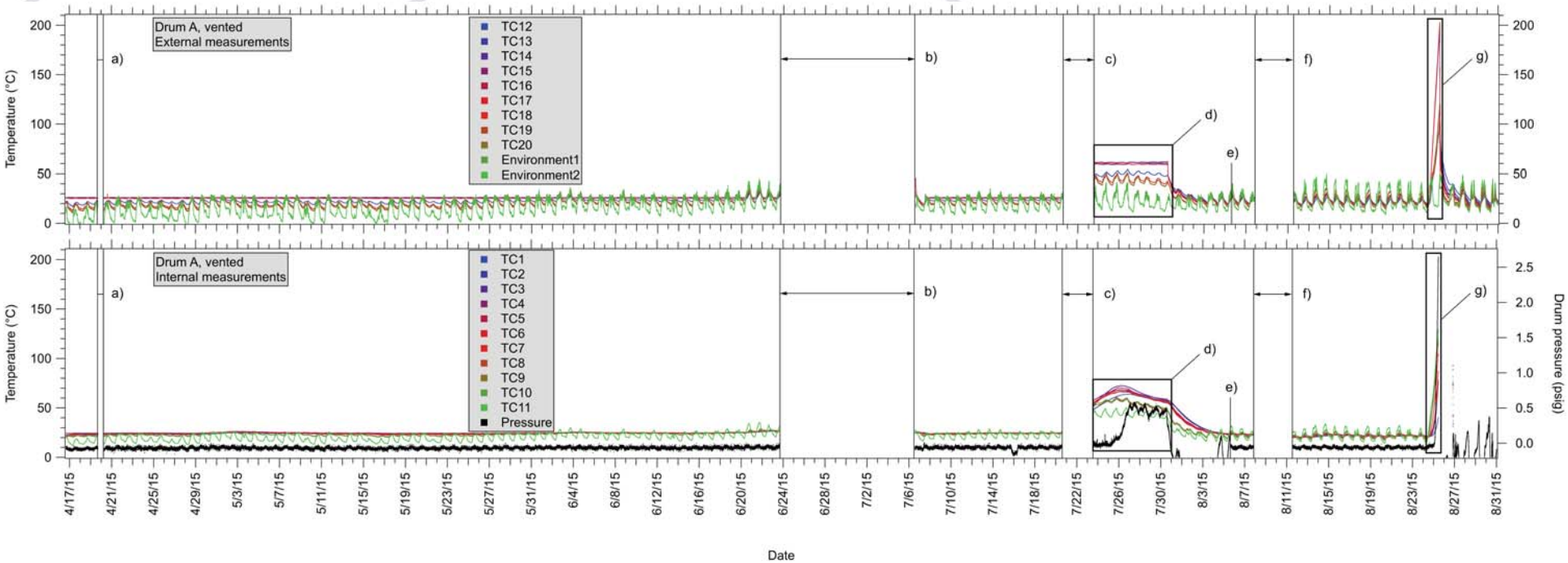
Drum B: Summary

- Reactivity and self-heating occurs at 25˚C.
  - Self heating did not activate the next higher-temperature reaction(s) and quenched.
    - Low-rate, low-temperature reactivity depleted reactants.
- Quench of self heating coincident with venting of gas and pressure stabilization
  - Evidence for the importance of gaseous reactants.
- Upon heating to 160˚C, there was evidence of combustion
  - Despite depletion of low-temperature reactants, higher temperature reactivity, or pockets of unreacted material, persisted.

Results

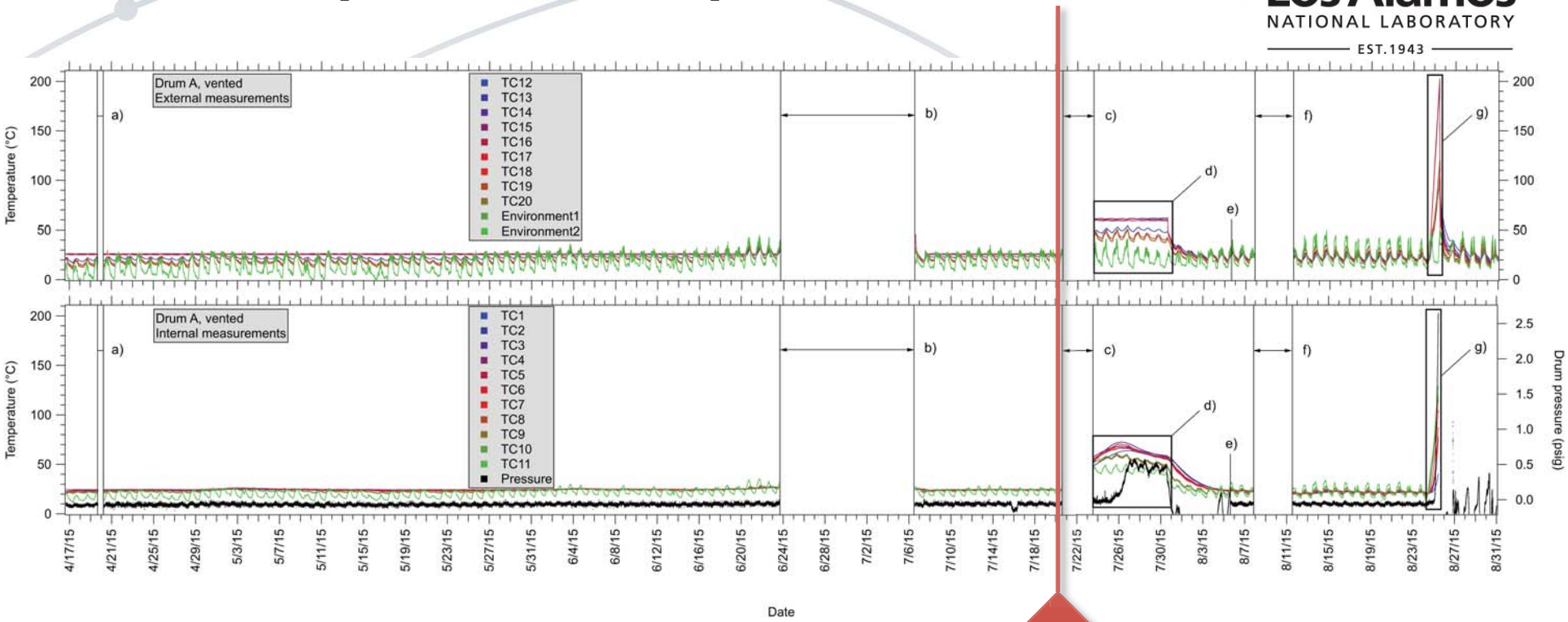
Drum A

Drum A (25°C, vented): Full dataset



This drum represents the normal storage conditions for an RNS drum in the WIPP

Drum A (25˚C, vented): Full dataset



After ~94 days without signs of self-heating, facility was lost and controller reset to 60˚C.

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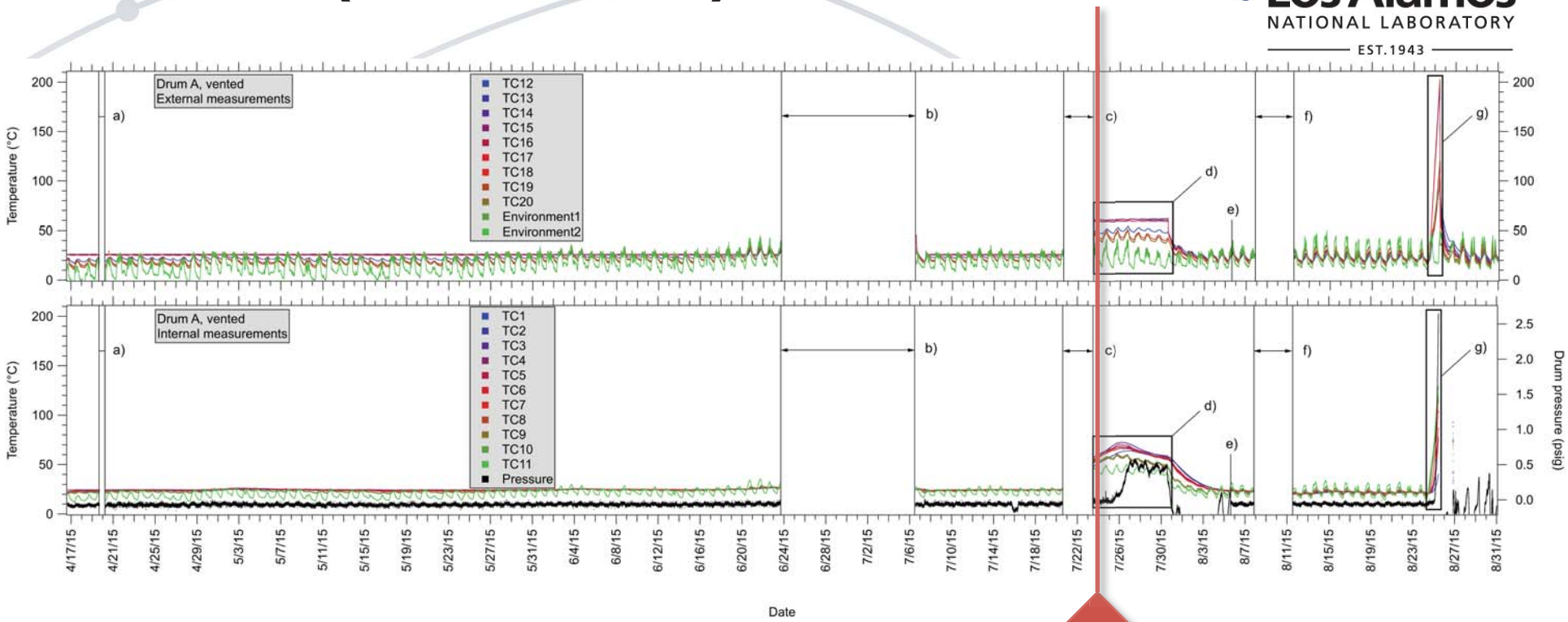
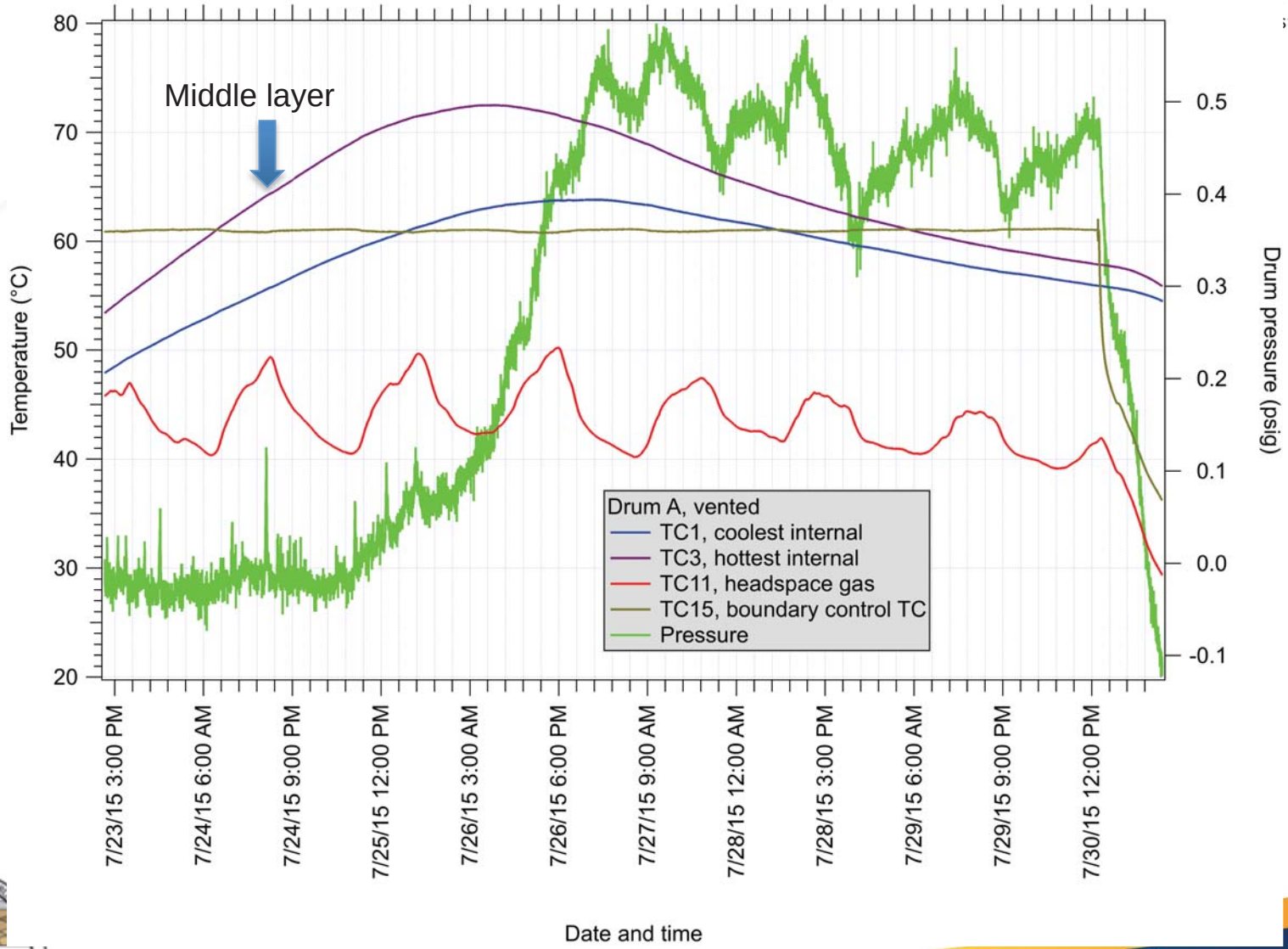
Drum A (25˚C, vented): Full dataset

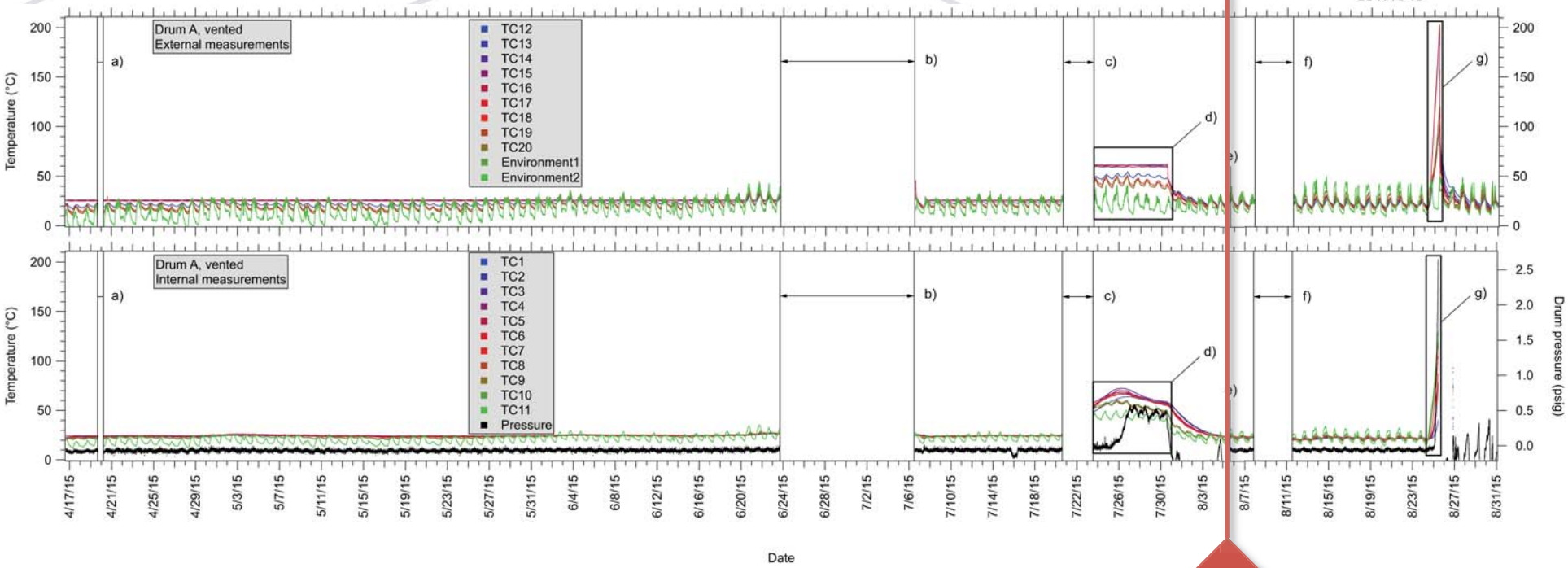


After 3 days at 60˚C, the contents began to self-heat, but quenched.

Drum A (25°C, vented) self heating-quench event: detail



Drum A (25˚C, vented): Full dataset

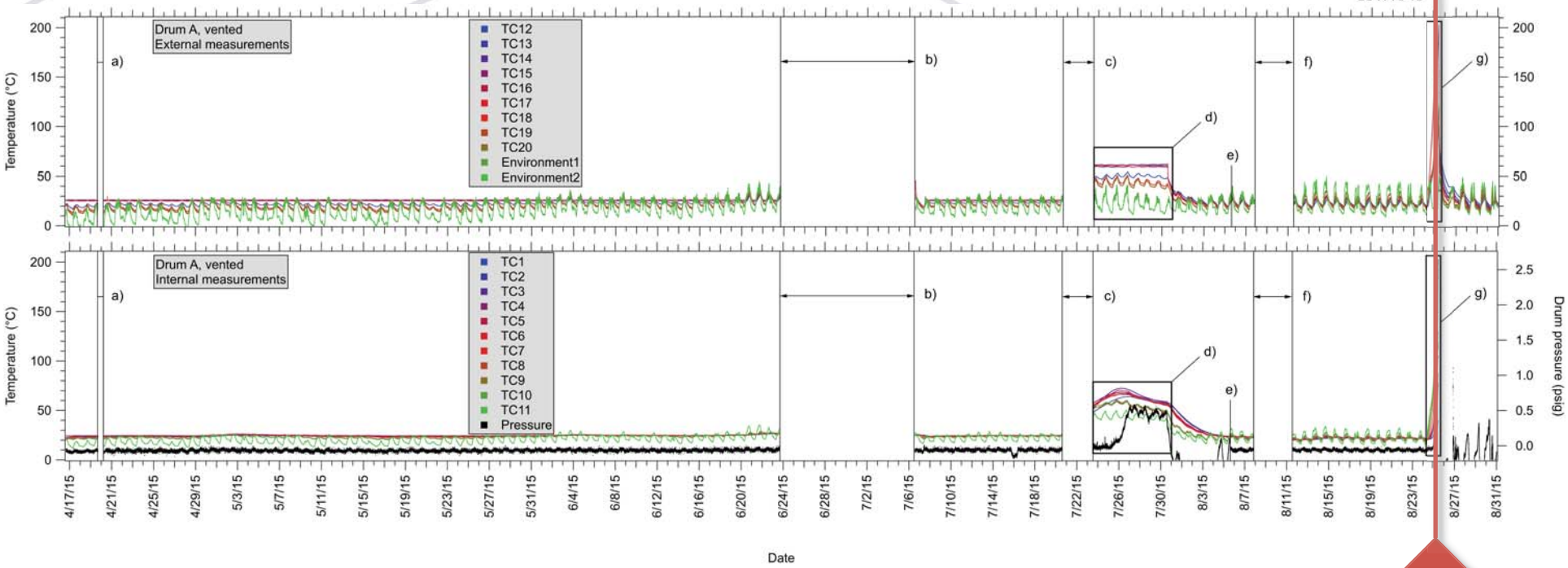


Heater was turned off and drum was opened for examination of contents.

Drum A (25˚C, vented): Post-mortem

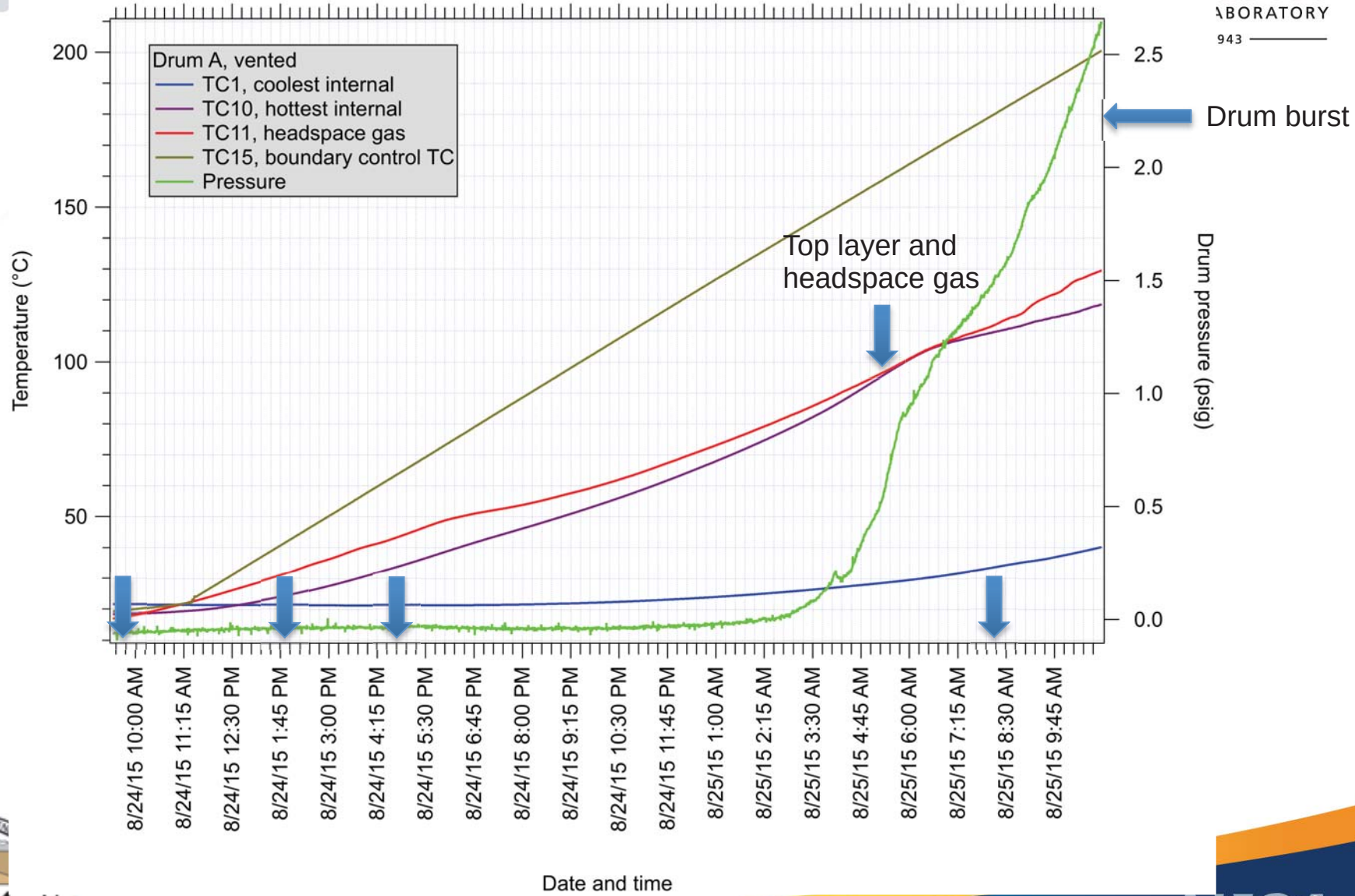
- Lid was intact
  - Corrosion present
- Bag slightly yellowed
- Contents were heterogeneous and damp
  - Material had reddened slightly, but otherwise looked like its original condition
- Likely the reactive potential still existed

Drum A (25˚C, vented): Full dataset



Drum was heated to 200˚C to render the contents safe. Reaction was observed.

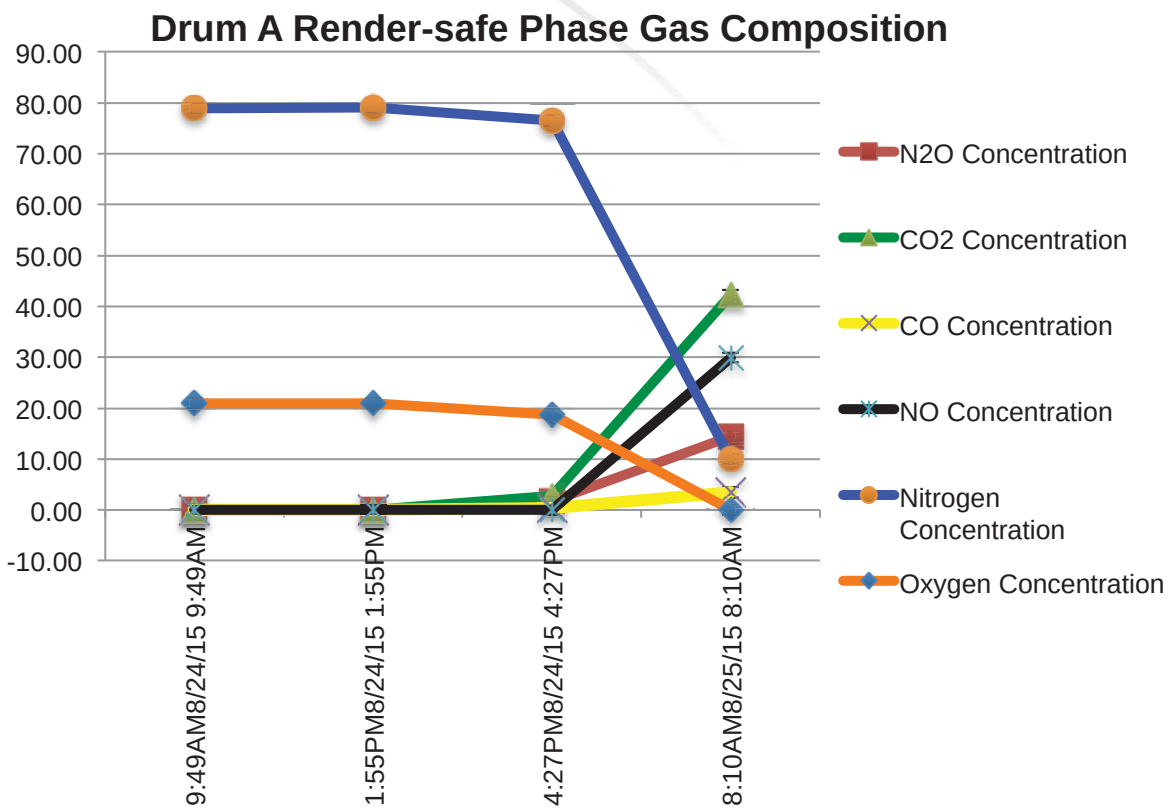
Drum A (25°C, vented) self heating-quench event: detail



LA-UR-16-21760

Drum A (25°C, vented): headspace gas analysis

- The nitrogen observed is attributed to nitrogen from ambient air. Other gases observed were likely displacing the nitrogen as they were generated within the drum.
- Significant quantities of NO, N_2O and CO_2 were measured.
- Oxygen was gradually depleted until it was not detected in the sample above the reporting limit of 30 mtorr.
- NO_2 cannot be measured directly with the GC/TCD, though pressure balance might indicate very little concentration.



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Drum A: Summary

- In first 94 days, lack of measurable temperature rise shows that a normally configured drum should be able to adequately dissipate heat and products gases so that thermal runaway is not possible.
- After 94 days, upon heating to 60˚C, self-heating began showing that reactive potential remained. However, even these reactions quenched.
- Upon heating to 200˚C, there was evidence of combustion after internal temperatures exceeded 120˚C.
  - Sudden rupture of the drum and dispersal of glowing embers.

Discussion: Comparisons

- Drum A (vented) vs. Drum B (sealed), both at 25˚C
  - Drum B showed pressure rise from the start and self-heating after 12 days. Drum A showed neither.
    - Suggests reaction is occurring at 25˚C, but slowly.
  - Key points:
    - If vented, the heats of reaction and product gases are dissipated to the environment efficiently and reactive NO_x gas concentrations stay low. Low-temperature NO_x-producing reactions eventually deplete reactants.
    - If sealed, reactant gas concentration increases as do kinetics. This low-temperature chemistry does not, however, liberate enough heat energy to self-heat the mass up to the next "rung" on the notional "ladder" of ever-higher-temperature reactions.
    - These drums were insulated, whereas actual drums are not.

Discussion: Comparisons

- Drum C (vented) vs. Drum D (sealed), both at 60˚C
  - Drum D exhibited thermal runaway and pressure burst. Drum C did too, but only so long as gas flow was restricted or blocked. Once pressure was relieved, thermal runaway was halted (at 115˚C).
    - Remarkable turnaround late in the runaway.
  - Key points:
    - Two conditions—a blocked vent *and* elevated temperature—were required to cause thermal runaway and drum breach.
    - Neither blockage, nor 60˚C boundary temperature, alone caused breach.

Discussion

- If Drums A & B did *not* undergo thermal-runaway-to-ignition, why did Drum 68660?
  - Surrogate-filled drums had more water
    - A critical fraction of heat energy was partitioned into water's heat capacity and latent heat of vaporization, hence was unavailable to raise the temperature of the bulk sufficiently to access the next rung of the "ladder".
  - Our surrogate mixture had higher activation energy than the contents of Drum 68660
    - Recent formulations (e.g. SFWB11) shows lower temperature activation and higher reaction rates.
  - The drum contents—both physical and chemical makeup — are widely variable and Drum 68660 had a rare combination that put it on the tail of the distribution of potential compositions.
    - With this possibility, and the fact that no other drums have behaved similarly, statistical analysis can be attempted.

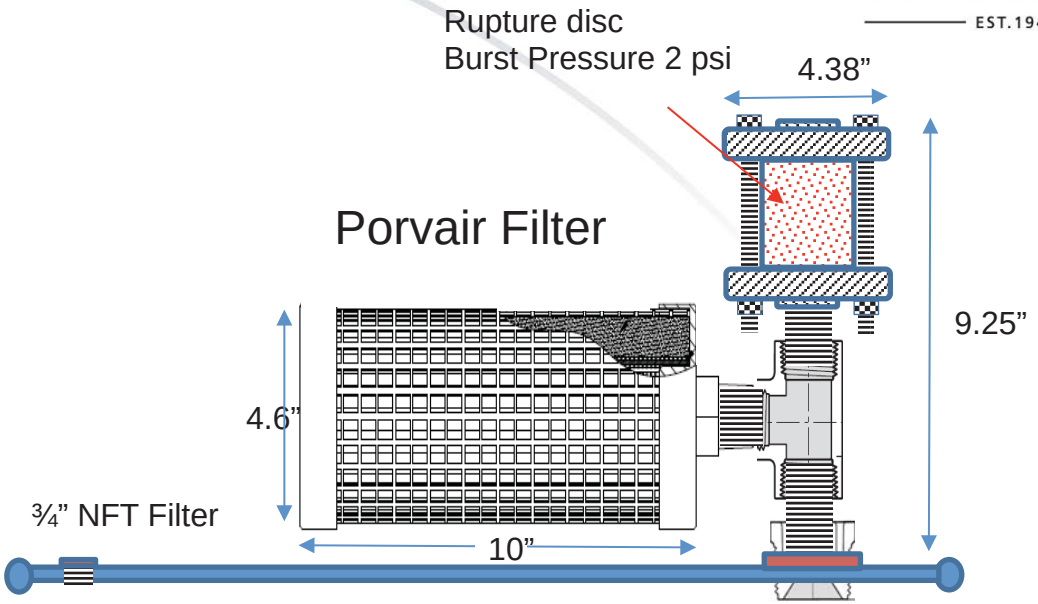
Conclusions from the full scale tests

- These tests demonstrated thermal runaway and drum rupture with a plausible surrogate nitrate salt/Swheat mixture
  - Supports the hypothesized “ladder” of reactions
  - Evidence supports the hypothesis that NO_x product gases from hydrolysis of metal nitrate salts are responsible for exothermic oxidation of the organic pet litter.
- Pressurization is required for runaway
  - Very sensitive to gas concentration (correlated with pressure).
- Reactant concentrations for the low-temperature chemistry can be diminished with sufficient time at ambient temperature.
  - Likelihood for bootstrapping up to the next higher-temperature chemistry goes down.
  - This does not mean that higher-temperature reactions can’t be activated if external heating is applied. In fact, we have shown this can happen.
- Accident prevention strategies include:
  - Elimination of the potential for pressurization.
  - Reduction in storage temperature.

Strategy for enhanced safing is being developed

Estimate Flow of Gas in Large Drum Tests

time	Temp	Pressure	Flow Rate	
hrs	°F	psig	l/m	cfm
24	73	0.7	0.003	0.000
48	84	2.0	0.012	0.000
60	82	3.7	0.020	0.001
62	89	4.8	0.055	0.002
64	96	5.2	0.063	0.002
66	101	6.5	0.104	0.004
68	104	8.7	0.214	0.008
70	106	13.3	0.430	0.015
72	110	21.7	1.16	0.041
72.6	146	30.9	2.75	0.1



Rupture Disc

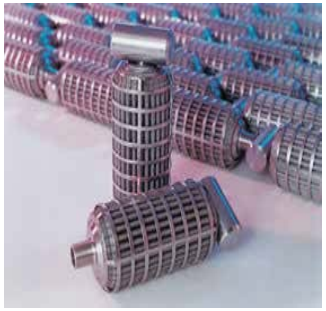
Holder

Disc

1.5 to 10 psi

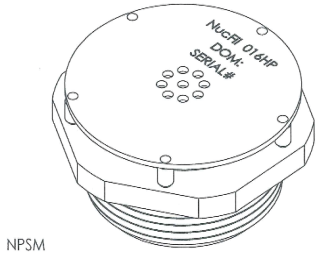
Porvair Filter

91,000 ml/m @ 1 “ WC
3.25 CFM @ 1” WC



NFT Filters

¾” NFT = 200ml/m @ 1 “ WC



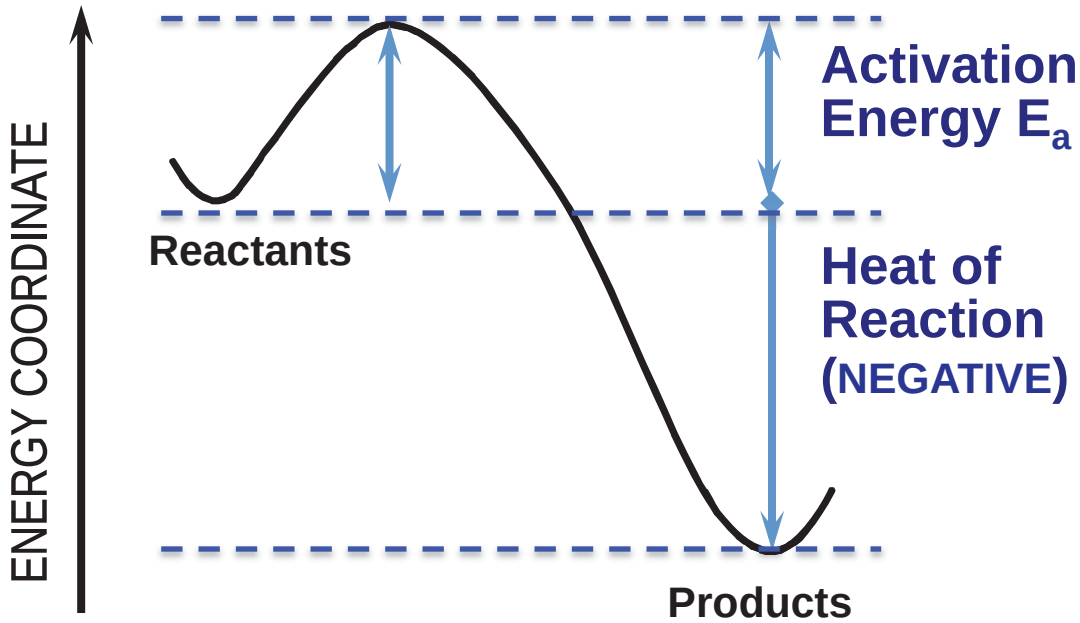
Small Scale Follow-On Work

- Validate thermal sensitivity decreases with time
  - Simply put, the lower the onset temperature, the more reactive the the species and the greater their subsequent depletion at ambient temperatures
- Investigate whether agitation can reset drum contents (and to what level); significant concern about this
  - Plan:
    - Prepare 12 or more salt/swheat mixtures in Nalgene bottles equipped with NFT filters
    - Once a week test with APTAC to evaluate whether we observe increasing/decreasing thermal/ignitability behavior
    - At the end of the test period (12 weeks), we can shake them and retest a subset, to observe the effect of agitation

Temperature Control Strategy: Technical Basis

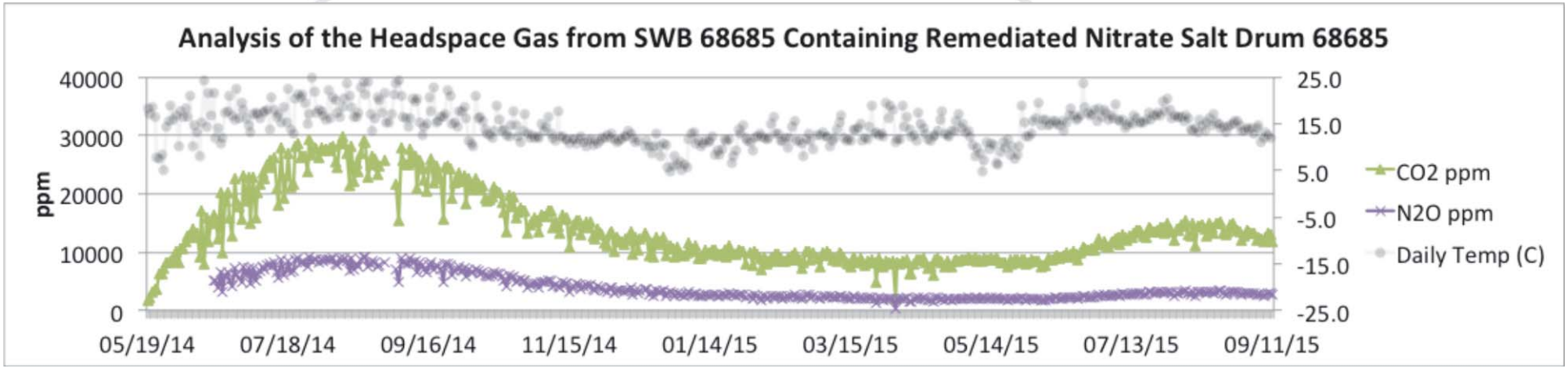
Arrhenius equation – first order kinetics:

$$k(T) = A\, e^{(-Ea/RT)}$$



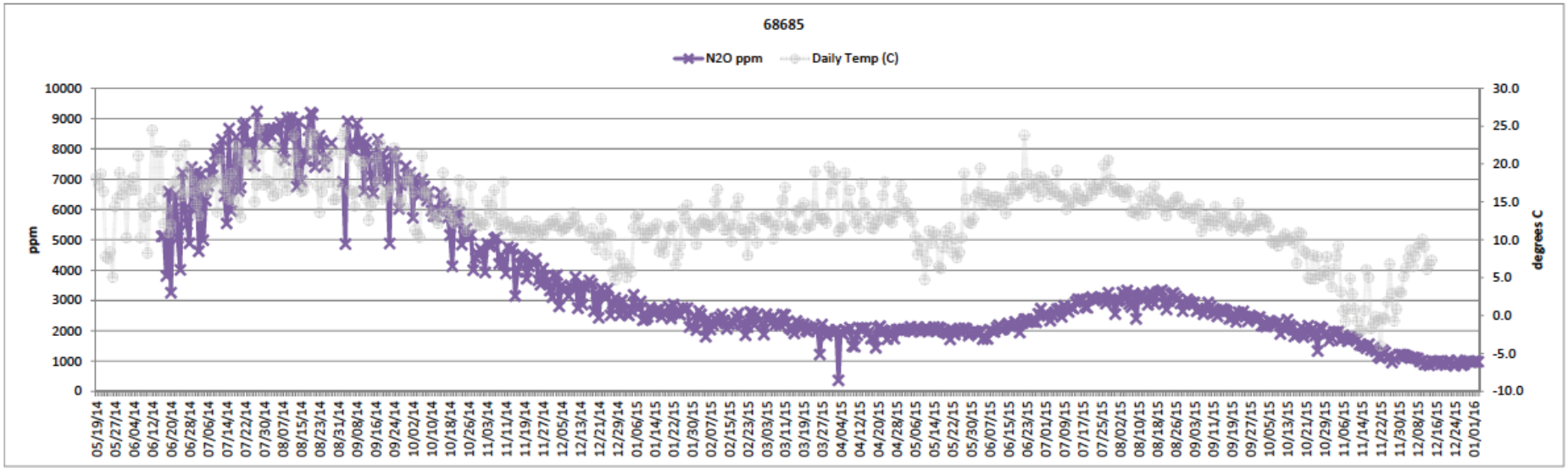
Energy diagram for exothermic reaction

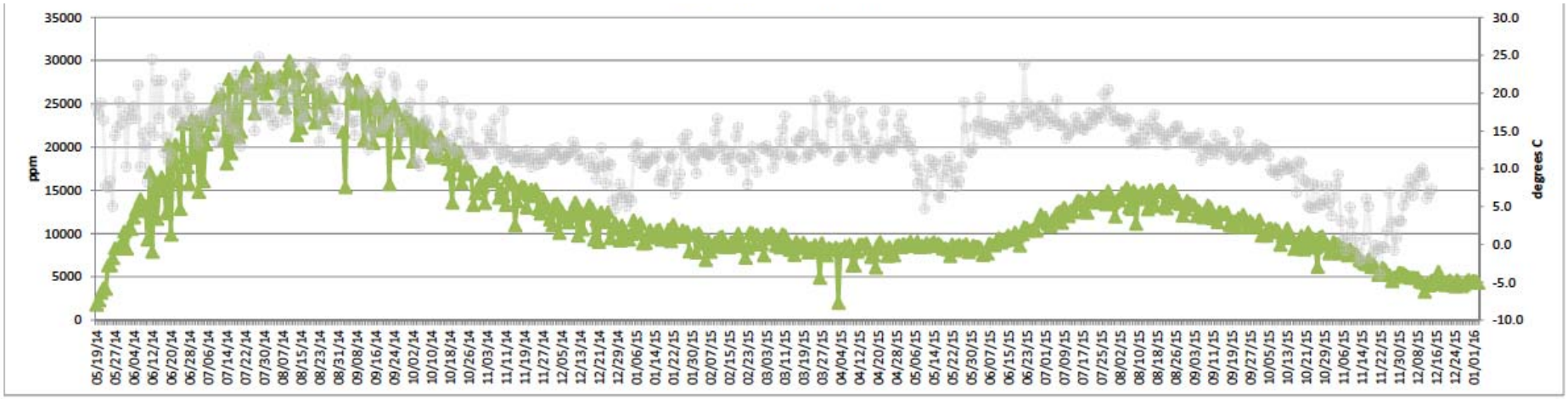
Temperature Control: Headspace Gas Analysis Indicates Decreasing Reactivity



- Robinson developed a model of headspace gas concentration that includes chemical reaction production, venting, and air exchange
- The model yielded activation energies of ~15-20 kcal/mol and heat generation rates of less than one Watt (Summer of 2014)
- Qualitatively, thermal runaway requires increasing chemical reaction and heat production – decreasing concentrations would suggest that we are on the “back side” of the reactivity curve

Temperatures and the correlated N_2O and CO_2 concentrations are at their lowest points ever





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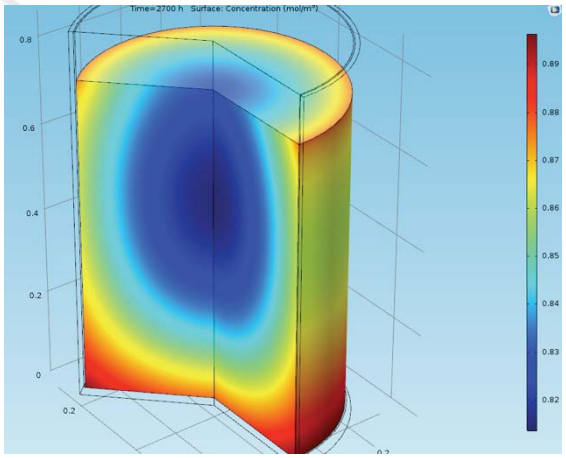
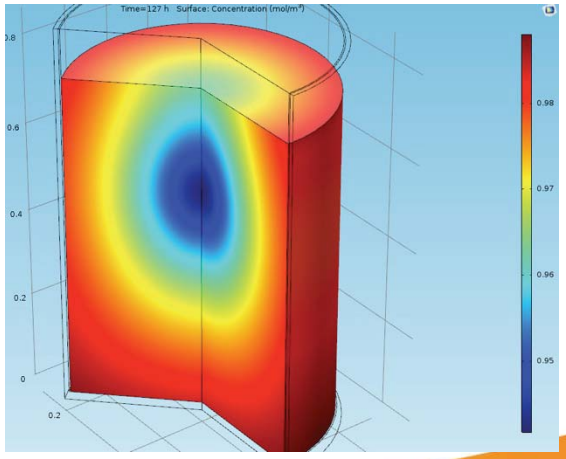
Temperature Control: simulation of drum kinetics
25 °C: no runaway after 2700 hours (top)
30 °C: runaway after 127 hours (bottom)

- Drum modeled as containing homogenous contents obeying single Arrhenius kinetic rate: $k=A*e^{-E_a/RT}$
  - Thermal conductivity derived from cold temperature tests (precise measurements in process)
  - Legend is fraction of reactants remaining
- Identical kinetics used in both calculations on right: 5 °C makes the difference between "go" and "no-go"
- Sensitivity has explored by varying A and E_a identifying those parameters that support runaway ("go")
  - Nonphysical parameters ruled out

Analysis supports our current hypothesis of drum behavior (safety increases with time, barring upset conditions): defense-in-depth

Remaining "Fuel"





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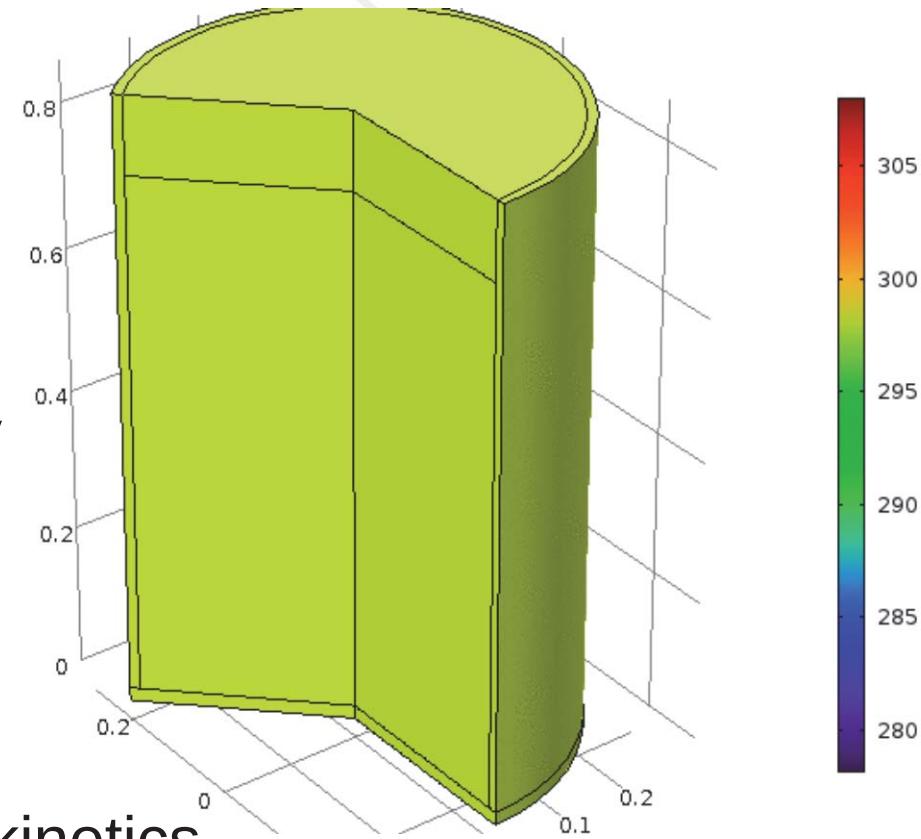
Temperature Control: Finalize process parameters using modeling informed by experiment

- Automatic Pressure Tracking Adiabatic Calorimetry (APTAC) will be used to determine thermally sensitive surrogate and establish kinetic parameters (NQA-1 Test Plan:PLAN-TA9-2243)
  - APTAC testing being conducted
  - Feeds COMSOL modeling effort
  - Finalize process parameter selection
- Differential Scanning Calorimetry (DSC) will be used to compare surrogates spiked with actinides and those created using UNS samples
  - Data expected to validate use of surrogates and evaluate effect of actinides on thermal sensitivity (use TA-55 procedures; PMT2MPRDOP-015)

Temperature Control: Simulation is guiding our process parameter selection

- Drum with homogenous contents exhibits thermal runaway in 12 days @ 25°C
- On day 11 of the simulation, the drum is placed in a refrigerator at 5 °C (boundary condition changed)
- The drum does not exhibit runaway



APTAC data generates kinetics, COMSOL used for simulation

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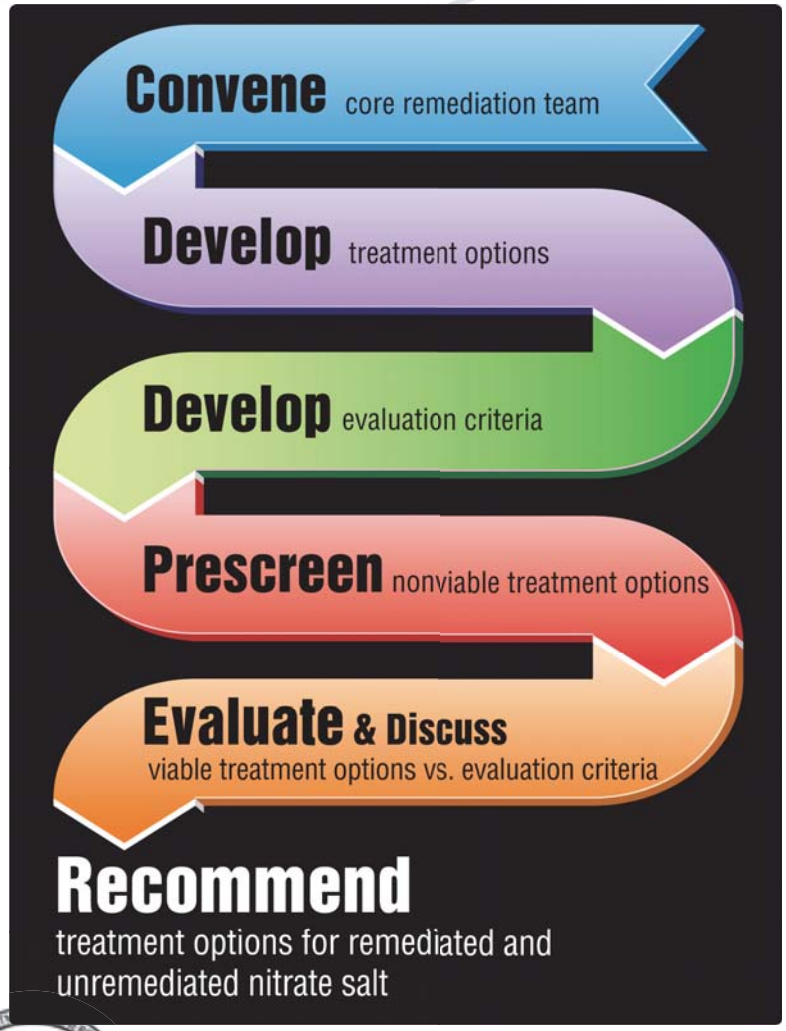
A Panel of Experts Assessed Treatment Options for the Nitrate Salt Waste

Core Team Process



Bruce Robinson — Lead

David Clark — Technical Advisor
David Funk — Technical Advisor

Enrique Torres	Benchmarking
Philip Leonard	Energetic Chemistry
Stephen Yarbro	Actinide Chemistry
Robert Wingo	Cementation
Scotty Miller	Operations
Steve Clemmons	Operations
Gian Bacigalupa	Regulatory
John Hopkins	Regulatory
Faris Badwan	Quality Assurance
Randall Erickson	ADEP
Kapil Goya	TA-55 Waste Expert
Jeff Carmichael	TA-55 Waste Expert
Andrew Baumer	FOD
Charles Conway	FOD
Rick Alexander	FOD
Robert Stokes	ES&H
Ronald Selvage	Safety Basis
Timothy Burns	Carlsbad RSO
Christopher Chancellor	Carlsbad RSO
Patrice Stevens	Project Management

Independent peer review was important for completeness

LA-UR-16-21760

Treatment Options were scored

EVALUATION CRITERIA

	POTENTIAL TREATMENT OPTIONS	Robust to waste stream variability	Ease of permitting (permitting difficulties)	Safety basis challenges	Extent of testing required	Reduction of toxicity and mobility	Reduction in volume	Short-term and long-term effectiveness	WCS implications	Scalability and complexity	Facilities challenges	Schedule	Cost (not a primary evaluation criterion) *	SCORE
1	Stabilization Using Zeolite (remediated)	5	3	4	5	4	2	4	2	4	4	4	4	41
	Stabilization Using Zeolite (unremediated)	5	3	4	5	4	2	5	N/A	5	5	5	5	43
2	Stabilization Using Zeolite With Cementation (remediated)	5	2	3	3	4	1	4	1	2	2	1	1	28
	Stabilization Using Zeolite With Cementation (unremediated)	5	2	3	3	4	1	5	N/A	3	3	2	1	31
3	Stabilization Using Dry-Process Cementation (remediated)	5	2	2	3	4	3	4	1	3	2	2	2	31
	Stabilization Using Dry-Process Cementation (unremediated)	5	3	2	3	4	3	5	N/A	4	3	3	3	35
4	Stabilization Using Wet-Process Cementation (remediated)	3	4	1	3	4	2	4	1	1	1	1	2	25
14	Salt Dissolution With Cementation/Stabilization (remediated)	3	3	1	3	4	2	4	1	2	1	2	2	26
5	Incineration		■	■										
6	Thermal Oxidation of Organics		■	■										
7	Biodegradation			■										
8	Chemical or Electrolytic Oxidation			■										
9	Chemical Reduction			■				■						
10	Vitrification									■		■		
11	Alternate Macro-Encapsulation							■						
12	Neutralization					■								
13	Controlled Reaction or Leaching of Reactive Inorganic Chemicals With Water							■						

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Zeolite addition or cementation are the top treatment recommendations for both unremediated and remediated nitrate salts

RCRA Stabilization Options	
1. Stabilization Using Zeolite	Mix waste into inorganic natural mineral to eliminate ignitability potential of the waste
2. Stabilization Using Zeolite With Cementation	Option 1 followed by production of cement waste form
3. Stabilization Using Dry-Process Cementation	Production of cement waste form with water added only at the time of cementation
4. Stabilization Using Wet-Process Cementation	Initial water addition to eliminate potential thermal runaway reactions, followed by production of cement waste form
14. Salt Dissolution With Cementation/ Stabilization	Water addition followed by filtration and cementation process of Swheat™ cake and nitrate salt solution
Other RCRA Options	
5. Incineration	Burning of waste in a radiological incinerator
6. Thermal Oxidation of Organics	Treatment of waste in air to oxidize without flame
7. Biodegradation	Biological breakdown of organics or non-metallic inorganics under aerobic or anaerobic conditions
8. Chemical or Electrolytic Oxidation	Breakdown of organics through the addition of oxidation reagents
9. Chemical Reduction	Breakdown of nitrate constituents through the addition of reducing reagents
10. Vitrification	Incorporation of waste into a glass waste form
11. Alternate Macro-Encapsulation	Coating of the waste with an organic polymer to reduce surface exposure
12. Neutralization	Reagent addition to neutralize the pH
13. Controlled Reaction or Leaching	Removal of soluble salts by leaching with water

Treatment methodologies are being evaluated for efficacy to support permit mod request

- EPA testing methodologies are being used to evaluate RCRA Characteristics of Ignitability (D001) and Corrosivity (D002)
  - Southwest Research Institute (SwRI, EPA Certified Lab)
    - Conduct SW-846 1030 (burn rate), 1050 (spontaneous combustion), UN DOT O.1 and O.2 (oxidizers), 9095B (liquids) tests
  - Initial testing in progress
    - Tests include controls and treated surrogates
      - Nitrate salts mixed in various ratios with Swheat and then mixed with zeolite (1:3) or grout
      - Initial results are confirming that the remedy is effective

After demonstration, need engineered implementation

An Engineering Options Assessment was Conducted

- Evaluation Approach
  - Characterize Waste Streams
    - RNS, UNS
  - Examine Treatment Approaches
    - Blending & Cementation
  - Evaluate Remediation/Repackaging Systems
    - WCRRF, Modulars, Gloveboxes at TA-54
    - RNS and UNS streams
    - Remaining Legacy Waste

We will be taking an additional look at our options using a broad, national team of experts

Preferred Process Options

1. **Drum blending** is easiest, fastest, best ALARA option
   - Concerns related to quality of blend and verification of mix quality
2. **Batch blending** is simple, slower than drum blending
   - Zeolite introduce in daughter drum
   - Operators will get more dose compared to drum blending
3. **Cementing in a drum tumbler**
   - Eliminates adding cement in the glovebox
   - Still requires dissolution and pH adjustment in drum
4. **Cementing in glovebox** is most difficult option
   - Add cement in glovebox
   - Mix cement in glovebox
   - Sacrificial agitator

Preferred Process Options

1. **Batch blending** is simple, slower than drum blending
   - Zeolite introduce in daughter drum
   - Operators will get more dose compared to drum blending
   - Only a 60 Drum Campaign for RNS
2. **Drum blending** is easiest, fastest, best ALARA option
   - Concerns related to quality of blend and verification of mix quality
   - Time to prove-in likely extensive
3. **Cementing in a drum tumbler**
   - Eliminates adding cement in the glovebox
   - Still requires dissolution and pH adjustment in drum
4. **Cementing in glovebox** is most difficult option
   - Add cement in glovebox
   - Mix cement in glovebox
   - Sacrificial agitator

Blending process has been developed

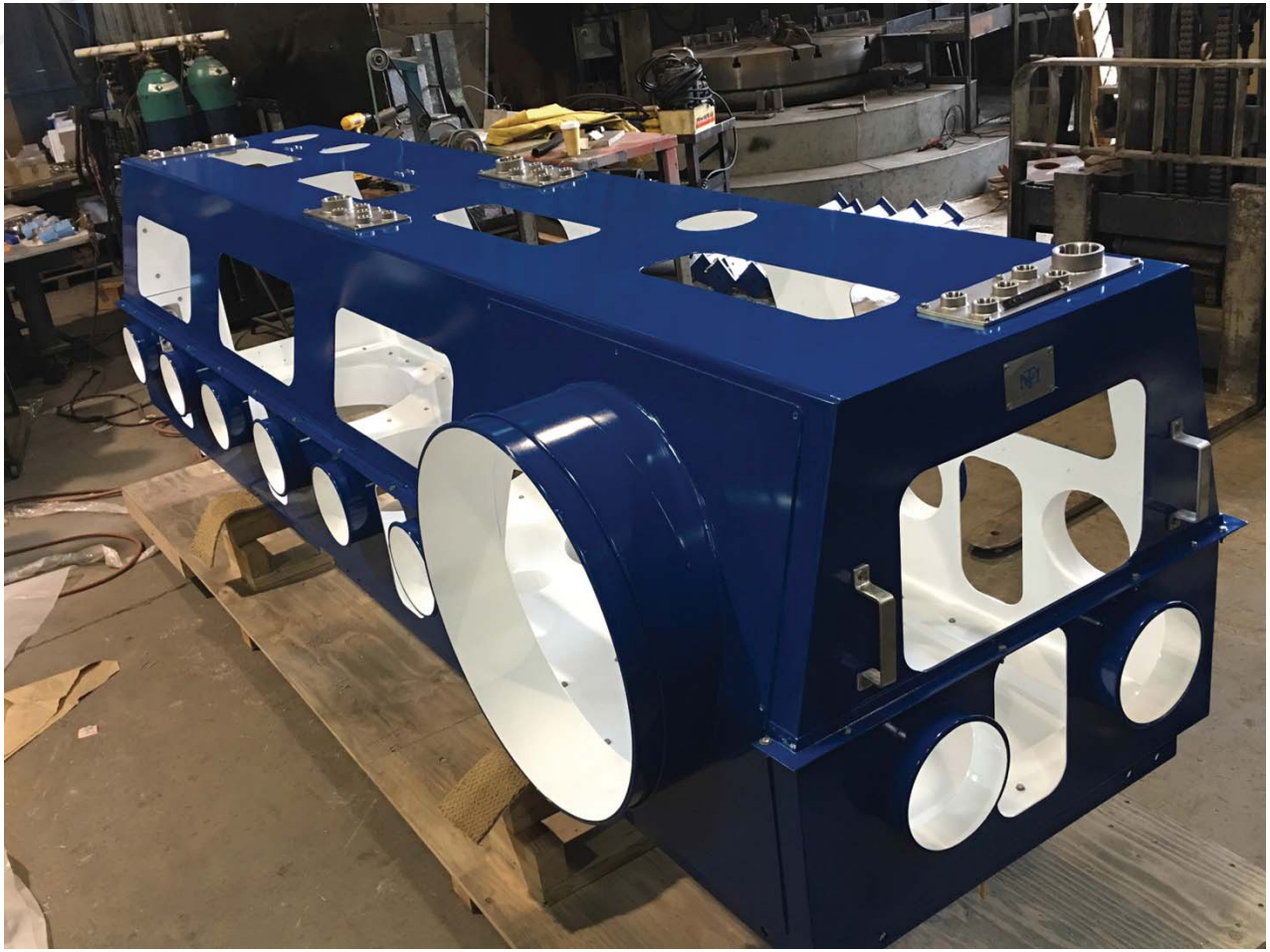
Remediation/Repackaging Systems

- Waste Characterization, Reduction, and Repackaging Facility
  - Restart WCRRF and use glovebox
- Modulars
  - MObile Visual Evaluation and Repack (MOVER)
  - MObile RepacK (MORK)
- Add Glovebox at Area G
  - 2 candidates in storage @ TA-54
  - MORK type glovebox
  - Relocate WCRRF glovebox

System Evaluation

	Blending	Cementation	Debris Waste	RNS & UNS Drums	Legacy Drums	AB Issues	RCRA Issues	Installation	Fabrication	Readiness	Operation	Schedule	Cost	Total
WCRRF	3	3	4	5	5	4	3	4	5	1	3	4	4	48
MOVER	3	1	2	5	4	4	3	4	4	1	3	3	3	40
MORK	3	2	5	5	5	2	1	2	3	1	4	1	2	36
GB 1121	5	5	5	3	3	3	3	3	2	1	4	3	3	43
GB 412	4	4	2	3	3	3	3	3	4	1	4	4	4	42
MORK GB	4	4	4	3	3	4	3	3	1	1	5	2	1	38
WCRRF GB	3	3	4	3	3	4	3	2	3	1	4	4	2	39

We have developed a mock box a for process prove in and to develop proficiency



System Options – Nitrate Salts

1. WCRRF Glovebox – Class 2 Permit Mod

- Long track record
- Infrastructure in place and tested
- Glovebox in place and tested
- BIO in place needs adjustment to handle oxidizers
- MAR limit is 800 ECPE –Ci
- Haz Cat 2 Facility – Safety Significant glovebox (Safety Class?)

2. Glovebox in Area G – Class 3 Permit Mod

- Pedigree of glovebox
- 18 ECPE-CI limitation
- Modifications and configuration issues
- Safety basis challenges

Need Safety Basis Strategy to support engineered implementation

Summary of the Anticipated Control Set for RNS Processing

- Temperature and Pressure Control: Mitigate the Possibility of Thermal Runaway During Handling
  - Work conducted to support temperature and pressure controls
    - Head Space Gas Analysis
    - Modeling – COMSOL Simulations and Small Scale APTAC studies
    - Full Scale Drum Testing – Small Scale Follow-on
- Credited Glove Box: Protection During Treatment of Waste
  - Evaluate WCRRF to validate adequacy under credible accident scenarios – can contents runaway in DBAs?
- Processing Order of the Drums
  - Process in order of increasing consequence
    - Quantity of Salt/Swheat influences consequence and likelihood

Control: Processing Order of Drums

- Thermal runaway also depends on the quantity of material and configuration (geometry)
- We can minimize consequence and establish additional confidence in our understanding by processing drums with low volume/mass of salt/ Swheat mixture (and likely low MAR) and low probability of runaway

Row Labels	Values Count of Container	Sum of Total All Inorganic (kg)
LA-CIN01.001-Cans	**1**	**143.1**
75-100%	1	143.1
LA-MHD01.001	**12**	**426.2**
0-25%	5	57.1
25-50%	6	355
50-75%	1	14.1
LA-MIN02-V.001	**47**	**4418.2**
50-75%	5	282.3
75-100%	42	4135.9
Grand Total	**60**	**4987.5**

Row Labels	Count of Container
LA-CIN01.001-Cans	**1**
SWB-55	1
LA-MHD01.001	**12**
POC	2
SWB-55	10
LA-MIN02-V.001	**47**
POC	2
SWB-55	37
SWB-POC	8
Grand Total	**60**

Summary of the Overall Steps for Treatment of Nitrate Salt Wastes

- Temperature and Pressure Control – Safing
  - Implement supplemental cooling to keep waste cool
  - Open SWBs, add vent/pressure relief to prevent accident – our top priority
- Treatment of Waste – Stabilization (zeolite/cement)
  - Treatment Study
    - Complete testing of treatment option and final waste form using surrogates
      - Spike surrogates with actinides, sample unremediated nitrate salt waste and combine with Swheat
      - Conduct comparison studies of thermal sensitivities
  - Develop Engineered Implementation
  - Treat the nitrate salt wastes: stabilization using zeolite addition or cementation

A Senior Integrated Project Team (IPT) has been stood up:

- Dave Nickless (EM-LA) and Dave Funk (LANL) co-leads
- Contracting: Chris Lockhart (EM-LA) and Jerry Ethridge (LANL)
- Safety Basis: Jim O'Neil (NA-LA) and Derek Gordon (LANL)
  - Mark Kobi, Sharon Walker (LANL)
- Regulatory: Brian Hennessey (EM-LA) and John McCann (LANL)
  - Mark Haagenstad, Luciana Vigil-Holtermann, Susan McMichael (LANL)
- Operational Readiness: Greg Jones (NA-LA) and Mandy Krenek (LANL)
  - Chris Jones (LANL)
- Engineering: Dave Nickless (EM-LA) and Larry Goen (LANL)
  - Julie Minton-Hughes and Kurt Anast (LANL)
- Maintenance: TBD
- Operations: Bill Mairson (LANL)
  - EWMO: Chuck Conway and and WD-DL (LANL)
  - Start-up: David Solms (LANL) and David Frederici (LANL)
  - Emergency Preparedness: Bill Gentile (NA-LA) and Marla Brooks (LANL)

Questions?

Appendix 5

Treatment Study Plan for Nitrate Salt Waste Remediation

LA-UR-15-27971
Approved for public release; distribution is unlimited.

Title: Treatment Study Plan for Nitrate Salt Waste Remediation Revision 2.1

Author(s): Juarez, Catherine L.
Funk, David John
Vigil-Holterman, Luciana R.
Naranjo, Felicia Danielle

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Treatment Study Plan for Nitrate Salt Waste Remediation

Revision 2.1

LA-UR-15-27971

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1.0 Introduction

The two stabilization treatment methods that are to be examined for their effectiveness in the treatment of both the unremediated and remediated nitrate salt wastes include (1) the addition of zeolite and (2) cementation. Zeolite addition is proposed based on the results of several studies and analyses that specifically examined the effectiveness of this process for deactivating nitrate salts (Walsh, 2010). Cementation is also being assessed because of its prevalence as an immobilization method used for similar wastes at numerous facilities around the DOE complex, including at Los Alamos. The results of this Treatment Study Plan will be used to provide the basis for a Resource Conservation and Recovery Act (RCRA) permit modification request of the LANL Hazardous Waste Facility Permit (Permit) for approval by the New Mexico Environment Department-Hazardous Waste Bureau (NMED-HWB) of the proposed treatment process and the associated facilities.

The specific purpose of this Treatment Study Plan is to determine the tests necessary to establish which treatment methods, zeolite addition or cementation, would be more effective at safely removing the Environmental Protection Agency Hazardous Waste Numbers (EPA HWNs) D001 and D002 from both the unremediated and the remediated nitrate salt wastes. The results of these studies will provide information to determine which treatment method is technically preferable, and will also determine the mixture volumetric quantities that are sufficient to ensure the removal of the EPA HWNs D001 and D002 (ignitability and corrosivity characteristics) as required for disposal at WIPP. The characteristic for reactivity (EPA HWN D003) has not been assigned to nitrate salt waste and further evaluation, as discussed in Section 3.2, is underway to confirm this characterization.

These tests will be performed by an independent contract laboratory, Southwest Research Institute (SWRI), located in San Antonio, Texas. Testing will be performed using non-radioactive surrogate samples to avoid the worker safety risks associated with testing, packaging, and transporting samples of the actual radioactive waste materials. Additional characterization and treatment testing activities are being conducted onsite at LANL. Results from these studies will be used to develop a workable full-scale treatment procedure for the containers currently stored at LANL.

1.1 Background

On February 14, 2014, a radiological release occurred at the U.S. Department of Energy, Waste Isolation Pilot Plant (WIPP). A breached nitrate salt waste container originating from Los Alamos National Laboratory (Los Alamos National Laboratory, 2015), was later identified as the source of the release. The waste container in question, Drum 68660, was determined to have been inadequately remediated and contained a potentially ignitable mixture of nitrate salt waste and organic absorbent material.

At the time of generation, the damp salt wastes from plutonium recovery operations were packaged in plastic bags, placed in containers, and put into storage at LANL until such time as a final disposition path was identified. In 2012 a remediation path was identified for the uncemented nitrate salt waste which included the addition of kitty litter/zeolite clay to absorb liquids in the containers. This resulted in the generation of an incompatible mixture that led to spontaneous

combustion of the waste, as documented through investigation into the WIPP event (Clark & Funk, 2015a).

From these waste processing activities, daughter containers were generated containing the absorbed liquids, nitrate salts mixed with absorbent, and debris from the parent waste container or as generated from the processing of the waste. Containers remaining at LANL include 29 of the original, unremediated nitrate salt wastes, as well as 60 containers with remediated, absorbed, and repackaged nitrate salt wastes. Containers of remediated and unremediated nitrate salt waste are characterized as exhibiting the EPAHWN D001 for ignitability (both remediated and unremediated nitrate salt waste) and D002 for corrosivity (remediated and unremediated nitrate salt waste containers with liquids only). Mixed transuranic waste with D001 and/or D002 EPA HWNs cannot be accepted for disposal at WIPP; therefore, waste treatment of both remediated and unremediated nitrate salt waste must be conducted before certification, shipment, and disposal at that facility.

1.2 Project Objectives

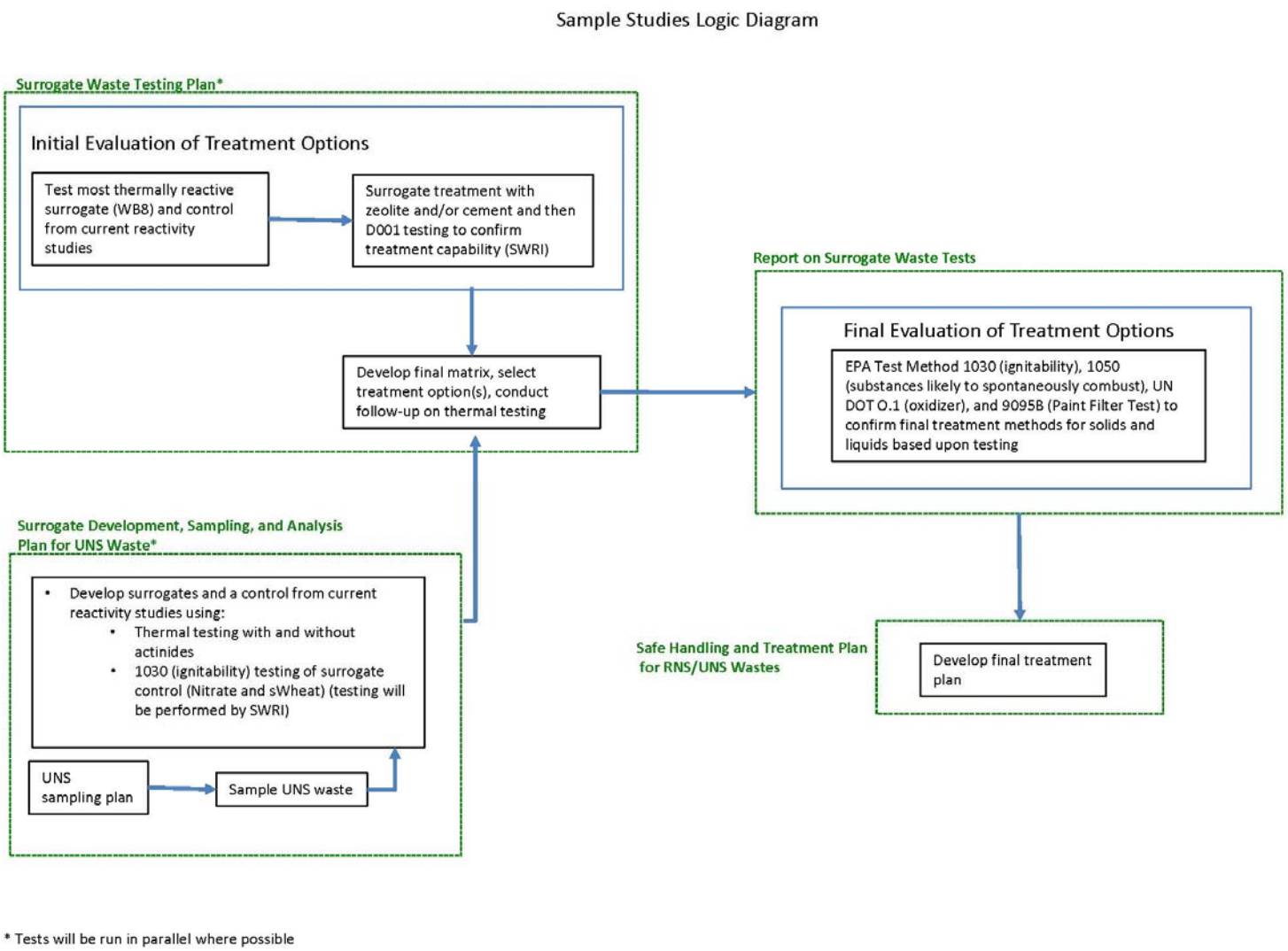
Twenty nine unremediated nitrate salt waste parent containers and 60 remediated nitrate salt waste containers must undergo treatment prior to off-site disposal. The objective of this study is to determine which treatment method, zeolite addition or cementation, will be most effective at safely removing the ignitability (D001) and corrosivity (D002) characteristics from both the unremediated and the remediated nitrate salt wastes.

The results of the treatment study plan will be used to support selection of:

1) the optimal treatment method for final remediation of the nitrate salt waste, and
2) the level of detail necessary to support an approvable permit modification to the LANL Hazardous Waste Facility Permit.

If zeolite addition is determined to be effective and selected as the treatment option, glovebox operations would be similar to those that were employed in the original processing activity (using the zeolite as an absorbent), with modifications as dictated by the results of the treatment study and other operational safety considerations. If cementation is ultimately proposed; new processing equipment would be required. Both of these options will require a modification to the Permit. Any necessary modifications will be submitted by the US Department of Energy (DOE) and the Los Alamos National Security, LLC (LANS), the Permittees, for review and approval by the NMED-HWB.

Figure 1. Proposed Sample Studies Logic Diagram.



2.0 Treatment Technology Description

Experimental and modeling studies performed at LANL indicate that mixtures of metal nitrate salts (an oxidizer) with organic kitty litter (a fuel) create the potential for an exothermic chemical reaction to occur (Clark & Funk, 2015a). This combination of materials is in the remediated nitrate salt waste. The unremediated nitrate salt waste does not include fuel and exhibits the characteristic of ignitability (D001) due to the oxidizing properties of the salts.

The first step in identifying possible treatment methodologies for both the unremediated and remediated nitrate salt waste streams included an evaluation of known available treatment options to remove the characteristics from the waste. The *Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory* (Robinson & Stevens, 2015) outlines viable treatment technologies for these types of wastes, and weighs each of the options for the nitrate salt waste located at LANL against a number of criteria that include construction/ installation at the facility. The report concludes recommending further testing on two treatment options: the addition of zeolite and cementation/grouting. The methods for each of the recommended treatment technologies are also discussed within the Options Assessment Report.

3.0 Characterization Testing to be Performed at LANL

The Permittees have undertaken various characterization efforts to better understand the properties and constituents of the remediated and unremediated nitrate salt waste. Analysis of the contents of unremediated waste containers and sensitivity testing conducted onsite at LANL is described below.

The characterization information will be used as input for preparation of non-radioactive surrogate preparation. These non-radioactive surrogates will be used to evaluate the effectiveness of the proposed treatment options, eliminating the hazards associated with the radioactive elements. To estimate the potential effect of radioactive species, small-scale testing that will include samples derived from the unremediated salt waste as well as surrogates spiked with radioactive elements will be conducted to demonstrate the equivalence of the surrogates when evaluating the proposed treatment options.

Similarly, sensitivity testing will be conducted with surrogates to evaluate their hazard potential for personnel (and the public) when processing these waste forms to remove their hazardous characteristics. This data will be crucial for establishing the appropriate controls to keep both the worker and public safe when processing these waste streams.

3.1 Sampling and Analysis of Unremediated Nitrate Salts

Unremediated nitrate salt waste containers will be sampled and analyzed for metals, other major elements, anions, radiological constituents, and pH. Analyses of the samples collected will be used to augment surrogate waste samples that will be tested off-site by SWRI, as discussed later within this plan.

Samples will be collected as described in *Sampling and Analysis Plan, Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory* (LANL, 2015). The objective of this

sampling and analyses is to obtain useful information regarding the constituents and mixtures of salts and liquids within the unremediated nitrate salt waste containers to acquire additional waste characterization information about the waste stream for use in evaluating treatment and disposal pathways. Samples of solids and liquids will be collected and analyzed at an onsite analytical laboratory. Onsite analysis is necessary in this case because the Permittees have been unsuccessful at identifying a safe and effective method for shipping previous samples of similar material. The Permittees continue to assess off-site facilities, shipment methods, and other avenues to obtain independent data from an off-site EPA-certified laboratory. Once the analysis is complete, the information will be used to prepare additional surrogates that will be tested to ensure efficacy of the treatment options and to ensure that the control set chosen will enable safe processing.

3.2 Sensitivity (Reactivity) Testing

Sensitivity testing for the EPA HWN D003 (reactivity) will include differential scanning calorimetry, vacuum thermal stability, drop weight impact testing, friction sensitivity, electrostatic spark discharge testing, and accelerated-rate pressure-tracking adiabatic calorimetry testing. These tests will be conducted on lab scale formulations of surrogate salt and salt-organic kitty litter formulations. All testing will be conducted in triplicate, and the individual results and averages will be assessed to determine the most reactive surrogate formulation. The most reactive surrogate formulation will then be used in initial testing for treatment technology effectiveness in the removal of ignitability and corrosivity characteristics from the nitrate salt waste.

Nuclear Quality Assurance (NQA-1) regulatory standards and controls will be implemented on all LANL reactivity testing. NQA standards are part of a quality assurance program for nuclear facilities that ensure that structures, systems and components important to safety are tested to quality standards.

3.2.1 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC), is a thermal analysis technique that looks at how a material's heat capacity is changed by temperature. A sample of known mass is heated or cooled and the changes in its heat capacity are tracked as changes in the heat flow. This allows the detection of transitions such as melts, glass transitions, phase changes, curing, and the determination whether the transition is endothermic (absorbs heat) or exothermic (releases heat).

In the interest of evaluating the effect of radioactive constituents on the ignitability characteristic, alternative methods were researched to evaluate whether the radioactive material acts as a catalyst to increase the burn rate or increases the likelihood of the material to self-combust. DSC will be used for this purpose.

The evaluation will be conducted in two ways. The first involves spiking the most reactive surrogate (known as WB8) with radioactive salts and running the DSC on samples with and without the radioactive constituents to examine the effect of exothermic onset of the surrogates. The second involves the testing of formulated samples using salts from the unremediated nitrate salt sampling effort and comparing to the WB8 surrogate. If the onset temperature lowers significantly (greater than experimental error), surrogate formulations may need to be altered and revisited.

3.2.2 Vacuum Thermal Stability

Vacuum Thermal Stability (VTS) is used to determine the gas generation of a material when it is held at constant, but above ambient conditions. A sample of material will be placed in a stainless steel test tube that is then inserted into a heater block set to the desired temperature. The sample tube is instrumented with a pressure transducer and all transducers are read by a computer-interfaced control box. Knowing the volume of the tube and the mass of the sample, the pressure generation during heating can be integrated to determine the volume of gas generated per gram of material. This value is compared to known stable standards for relative evaluation of thermal stability.

3.2.3 Drop Weight Impact Testing

Drop Weight Impact (DWI) is a statistical test used to measure the reaction level of a material to direct impact in order to help determine if the substance is too dangerous to transport in the form tested. In this test, a fixed volume of material is placed on a sand paper disk on top of a steel anvil. A steel striker is placed on the sample and impacted by a 2.5 kg mass falling from a predetermined height. Microphones record the sound generated by the impact. Sound above the intensity due to a blank sandpaper disk is attributed to a reaction in the material (a GO event). Sound below that intensity indicates no reaction in the material (a NO GO event). Commercial software is used to evaluate the GO and NO GO events and adjusts the required height of the 2.5 kg mass to map out the reaction probability for the material to determine the sensitivity to impact.

3.2.4 Friction Testing Sensitivity

The test is used to assess the reaction level of a material to frictional impact. In this test, a fixed volume of material is placed on a ceramic plate on a movable platform. A ceramic pin on a lever arm is lowered onto the sample and weight is added to the arm to produce a predetermined friction force. The platform is forced to move under the pin by a motor and reaction indications are assessed by the instrument operator. Smoke, sound, or black marks on the ceramic are attributed to a reaction in the material (a GO event). Lack of these features indicates no reaction in the material (a NO GO event). Commercial software is used to evaluate the GO and NO GO events and adjusts the required weight to map out the reaction probability for the material to determine the sensitivity to friction.

3.2.5 Electrostatic Spark Discharge

Electrostatic Spark Discharge (ESD) is a threshold level determination test that evaluates sensitivity of a material to a spark discharge. In this test, a fixed volume of material is added to a

sample holder that insulates the material from everything except the bottom electrode of the platform. A piece of scotch tape is placed over the sample holder, enclosing the sample area. The sample holder is placed on the platform and a needle is charged to a predetermined energy with a capacitor bank. The needle is then pushed through the tape and the energy is discharged to the bottom electrode through the sample. If the sample reacts, gas is generated and the tape is torn and sometimes obliterated. If there is no reaction, the tape is only punctured by the needle. The operator assesses the result of the test and varies the energy over a number of different replicates to determine the energy at which there are 20 consecutive NO GO events with at least one GO event at the next higher energy level. The level of the 20 consecutive NO GO events is reported as the Threshold Initiation Level.

3.2.6 Automatic Pressure-Tracking Adiabatic Calorimetry

Automatic Pressure-Tracking Adiabatic Calorimetry (APTAC) is a measurement that determines the temperature at which a material begins to self-heat and monitors the thermal and pressure behavior of that material during the self-heating. In this test, several grams of material are loaded into a titanium sample bomb that is mounted inside a furnace. The bomb is instrumented with a pressure line and thermocouple that is inserted into the sample. In a typical experiment, the sample is heated in 5 ˚C steps and the temperature is monitored at each step for some tens of minutes. If there is no indication of self-heating, the next step is taken. If the sample does begin to self-heat, the instrument switches to its tracking mode and ramps the furnace at the same rate that the sample is self-heating. This produces adiabatic conditions – the sample cannot lose heat to the surroundings. The heating stops when the heating rate exceeds the limit of the instrument or the sample temperature exceeds a predetermined threshold. The onset temperature of the self-heating is an important metric for ranking materials relative to one another in terms of thermal stability. The adiabatic nature of the measurement makes this more relevant to larger masses whose thermal conductivity may inhibit heat loss from a hot spot. The onset and rate of heating can also be used to determine kinetic parameters that allow predictions to be made for the material in other scenarios, enabling the development of process parameters for reprocessing of the remediated nitrate salt waste stream.

4.0 Off-site Testing of Treatment Methods

Treatment technology effectiveness for the addition of zeolite and cementation must be assessed for nitrate salt wastes to ensure that the RCRA characteristics of ignitability (and corrosivity where applicable) are removed from the waste after treatment. The Permittees have contracted SWRI, an EPA-certified laboratory, to conduct testing to assess the proposed treatments for the remediated and unremediated nitrate salt waste. The surrogate mixture recipes and proposed treatment methods will be provided to SWRI and all surrogate formulations and treatment testing will be created and analyzed by SWRI. This testing will be used to determine the treatment technology (addition of zeolite or cementation) that will be used to treat unremediated and remediated nitrate salt waste located at LANL. The following sections describe anticipated testing necessary to choose a single treatment method and confirm the effectiveness of that

treatment method. It is expected that this section will be updated as testing is undertaken and as we learn more about the treatment options and their effectiveness.

4.1 Initial Off-site Treatment Testing

The initial phase of treatment testing will include the formulation of surrogates based on a control formulation (potassium nitrate) and the most sensitive surrogate formulation that the Permittees have developed through onsite testing (known as WB8). These surrogates will be analyzed to confirm the presence of the ignitability characteristic. At the conclusion of these tests, the Permittees anticipate that this initial testing will lead to a selection of either addition of zeolite or cementation as the primary option.

4.1.1 Surrogate Mixtures

The surrogate salts created for the initial testing will be based upon studies conducted at LANL in Section 3.2 to ensure that the most sensitive surrogate to date is the surrogate created and tested (known as WB8). The second surrogate is a control surrogate (consisting of potassium nitrate only) and will be used to test the simplest surrogate of the nitrate salt waste. SWRI will utilize the recipes shown in Table 1 for the blending and testing of the treated surrogates to make a determination of treatment effectiveness.

Table 1. Surrogates for Initial Treatment Testing

Test ID	KNO_3 (g)	WB8 Salt (g)	Salt : SWheat Vol Ratio
Blend 1	50	0	NA
Blend 2	50	0	1:1
Blend 3	50	0	1:3
Blend 4	50	0	1:4
Blend 5	0	50	NA
Blend 6	0	50	1:1
Blend 7	0	50	1:3
Blend 8	0	50	1:4

4.1.2 Zeolite Blending

The recipes that will be tested for zeolite blending represent the remediated nitrate salt and unremediated nitrate salt waste as outlined in Blends 1, 5-8 in Table 1. The zeolite used will be KMI Zeolite, 100% Multipurpose Zeolite (14 X 40 mesh). Free liquids (mainly in the unremediated nitrate salt waste stream surrogates) will first be absorbed with zeolite and then the resulting wet zeolite is blended at the same test ratio (1:1, 3:1 or 4:1) with dry zeolite. Table 2 summarizes this plan.

Table 2. Initial Zeolite Blending

Test ID	KNO_3 (g)	WB8 Salt (g)	Salt : SWheat Vol Ratio	Water: Salt/SWheat Ratio	Zeolite : (Salt/SWheat) Vol Ratio
Zeolite 1	50	0	NA	NA	1:3
Zeolite 2	0	50	NA	NA	1:3
Zeolite 3	0	50	1:1	1:1	1:3
Zeolite 4	0	50	1:3	1:1	1:3
Zeolite 5	0	50	1:4	1:1	1:3

4.1.3 Cementation

The recipe for cementing the surrogate waste with Type I/II Portland Cement is shown in Table 3.

Table 3. Cementing Recipes

Test ID	KNO_3 (g)	WB8 (g)	SWheat (g)	Water (g)	NaOH (g)[1]	Cement (g)
Cement 1	100			300	~2	400
Cement 2		100		300	~55	400
Cement 3		100	33	300	~55	400
Cement 4		100	100	400	~55	535
Cement 5		100	133	530	~55	710

[1] 10 molar NaOH – values are estimates – requires a solution pH of 9

4.1.4 Analytical Testing

In order to prove that one or both of the treatment methods was successful at removing the characteristics of ignitability and corrosivity analytical testing must be conducted. The objective of ignitability of solids (EPA Test Method 1030) and oxidizer (DOT oxidizer test UN Test 0.1) potential tests of this treatment study plan are intended to:

1) determine if the nonradioactive nitrate salts samples, salt mixed with kitty litter, are ignitable as either wet or dry materials;
2) identify the combination of salt sample and SWheat/salt ratio that burn remediated nitrate salts most vigorously; and
3) evaluate the amount of zeolite required to render the mixture a non-oxidizing solid.

Analysis of the surrogates will be conducted in accordance with quality assurance (QA)/quality control (QC) procedures defined by the latest revision of SW-846, or other Department-approved procedures. Analytical data generated by the treatment method testing on surrogate nitrate salt waste activities described in this section will be verified and validated. Data reduction is the

conversion of raw data to reportable units, transfer of data between recording media, and computation of summary statistics, standard errors, confidence intervals, and statistical tests.

The laboratory will describe the analysis in sufficient detail so that the data user can understand how the sample was analyzed. Analytical reports will include:

- a summary of analytical results for each sample;
- results from QC samples such as blanks, spikes, and calibrations;
- reference to standard methods or a detailed description of analytical procedures; and
- raw data printouts for comparison with summaries.

EPA SW-846 Test Methodology and Department of Transportation (DOT) procedures will be utilized to test the properties of ignitability of the salt mixtures, zeolite blending, and cementation.

EPA Test Method 1050 provides test procedures which may be used to evaluate and categorize liquid and solid wastes that are likely to spontaneously combust.

Analyses as summarized in Table 4 will be conducted to narrow down a single treatment method (zeolite blending or cementation) for final testing and experimentation with other aspects of the waste (e.g. liquids, neutralizers, and debris) in Phase 2 of off-site testing. After the analyses in Table 4 are complete, if both cementation and zeolite blending are viable treatment methods for nitrate salt waste located at LANL, the preferred method will be chosen and further tested for effectiveness.

Table 4. Initial Analyses Required

Surrogate Description (vol:vol ratios)	SW-846 Test Method 1030	SW-846 Test Method 1050	DOT O.1 Testing
Blend 1			X
Blend 2	X	X	X
Blend 3	X	X	X
Blend 4	X	X	X
Blend 5			X
Blend 6	X	X	X
Blend 7	X	X	X
Blend 8	X	X	X
Zeolite 1			X
Zeolite 2	X	X	X
Zeolite 3	X	X	X
Zeolite 4	X	X	X
Zeolite 5	X	X	X
Cement 1			X
Cement 2			X
Cement 3	X	X	X
Cement 4	X	X	X
Cement 5	X	X	X

4.2 Final Off-site Treatment Testing

The final phase of treatment testing will utilize only a single treatment method (zeolite blending or cementation) to develop ratios and verify all of the waste present at LANL can be treated through the chosen treatment method. To fully test the treatment method effectiveness on all known components of the unremediated and remediated nitrate salt wastes, future testing is expected to be necessary. Any additional surrogate(s) will be developed from the analyses of unremediated nitrate salt waste and various sensitivity tests described in Section 3. Surrogate(s)

will be tested as described in the following sections to ensure confirmation of treatment effectiveness.

Table 5. Final Evaluation of Waste Surrogates

Requested Analysis				
Surrogate Description (vol:vol ratios)	**SW-846 Test Method 1030**	**SW-846 Test Method 1050**	**DOT O.1 Testing**	**Test Method 9095B (Paint Filter)**
Solid Surrogate(s)				
UNS			X	
UNS + SWheat 1:1	X	X	X	X
UNS + SWheat 1:3	X	X	X	X
UNS + SWheat 1:4	X	X	X	X
1 UNS 3 zeolite or cemented			X	X
(UNS + SWheat 1:1):3 zeolite or cemented	X	X	X	X
(UNS + SWheat 1:3):3 zeolite or cemented	X	X	X	X
(UNS + SWheat 1:4): 3 zeolite or cemented	X	X	X	X
Liquid Surrogate				
(UNS Liquid + SWheat 1:1):3 zeolite or cemented	X	X	X	X
1 (UNS Liquid + SWheat 1:1):1 Spillfyter:3 zeolite or cemented	X	X	X	X
1 (UNS Liquid + SWheat 1:1):1 Spillfyter:1 citric acid:3 zeolite or cemented	X	X	X	X
1 (UNS Liquid + Wastelock 16:1):3 zeolite or cemented	X	X	X	X
Debris Surrogate				
Debris contaminated with a mixture of salt/SWheat (20%)			X	
Debris contaminated with a mixture of salt/SWheat (15%)			X	
Debris contaminated with a mixture of salt/SWheat (5%)			X	

4.2.1 Debris Testing

The remediated nitrate salt (RNS) waste drums contain debris. The debris is typically composed of plastic, cardboard, rubber gloves, rags and lead. It is unclear if the debris that has been comingled with RNS waste should carry the D001 code for an oxidizer. To examine this aspect of the waste stream, various tests are requested. Samples of the debris types commonly found in RNS waste drums will be subjected to environments that simulate the conditions in the RNS drums and those samples will be tested to see how they respond to SW-846 Test Method 1030 and SW-846 Test Method 1050.

Table 6. Testing of Debris Samples

Debris Type	SW-846 Test Method 1030 **	SW-846 Test Method 1050 **
Cardboard 1	3*	3*
Cardboard 2	2	2
Plastic 1	3*	3*
Plastic 2	2	2
Rubber glove 1	3*	3*
Rubber glove 2	2	2
Rag 1	3*	3*
Rag 2	2	2

* Number of tests includes a baseline test of material not treated with solution or blended
** If the initial 1030 or 1050 test does not pass, do not perform a duplicate

The information contained in the tables above are what are proposed based upon current knowledge. The tables may be modified as more knowledge is garnered along the testing process.

5.0 Other Treatment Evaluations Performed at LANL

As part of the ongoing planning to execute safe, and efficient treatment of unremediated and remediated nitrate salt wastes; two supplemental evaluations have been conducted at LANL. These include an examination of engineering systems available that could be utilized for treatment of unremediated and remediated nitrate salt waste at LANL, and a plan for blending tests to determine how to ensure adequate mixing of the waste and the chosen treatment method (i.e. zeolite blending or cementation).

The *Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing* (Anast, 2015) details the examination of six processing/repackaging systems for their applicability to support zeolite blending and cementation of nitrate salt waste.

The evaluation concluded that the Waste Characterization, Reduction and Repackaging Facility (WCCRF) glovebox was the preferred system to use for processing unremediated and remediated nitrate salt waste containers located at LANL.

Blending is the preferred approach to remediate the RNS drums and will be tested initially to identify appropriate equipment for blending and to evaluate the effectiveness of the equipment to adequately blend the salt/Swheat waste with zeolite. Blending scoping tests will be conducted to determine the equipment and the optimal approach that could be best utilized for the physical mixing of the nitrate salt waste with zeolite. This testing will examine a total of three approaches to blend surrogate salt/SWheat mixtures with the chosen treatment method. The first approach is a batch process using a KitchenAid (KSM8990) 8 quart bowl commercial stand mixer. These units will easily fit into the WCCRF glovebox. The other two approaches are drum blending processes. Drums will be loaded with bulk zeolite and then the surrogate material added. The contents will then be blended in the drum using a drum tumbler or a drum roller. Internal baffles may be added to the interior of the drum to aid in blending. Cementation process options will be evaluated if the results from LANL characterization testing and offsite testing indicate zeolite blending is not effective and cementation is effective. Once scoping tests are completed, focused surrogate testing will be planned and carried out to provide large scale verification, sampling and quantitative analyses using the candidate equipment, recipe and procedure.

6.0 Results and Conclusions

Sections 3, 4, and 5 of this treatment study plan outline the testing and evaluations that have been or will be conducted to determine the treatment methodology for nitrate salt waste containers located at LANL. Upon completion, a report will be drafted and submitted to the NMED-HWB. The report will be accompanied by or be drafted closely before the submittal of permit modification request(s) necessary to include the proposed activities into the LANL Hazardous Waste Facility Permit.

7.0 References

Anast, K. (2015). *An Engineering Options Assessment Report*. (Currently in draft form and undergoing peer review).

Clark, D. L., & Funk, D.J. (2015a). *Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes*. (LANL Report ESHID-600350; ENV-DO-15-0097; LA-UR-15-22393). Los Alamos, NM: Los Alamos National Laboratory. http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600350

Clark, D.L., & Funk, D.J. (2015b). *Transmittal of Los Alamos National Laboratory Information Regarding Legacy Nitrate Salt Waste Containers.* (LANL Report ESHID-600216; ENV-

DO-15-0007; LA-UR-15-20084). Los Alamos, NM: Los Alamos National Laboratory. http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600216

Los Alamos National Laboratory. (2015). *Transmittal of Sampling and Analysis Plan for Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory.* (ESHID-600920; LA-UR-15-26357; ENV-D15-0248). Los Alamos, NM: Los Alamos National Laboratory. http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600920

Robinson, B. A., & Stevens, P.A. (2015). *Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory*. (LANL Report LA-UR-15-27180). Los Alamos, NM: Los Alamos National Laboratory.

Walsh, G. (2010). *Results of Oxidizing Solids Testing*. (EMRTC Report RF 10-13 prepared for Washington TRU Solutions, LLC.). Socorro, NM: Energetic Materials Research and Testing Center, New Mexico Institute for Mining and Technology (Certified DOT Testing Laboratory).

Appendix 6

Statistical Modeling Effects for Headspace Gas

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Title: Statistical Modeling Efforts for Headspace Gas

Author(s): Weaver, Brian Phillip

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Statistical Modeling Efforts For Headspace Gas

Brian Weaver, CCS-6

March 15, 2016

The purpose of this document is to describe the statistical modeling effort for gas concentrations in WIPP storage containers. The work was performed primarily by Brian Weaver of CCS-6 (Statistical Sciences) and included input from Joanne Wendelberger (CCS-6), Bruce Robinson (ADEP), David Funk (ADEP), and Eric Heatwole (M-6).

Headspace Gas Data

Figure 1 shows the concentration (in ppm) of CO_2 in the headspace volume of standard waste box (SWB) 68685. The different colors represent the temperature that the measurement was taken where red denotes higher temperatures (in Celsius) and blue denotes lower temperatures. The data spans from May 19, 2014, to February 3, 2015. The goal of this analysis is to utilize the information within this data, along with current physics knowledge, to predict what future concentrations levels will be.

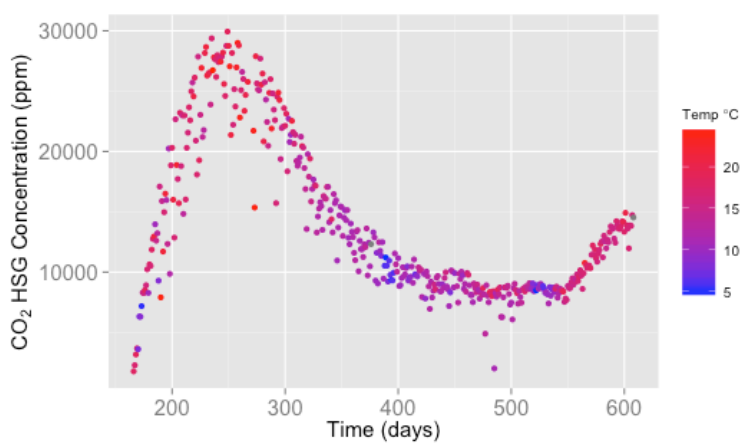

Figure 1: CO_2 gas concentration as a function of time and temperature (represented by color)

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Modeling Efforts

Physical Model

Let $C(t,T)$ denote the concentration of a particular gas at time t (in days) in a headspace container at temperature T. Then the concentration changes according to the following model:

$$V_{HSG}\frac{dC}{dt} = -Q_{out}C(t,T) + Q_{in}C_{in} + M(t,T), \tag{1}$$

where

$$\begin{aligned} Q_{out} &= Q_{in} + Q_{gen}, \\ Q_{gen} &= \frac{M(t,T)R_1T}{P_{HSG}X_g}, \\ M(t,T) &= \chi(T)e^{-\beta t}, \\ \chi(T) &= Ae^{-E_a/R_2T}. \end{aligned}$$

The first term $-Q_{out}C(t,T)$ describes how the gas flows out of the SWB into the atmosphere, $Q_{in}C_{in}$ describes the flow of gas from the outside atmosphere into the SWB, and $M(t,T)$ describes how gas is generated by the substances of interest within the SWB for temperature T. $C(t,T)$ is given as the solution to the differential equation in Equation (1) and must be solved using numerical methods.

In this model, the unknown parameters, denoted by the vector θ, are $\theta = (Q_{in}, A, E_a, \beta)$ and are to be estimated using the data collected from the headspace volume. The remaining parameters are known and their values are given in Table 1.

Quantity	Value
P_{HSG}	1
R_1	0.08206
R_2	1.987×10^{-3}
X_g	0.429
C_{in}	400 (for CO_2)

Table 1: Known quantities and their values in Equation (1)

Data Model

Let $Y(t,T)$ represent the random variable associated with the measured concentration at time t for temperature T and let y be an observation of Y. Then our statistical model is

$$Y(t,T) = C(t,T) + \varepsilon \tag{2}$$

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where ε represents random deviations from the physical model. Initially we assume that $\varepsilon \sim N(0, \sigma^2)$ independently. Here $\sigma > 0$ is the standard deviation of the random deviations. σ is also an unknown quantity and so it is estimated and added to our vector θ.

Bayesian Statistical Model

We use a Bayesian approach for estimating θ. The posterior distribution, $p(\theta|y_1, \ldots, y_n)$, is obtained using

$$p(\theta|y_1, \ldots, y_n) \propto L(\theta; y_1, \ldots, y_n)p(\theta)$$

where $L(\theta; y_1, \ldots, y_n)$ denotes the likelihood function and is derived using Equation (3) and $p(\theta)$ is the prior distribution for θ. The purpose of the likelihood is to describe which values of θ are most plausible (in some sense) given the observed data. $p(\theta)$ represents our current state of knowledge about θ (before observing any data) in the form of a probability distribution function. The posterior distribution is then a reweighting of $p(\theta)$ based on the information in the data through the likelihood. For this effort we assume uniform (flat) priors for our unknown parameters. Table 2 gives the upper and lower bound for these distributions for each parameter.

Quantity	Lower Bound	Upper Bound
Q_{in}	0	1
A	0	1,000,000
E_a	0	100
β	0	100
σ	0	100

Table 2: Upper and lower bounds for the uniform prior distributions assigned to the unknown parameters θ

Data Analysis

An adaptive Metropolis-Hastings algorithm was used to obtain draws from the posterior distribution for θ. Table 3 gives the posterior point estimates for θ along with the upper and lower values for their corresponding 95% credible intervals.

The posterior estimate of $C(t, T)$, along with its 95% credible interval is given in Figure 2. Notice that the physics model tends to capture the general trend of the data but is discrepant in some specific features. For example, the main peak for the data tends to occur earlier than described by our model.

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Quantity	Estimate	Lower Bound	Upper Bound
Q_{in}	0.0014	0.00094	0.0095
A	378829.3	24395.5	516189.4
E_a	15.315	15.061	15.550
β	2.44×10^{-8}	2.26×10^{-8}	2.62×10^{-8}
σ	2254.2	2119.7	2426.1

Table 3: Posterior summaries for the unknown parameters θ

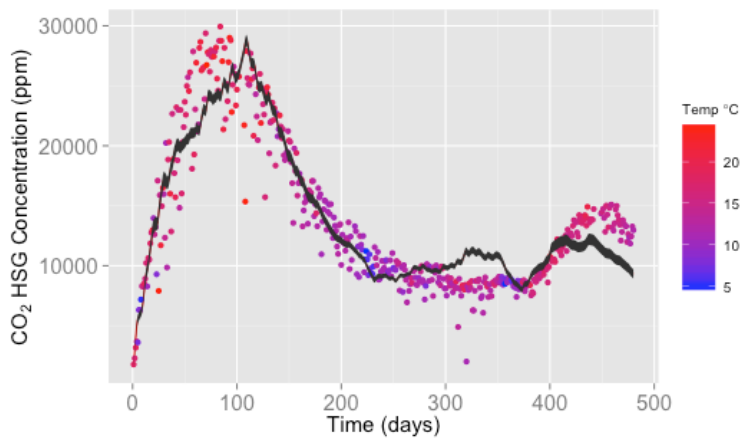

Figure 2: Posterior estimate of CO_2 gas concentration as a function of time and temperature along with its corresponding 95% credible interval (gray ribbon)

Figure 3 displays the residuals for the model fit, i.e., $Y(t,T) - \hat{C}(t,T)$ where $\hat{C}(t,T)$ is the estimate for the gas concentration as a function of both time (along the x-axis) and temperature (again indicated by color). The most striking feature is the large variability for earlier times. Additionally, it appears that the model is predicting higher gas concentrations for later times (say times larger than 350 days) than what is observed in the data.

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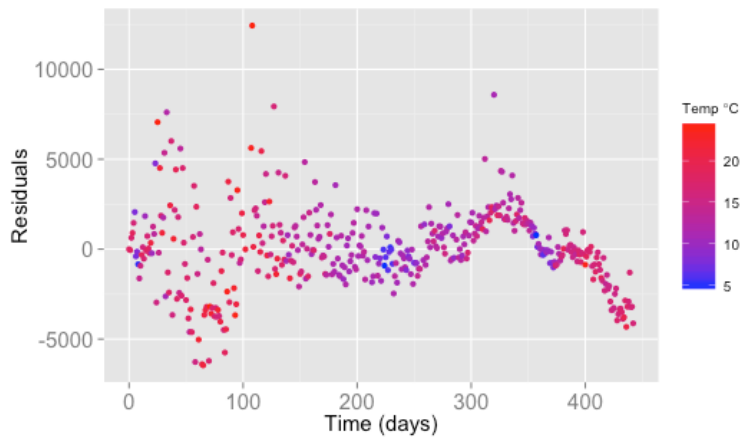

Figure 3: Model residuals as a function of time (x-axis) and temperature (color)

Lastly, Figure 4 displays concentration predictions for the last seven observations which were not used in the parameter estimation. The black points represent the posterior prediction and the vertical bars represent a 95% prediction interval. The actual observation is given as a red point. In all of these cases, the model has predicted the observation well because each of the red dots resides within the prediction interval.

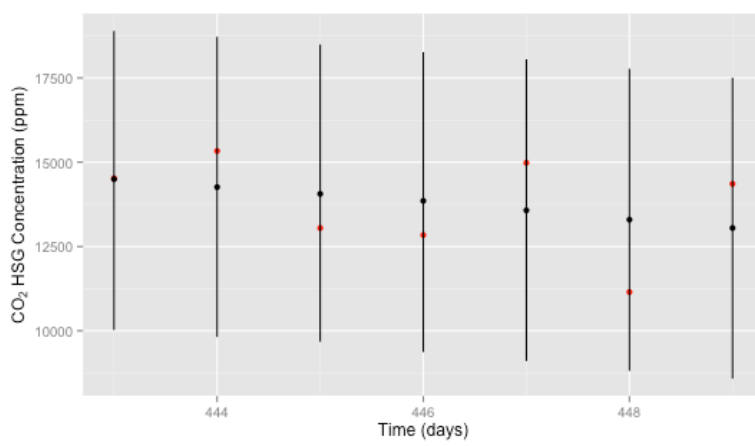

Figure 4: Posterior predicted gas concentrations (black dots) with corresponding 95% prediction intervals. The actual observations are given by red dots.

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Potential Model Enhancements and Proposed Areas for Future Work

Figures 2 and 3 indicate various discrepancies associated with our full statistical model given in Equation (3). First, recall that in Figure 3 the variability in the residuals decreases as a function of time. This is a clear violation of our constant variance assumption in Equation (3). It is believed this change in variability is due to the researcher making the gas concentration measurements getting better at making the measurements with time. One potential improvement to our statistical model would be to incorporate this time dependence into the measurement error portion of the statistical model:

$$Y(t,T) = C(t,T) + f(t)\sigma\epsilon \tag{3}$$

for some appropriate function of time $f(t)$ and where $\epsilon \sim N(0,1)$.

One assumption to the physics model in Equation (1) is that gas flow is only occurring between the SWB and the surrounding atmosphere. It is observed in Figure 2 that physics model seems to be missing the peak concentration by about two weeks. This could in part be due to the additional flow of gas from the drum within the SWB and the atmosphere in the SWB. In total, gas can flow between the drum and the SWB and then between the SWB and the surrounding atmosphere. By accounting for the additional avenue of gas flow might help shift the peak concentration predicted by the model to what is observed in the data. A potential physics model could take the following form:

$$\frac{dC_2}{dt} = Ae^{-Ea/RT}e^{\beta t} - Q_{out,2}C_2 + Q_{in}C_1 \tag{4}$$

$$\frac{dC_1}{dt} = Q_{in}C_2 - Q_{out,1}C_1 + Q_{atm}C_{atm} \tag{5}$$

where C_2 and C_1 are the gas concentrations in the drum and SWB, respectively, $Ae^{-Ea/RT}e^{\beta t}$ describes the gas being added to the drum from chemical reactions, $Q_{out,2}C_2$ describes the gas leaving the drum and entering the SWB, $Q_{in}C_1$ describes the flow of gas from the SWB into the drum, $Q_{in}C_2$ describes the flow of gas from the drum to the SWB, $Q_{out,1}C_1$ describes the flow of gas from the SWB to the atmosphere, and $Q_{atm}C_{atm}$ describes the flow of gas from the atmosphere into the SWB.

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Appendix 7

Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing

LA-UR-15-28900
Approved for public release; distribution is unlimited.

Title: Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing

Author(s): Anast, Kurt Roy

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November 2015
Revision A

Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing

LA-UR-15-28900

Prepared by Kurt Anast

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LA-UR-15-28900

EXECUTIVE SUMMARY

This report examines and assesses the available systems and facilities considered for carrying out remediation activities on remediated nitrate salt (RNS) and unremediated nitrate salt (UNS) waste containers at Los Alamos National Laboratory (LANL). The assessment includes a review of the waste streams consisting of 60 RNS, 29 aboveground UNS, and 79 candidate belowground UNS containers that may need remediation. The waste stream characteristics were examined along with the proposed treatment options identified in the Options Assessment Report[1]. Two primary approaches were identified in the five candidate treatment options discussed in the Options Assessment Report: zeolite blending and cementation. Systems that could be used at LANL were examined for housing processing operations to remediate the RNS and UNS containers and for their viability to provide repackaging support for remaining LANL legacy waste.

The waste streams for RNS and UNS differ not only in the presence of organic kitty litter found in the RNS drums but also in the amount of and type of debris as well as the free liquid content. RNS drums contain significant volume percentage of debris waste while the UNS waste is relatively free of debris. Conversely, RNS drums are nearly free of free liquids while the UNS drums all can be expected to contain free liquids. These differences, along with the related radiological makeup, were considered when assessing the treatment process and associated containment systems.

The preferred treatment option is blending the waste with zeolite (although the efficacy of this option needs to be confirmed early with ignitability [D001] testing). Blending with zeolite was the top remediation option identified in both the Options Assessment Report[1] and was originally proposed as the best option for remediation by Clark and Funk in their report, Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes[2]. It would also be the least complex to install and implement in the available glovebox systems. Blending requires little or no modification to the glovebox, is operationally simple, and has been shown to be effective at treating nitrate salt surrogates to remove the ignitability (D001) characteristic[3]. Two approaches were considered: batch blending (1) using 3- to 5-gallon blenders to batch blend nitrate salt waste with zeolite in the glovebox or (2) adding salt waste directly to drums preloaded with zeolite and bulk blending in the drum using a drum tumbler. Bulk blending in the drum is the preferred option but will require extensive proof testing. This option, if effective, is less complicated and reduces the radiation dose to operators. The fall-back option would be batch blending in the glovebox.

Cementation is more complex to install, is operationally more complicated, and adds additional risks. The cementation process requires repulping the salt/Swheat in water, adjusting the pH, transferring cement, mixing cement, and curing the product. Accommodating these operations requires installing equipment, modifying the glovebox and the facility, and adding complexity to the operations. Additionally, the cementation process is not reversible, is time dependent, and generates heat—all of which add risk.

[1] Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory (B.A. Robinson, P.A. Stevens)

[2] Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes (D.L. Clark, D.J. Funk: LA-UR-15-22393)

[3] Results of Oxidizing Solids Testing (Energetic Materials Research and Training Center Report FR 10-13) (G. Walsh, New Mexico Institute of Mining & Technology, Socorro, NM; March 2010)

Six processing/repackaging systems were examined and assessed for their applicability to support zeolite blending and cementation of RNS and UNS waste streams. These systems options were as follows:

- Waste Characterization Reduction and Repackaging Facility (WCRRF) glovebox
- Mobile Visual Examination and Repackaging (MOVER) trailer
- Mobile Repackaging (MORK) system
- Modification of available on-site gloveboxes for placement in 231 Perma-Con®
- Fabrication of a new glovebox
- Relocation of the WCRRF glovebox

The preferred processing/repackaging system is the WCRRF glovebox because it provides the least risk, least equipment and facility modifications, least authorization basis (AB) modification, adequate flexibility, and likely the optimal path to remediating the nitrate salt drums. The glovebox is well configured to accommodate blending with zeolite but is less amenable to supporting cementation, especially cementing in the glovebox because of space limitations and material-handling requirements associated with the cementation process. The WCRRF Basis for Interim Operation is already in place, and updating to allow for nitrate salt processing should be straightforward because similar operations have been performed at WCRRF, although not with the same hazards. The infrastructure is in place and has been well tested for the last 20 years. Transporting the waste and refrigerating it at WCRRF are negative aspects of this option because they introduce additional cost, safety concerns, and coordination difficulties. The reliability of a proven, tested, and operating glovebox that is approved for 800 equivalent combustible plutonium-equivalent curies (ECPE-Ci), compared with modifying or relocating competing systems that may require modification, have no operating record, and have no current LANL AB, make WCRRF the best choice for the short term to handle the nitrate waste streams.

Installing a glovebox in a Perma-Con® in Building 231 (a fabric-covered dome) to support nitrate waste repackaging and the remaining LANL legacy waste could provide added flexibility and may be a relatively inexpensive option to augment repackaging, depending upon AB requirements. Two issues need to be resolved for this option: (1) the necessity to provide a Safety Significant glovebox for worker protection and (2) the allowable ECPE-Ci for any drum in process. These are both AB issues that should be analyzed to determine if they can easily be resolved before moving forward with this option. The safety basis control will impact the specifics of the glovebox that may be utilized, the design and fabrication/modification requirements, and ultimately, the operating requirements. The flexibility to configure a new glovebox for drum repackaging and locate it in an open floor plan like a Perma-Con® room is an attractive option for a large subset (~3900 drums) of legacy waste that contain less than 18 ECPE-Ci.

If an additional capability is desired at Technical Area 54 for higher content plutonium-equivalent curie (PE-Ci) legacy waste containers, WCRRF can be utilized until a new system is installed, configured, tested, and approved for use. This ensures a repackaging capability is available and mitigates schedule risk that may be associated with initiating a new system. MORK, the only other system evaluated that is designed to handle more than 18 ECPE-Ci, has hurdles that must be overcome, including decontamination, transportation, and siting to meet seismic requirements. Maintaining the WCRRF glovebox operation ensures a viable capability until an alternate system can be approved, installed, tested, and brought online.

A concern that remains unresolved is the path forward for debris found in the RNS and UNS waste containers. It is unclear if the debris stream should be considered D001 and requires treatment. Early surrogate testing to determine if debris waste separated from the RNS or UNS drums is ignitable (D001) should be initiated. Transuranic debris waste that is D001 cannot be sent to the Waste Isolation Pilot Plant unless the D001 characteristic is removed. Results from surrogate testing will drive handling and processing this waste stream after it is separated from the salt wastes.

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Acronyms and Abbreviations

AB	authorization basis
ALARA	as low as reasonably achievable
BIO	basis for interim operation
CAM	continuous air monitoring
CC	contributing causes
CCP	Central Characterization Project
CFR	Code of Federal Regulations
CMR	Chemistry and Metallurgy Research (facility)
DOT	Department of Transportation (U.S.)
dpm	disintegrations per minute
DSA	documented safety analysis
ED	electrostatic discharge
ECPE-Ci	equivalent combustible plutonium–equivalent curies
EMRTC	Energetic Materials Research and Testing Center
EPA	Environmental Protection Agency (U.S.)
FGE	fissile gram equivalent
FOD	Facilities Operation Division

GBE	walk in glovebox
HEPA	high-efficiency particulate arresting
hp	horsepower
HVAC	heating, ventilation, and air conditioning
JON	judgement of need
LANL	Los Alamos National Laboratory
LLNL	Lawrence Livermore National Laboratory
LLW	low-level waste
LTA	less than adequate
MAR	material at risk
MLLW	mixed low-level waste
MORK	Mobile Repackaging (system)
MOVER	Mobile Visual Examination and Repackaging
MSA	management self-assessment
MU	modular unit
NEPA	National Environment Policy Act
NMED	New Mexico Environment Department
ORR	operational readiness review
PAAA	Price-Anderson Amendments Act
PC	performance category
PDSA	Preliminary Documented Safety Analyses
PE-Ci	plutonium-equivalent curie
POC	pipe overpack
PPE	personal protective equipment
QA	quality assurance
RC	root cause
RD&D	research development and demonstration
RLW	Radioactive Liquid Waste (facility)
RNS	remediated nitrate salt
RTR	real-time radiography
SCO	surface contaminated object
SME	subject matter expert
SRS	Savannah River Site
SSC	safety significant component
SSSR	sort segregate, size reduction, and repackaging
SWB	standard waste box
TA	technical area
TSR	technical safety requirement

TRU	transuranic
UNS	unremediated nitrate salt
WAC	waste acceptance criteria
WCRRF	Waste Characterization Reduction and Repackaging Facility
WCS	Waste Control Specialists, LLC
WIPP	Waste Isolation Pilot Plant

1.0 INTRODUCTION

1.1 Objective

This white paper examines the possible options related to repackaging the nitrated salt waste streams that currently exists at Los Alamos National Laboratory (LANL) for shipment to the Waste Isolation Pilot Plant (WIPP). More specifically the goal is to:

> *identify and assess the options for processing/repackaging the LANL waste drums containing reactive nitrate salts (both Remediated with Swheat and Unremediated) as well as other below grade drums that have yet to be removed for shipment to WIPP. Solutions that could accommodate drums at other locations or could be duplicated at other locations are of interest.*

The primary goal is to treat and repackage the remediated nitrate salt (RNS) drums and unremediated nitrate salt (UNS) drums that remain at LANL for WIPP acceptance. RNS drums are those that were repackaged from UNS drums with organic kitty litter (Swheat) with an intention to meet the WIPP waste acceptance criteria (WAC).

1.2 Background

The focus of this paper is on evaluating the available systems—gloveboxes and facilities—that may be used for processing and repackaging the RNS and UNS drums. Previous studies are used as guidance and a basis for selecting and evaluating candidate systems. These studies include the following:

- Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes (D.L. Clark, D.J. Funk: LA-UR-15-22393)
- Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory (Bruce Robinson)
- Results of Oxidizing Solids Testing (Energetic Materials Research and Testing Center [EMRTC] Report FR 10-13)
- Amount of Zeolite Required to Meet the Constraints Established by EMRTC (LANL-Carlsbad Office Difficult Waste Team: LA-UR-14-26860)
- Cementation study notes of surrogate nitrate salts and Swheat from Robert Wingo

The RNS drums that remain at LANL include 60 identified drums, of which 57 were repackaged with an organic kitty litter and 3 were repackaged with Waste Lock-770. The organic kitty litter, primarily a wheat-based product called Swheat Scoop, was added to the UNS during repackaging to absorb free liquids and remediate the ignitability characteristic of the nitrate salts. The resulting mixture was repackaged in daughter drums that became the RNS waste stream.

Swheat was found to increase the hazard associated with the UNS waste by creating a potential for exothermic chemical reactions[1]. After a release at WIPP from a stored LANL RNS drum containing Swheat, LANL initiated steps to isolate all remaining RNS waste drums located at LANL. The drums were overpacked in standard waste boxes (SWB) and placed in a Perma-Con®, in Dome 375, at Area G

[1] Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory (B.A. Robinson, P.A. Stevens)

located in Technical Area 54 (TA-54). The RNS drums are being stored in a temperature-controlled environment to mitigate the oxidizing behavior of the waste in the drums. LANL also designated all remaining RNS drums at LANL as “ignitable,” assigning U.S. Environmental Protection Agency (EPA) Hazardous Waste Number D001 after independent reactivity testing on surrogate samples containing Swheat and sodium nitrate salt[2]. Those drums containing free liquid have also been assigned D002 (corrosive) waste code.

The UNS drums remaining at LANL include 29 aboveground drums stored in a Perma-Con® in Dome 231 at Area G at TA-54 and approximately 79 candidate drums remaining belowground in Pit 9 and Trenches A, C and D. The 29 aboveground UNS drums were designated “ignitable” and those with identified liquid were deemed “corrosive,” as defined by EPA Hazardous Waste Numbers D001 and D002, respectively. The waste is considered ignitable because of the nitrate salt content and corrosive because of the presence of free acidic and nitrate salt–bearing liquids.

An Options Assessment Report was prepared to evaluate various treatment options for the RNS and UNS waste streams to allow removal of their hazardous characteristics and in response to a New Mexico Environment Department– (NMED-) issued Administrative Order. This assessment identified five candidate treatment options for remediation of both RNS and UNS drums at LANL. The preferred options included dry blending with zeolite and cementation as the primary unit operations for remediating the drums. This evaluation provides a review and assessment of the available process approaches and associated gloveboxes and facilities for implementing the remediation.

2.0 APPROACH

To effectively evaluate the available and potential systems that could be used for processing and repackaging RNS and UNS waste the following steps were utilized: characterizing the waste stream, evaluating treatment options, reviewing processing and repackaging systems, and assessing treatment options.

2.1 Waste Stream Characterization

Processes modify or alter feed stocks to meet product requirements. Understanding the feed stream characteristics and the product requirements ensures that the operations, process conditions, and equipment selection are based upon pertinent information. The feed stream for this study is limited to the RNS and UNS waste drums at LANL. Available information on these drums was collected and evaluated to properly characterize the feed stream that will be processed and repackaged.

2.2 Treatment Options Evaluation

The recently completed options assessment report, Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos, identifies five candidate process alternatives. The highest-ranked alternative is the blending of zeolite with RNS or UNS salts. The other four options include a cementation step:

1. Zeolite addition without cementation
2. Zeolite addition with cementation

[2] Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes (D.L. Clark, D.J. Funk: LA-UR-15-22393)

3. Dry process and cementation without zeolite
4. Wet Process and cementation without zeolite addition
5. Salt dissolution with cementation

Dry blending with zeolite and cementation were investigated as two different processing options for remediating the RNS and UNS drums, although the results are easily transferrable to the three remaining options.

2.3 Processing/Repackaging Systems

A review of the options available for remediating and repackaging the nitrated salt streams (RNS, UNS, and belowgrade) are presented. It is anticipated that the system will be used to process the nitrated salt waste streams using either blending or cementation, as described in Section 4. The repackaging systems examined include the following:

- existing on-site systems Waste Characterization Reduction and Repackaging Facility (WCRRF) glovebox and Mobile Visual Examination and Repackaging (MOVER)
- Mobile Repackaging ([MORK] a mobile, modular system at Savannah River Site [SRS])
- existing gloveboxes that would require modification
- fabrication of a new glovebox
- relocation of the WCRRF glovebox to TA-54

Processing the drums at TA-55 or at Chemistry and Metallurgy Research (CMR) was considered and investigated but excluded because the systems do not have the ability to accept waste drums as parent drums for bagging on to remove the waste stream for processing. The addition of this stream would likely require significant changes to the TA-55 and CMR safety basis as well, further reducing the attractiveness of this option.

2.4 Assessment of Treatment and System Options

Each system is evaluated against the following:

- supporting blending or cementation processing
- remediating and repackaging the various nitrated salt drums at LANL
- accommodating remediating and repackaging drums at other location
- providing capability for legacy drum repackaging operations
- addressing the complexity and risks associated with implementation

3.0 WASTE STREAM CHARACTERISTICS

The complete inventory of nitrate salt waste drums that require repackaging are as follows:

- 60 RNS drums in storage at LANL
- 114 RNS drums at Waste Control Specialists, LLC (WCS) in Texas[3]
- 29 UNS aboveground drums at LANL
- 79 UNS drums belowgrade at LANL

Presently, temperature control is used to maintain the aboveground RNS drums in a safe configuration and will be used before and during processing of the drums. Also, 114 drums at the low-level waste facility in Andrews, Texas, are managed by WCS in shallow underground storage that is effectively temperature controlled.

Developing an effective process and selecting a system to handle the process start with understanding the characteristics of the feed stream. For this effort, the initial feed streams are the RNS and UNS salt drums. Appendixes A through D highlight the RNS, UNS, belowgrade, and WCS drum information examined for this effort. Available information relating to these waste streams provides the following overview of the waste to be processed.

3.1 Waste Composition

3.1.1 UNS Waste Drums

Typically, the nitrate salt wastes were recovered from an evaporation process at TA-55 that was fed by either ion-exchange effluent or oxalate-precipitation filtrate. The salts, contaminated mostly with plutonium and americium, were packaged in bags and placed in drums. The real-time radiography (RTR) results from the aboveground drums are available and provide the composition characteristics of the waste. All the drums contain lead liners, and most contain plastic liners in which the bags or cans of salt were placed. The UNS aboveground drums are all over packed in 85-gallon drums.

Belowgrade candidate drums do not yet have RTR documentation but do have limited information-relating to the drum contents. The belowground drums appear to contain a more diverse suite of salts, leached solids, crucibles, ash, NaOH pellets, resin, hydroxide cake, etc., based upon the generator notes.

3.1.2 RNS Waste Drums

The RNS wastes were created from the UNS waste stream by mixing absorbents and/or neutralizers with the UNS wastes. The blended waste was placed in a fiberboard-insert liner that was placed inside a plastic bag in the 55-gallon drum. The salt/Swheat blend was placed directly into the fiberboard liner without any protective plastic around the waste, as was the case in the UNS drums. Debris waste was also often placed into the drum with the salt/Swheat mixture. Although the debris was typically placed atop the salt/Swheat blend, frequently the debris is intermingled rather than layered in the drum. Thirteen RNS drums are estimated to contain over 50 volume-percent debris and 23 RNS contain 20 volume-percent or more debris waste. The oxidizer and cardboard liner provide unique concerns not associated with the UNS drums. Twelve 12 RNS containers consist of 12-inch pipe overpacks (POCs).

[3] The same processing capability could treat the LA-CIN01 drums at WCS, if required.

3.2 Liquid

Free liquid can be identified utilizing RTR. All but four of the RTR videos of the RNS drums were taken between September 2013 and April 2014. The other four RTR records are of the POCs taken in 2011 and 2012. Five RNS drums (at that time) were reported to contain liquid: three contain less than 100 milliliters and two POCs contain about 2 liters located outside the containment bag in the POC.

Free liquids are found in nearly all of the UNS drums, typically in the 1- to 5-gallon range, with one drum containing 15 gallons. The liquid is either in the bags containing the salt waste or located on the bottom of the internal plastic liner.

3.3 Material at Risk

The current material at risk (MAR) limit for operations in the 231 and 375 Perma-Cons® is 18 Equivalent combustible plutonium-equivalent curies (ECPE-Ci) of material in process, with an additional 18 staged. For RNS waste, the current plutonium-equivalent curies (PE-Ci) values are assumed to be the actual ECPE-Ci since the waste is considered combustible. Based upon the current drum information:

Fifteen of the known RNS, UNS, and belowgrade drums exceed the 18 ECPE-Ci limit.

- 60 RNS Drums — 9 drums exceed 18 PE-Ci
- 29 UNS Drums — 0 drums exceed 18 ECPE-Ci
- 79 Belowgrade UNS Drums — 6 drums exceed 18 ECPE-Ci (31 exceed 18 PE-Ci)

Ten drums appear to have Hazard Category 2 levels of radionuclides.

- 60 RNS — 0 exceed Haz Cat 3 levels
- 29 UNS — 0 exceed Haz Cat 3 levels
- 79 Belowgrade UNS — 10 exceed Haz Cat 3 levels

The current Area G technical safety requirements (TSR) limit sort, segregate, size-reduction, and repackaging activities to 18 ECPE-Ci in process and 18 ECPE-Ci in container storage in the area of processing. It may be possible to utilize the entire 36 ECPE-Ci (18 for process and 18 for storage) for drum repackaging/remediation operations. If this were possible, then only one RNS drum exceeds 36 ECPE-Ci and it contains 39.1 ECPE-Ci.

All the drums contain less than 200 plutonium-239 fissile gram equivalent (FGE), and it does not appear this will be an issue for the nitrate salt drums. This is the FGE limit that any one drum can have for shipment to WIPP, but the WIPP limit includes two times the measurement uncertainty, and this information is only available for containers that have been assayed recently.

4.0 REMEDIATION PROCESS OPTIONS

The recently completed options assessment report (Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos) identifies five candidate process alternatives. The highest ranked alternative is the addition and blending of zeolite with the RNS or UNS salts. The other four options include a cementation step. The report predicts that the following number of RNS daughter drums (includes parent drums and debris drums) will be produced with each option (assuming 57 RNS drums):

- Zeolite addition without cementation 399
- Zeolite addition with cementation 798
- Dry process and cementation without zeolite 285
- Wet process and cementation without zeolite addition 342
- Salt dissolution with cementation 285

The exact number of daughter drums can better be estimated once the process option and the process operating conditions are resolved. However, this estimate provides some indication of the number of drums that are expected to be generated.

Four candidate processing approaches are presented as part of this review for the salt or salt/Swheat wastes. These approaches include two blending options and two cementation options. For each approach, a recipe is identified and a daughter drum count estimated. The daughter drum estimate is based upon the recipe and the drum information found in Appendixes A and B.

The debris stream is examined separately because it is not as homogenous, presents a different set of challenges, and will require different processing.

4.1 Impact of Process Option on Glovebox/System Selection

The process treatment option that is selected will impact how and where the processing will be carried out and present requirements for the confinement system. The dry-blending process, blending of the salt with zeolite, will be easier to process with the readily available repackaging system options. Cementation, a wet process, may require modifications or additional capability in addition to the available glovebox systems or may require a new glovebox. Cementation requires dissolution, pH adjustment, addition of cement, and agitation or blending of a heavy viscous paste.

4.2 Zeolite Blending

It is envisioned that dry blending with zeolite will require the following unit operations:

- Recovery or separation of the salt/Swheat matrix from debris waste
- Collection and absorption of free liquids in zeolite (expected to be minimal for RNS drums)
- Weighing of the salt/Swheat matrix or absorbed liquid/zeolite and zeolite components
- Blending of the salt/Swheat matrix and zeolite streams
- Processing of the debris waste—possibly washing or wiping and repackaging

These operations can be handled in typical repackaging gloveboxes. Two approaches to achieve a blended product include

1. small-scale batches that are then dumped into the daughter drum or
2. batching the appropriate ratio of zeolite and salt/Swheat into a drum and blending the entire contents.

Batch-blending operation can be achieved using a small drum blender or a conventional Hobart-type mixer, as shown in Figure 1. The mixer can be located in the glovebox at each daughter drum station. Selection of the size and type of blender will be based upon surrogate testing and size constraints of the glovebox.

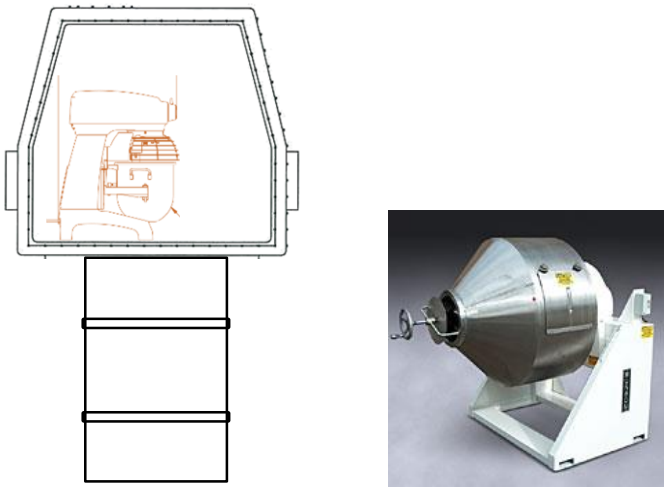

Figure 1 Glovebox end view showing Hobart blender for batch blending and picture of a drum blender

A second blending alternative uses the daughter drum to achieve blending of the salt/Swheat and zeolite. Dry blending is frequently performed in drums with lifters using a tumbling action. Bulk blending using the daughter drum after components are combined is achieved by using a drum tumbler or drum roller as those shown in Figure 2. Bulk blending using a drum tumbler or drum roller would likely require a drum insert with baffles to aid in the blending process. The optimal approach would be determined by conducting tests with surrogates. Drum tumblers are readily available and Radioactive Liquid Waste (RLW) facility uses a unit for cementation.

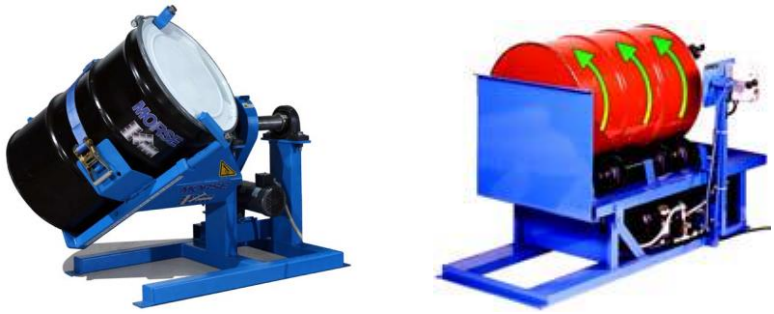

Figure 2 Drum blending options: Drum tumbler and drum roller

Blending is a dry process and may provide safety (and resulting safety basis) concerns related to sparking or electrostatic discharges (ED) during blending. The sensitivity of dry material may need to be examined to better understand these potential issues. Effective mitigation may be achieved through materials of construction and proper grounding of blending equipment.

4.2.1 Zeolite Blending Recipe

The recipe for blending RNS and UNS waste assumes a 3:1 volume ratio of zeolite-to-salt waste[1]. Free liquids (mainly in the UNS stream) are first absorbed with zeolite and then the resulting wet zeolite is blended at the same 3:1 ratio. This ratio is identified in the Options Assessment Report. For operational efficiency, a 2:1 ratio was recommended by the LANL-Carlsbad Difficult Waste Team from data provided by testing at EMRTC.[4] The more conservative 3:1 ratio is used for estimating purposes in this assessment.

Zeolite has the following characteristics[5]:

- Water absorption per pound of Bear River (BR) zeolite 0.55 lb
- Bulk density of dry BR zeolite 55 lb/ft^3
- Bulk Density of wet BR zeolite 85 lb/ft^3

Salt waste and blended salt/Swheat exhibited the following characteristics during formulation for cementation tests performed by Robert Wingo:

- Bulk density of surrogate salts for cementation 100 lb/ft^3
- Bulk density of surrogate salt/Swheat blend 57 lb/ft^3

Based upon the zeolite characteristics and the expected salt and salt/Swheat bulk densities, the following recipes are expected for blending. The number of daughter drums produced based upon these recipes and the waste stream information are also shown.

<u>RNS Drum Blending Information</u>

- Small-batch blending (fill drum 90%):
  - Salt/Swheat 12.5 gal. (96 lb)
  - Zeolite: 37.5 gal. (278 lb)
  - **132 blended daughter drums**
- Bulk blending in a drum (fill to 60%):
  - Salt/Swheat: 8.5 gal. (65 lb)
  - Zeolite: 25 gal. (185 lb)
  - **178 blended daughter drums**

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[4] Zeolite Required to Meet the Constraints Established by EMRTC (LANL-Carlsbad Office Difficult Waste Team: LA-UR-14-26860). Requirement was 1.2:1 but was rounded up to 2:1 for operational efficiency and provided additional conservatism.

[5] Specification from Bear River (BR) Zeolite, Preston, Idaho

UNS Drum Blending Information

- Small-batch blending (fill drum 90%):
  - Salt: 12.5 gal (168 lb)
  - Zeolite: 37.5 gal (278 lb)
  - **73 blended daughter drums**
- Bulk blending in a drum (fill to 60%):
  - Salt: 8.5 gal (114 lb)
  - Zeolite: 25 gal (185 lb)
  - **99 blended daughter drums**

The UNS stream has significant free liquids. It is assumed the free liquids are absorbed directly with zeolite before blending with more zeolite. Therefore, every gallon of free liquid (8.4 pounds) will require approximately 16 pounds of zeolite. The zeolite with absorbed free liquids is then further blended with 3 more equivalent volumes of zeolite. This is accounted for in the drum estimation calculation.

4.2.2 Implementing Small-Batch Blending for Nitrated Salt Waste Streams

Batch blending includes combining the salt or salt/Swheat mixture and free liquids with zeolite. Zeolite comes in a variety of particle sizes. The optimal size for blending is likely to be a 14 × 40 mesh that is equivalent to a 1.4 × 0.4-mm particle size. Most of the RNS drums do not appear to contain free liquids based on RTR evaluations, and this is likely the case for the WCS drums. Most of the UNS drums contain liquids (see Appendix B), and belowgrade drums are also likely to contain free liquids. Free liquids will first need to be absorbed with zeolite. The salt/Swheat or absorbed liquid/zeolite are then blended with zeolite in a 3 parts zeolite to 1 part salt/Swheat volumetric ratio (may be adjusted after treatability testing).

The waste drum (salt, Swheat, free liquids) is introduced into the box via the waste drum bag on port. Zeolite can be introduced via the daughter drum in bags that are removed and placed in the glovebox or via screw feeder through the side or top of the box. The parent drum is opened and any free liquid collected and mixed with zeolite to absorb the free liquid. The salt (UNS) or salt/Swheat (RNS) and the absorbed liquid on zeolite are then blended with zeolite.

Batch blending also provides flexibility for handling a range of salt/Swheat forms, from wet sloppy material to dry clumpy material. Most of the surrogate work has focused on a friable product, which is typically found when blending the nitrate salts with Swheat in a 3-to-1 volume ratio. However, it is possible that a more difficult physical form may be encountered, such as a wet “liquidy” consistency or a drier bread-like consistency. Batch processing provides the opportunity to add water or additional zeolite to get a proper consistency and mix regardless of the form obtained from the parent drum. Mixing the salt/Swheat with water before mixing it with zeolite also provides an opportunity to dilute the nitrate salt concentration found in the Swheat and allows the nitrates to report to the zeolite where it is of less concern.

A number of batch-blending systems can effectively provide the blending. The more homogeneous the feed stream, the easier it will be to blend. Large hard chunks of salt or excessively wet viscous material will be more difficult or require an approach that is less sensitive to particle size and viscosity. Initial candidate systems include a drum blender or a common Hobart blender used in the baking industry. Inserting the mixer can be achieved by bagging the blender into the box through the daughter or waste drum bag-on opening. Other blenders—drum or paddle—may be difficult to set in contaminated boxes because they are larger and heavier. The batch approach allows for verification of product quality before

drum loading. However, the process rate is slow because of the batch size, typically 3 to 5 gallons. Three gallons of a 3-to-1 volumetric zeolite-to-salt mixture weighs about 20 pounds (depending upon the moisture content). Approximately 17 batches will fill a daughter drum.

Challenges to Implementing Batch Blending in a Glovebox

- *Batch size.* Blending equipment typically used for blending moist solids and dry solids is of a size that is not easily loaded into an existing glovebox. It may be possible to get a 5-gallon unit into a glovebox through the daughter drum port, but it will be very tight and the unit weighs 190 pounds. Therefore, smaller more conventional equipment, such as a Hobart blender may be required. The result is a more time-consuming operation that requires numerous batches to fill a daughter drum.
- *Testing.* Blending equipment will need to be tested to ensure the unit will handle the variability in the waste streams; salt/Swheat (RNS) and the salt (UNS) and that potential ED and spark sensitivities are not realized.
- *As low as reasonably achievable (ALARA).* Operation is slower and requires multiple batches increasing the dose that operators receive from the waste. Previous repackaging of this waste stream resulted in short duration (less than an hour) shifts by operators from radiation exposure.

Benefits to Batch-Blending Approach

- *Process is simple.* Process requires combining preset volumes of two ingredients into a set volume blender, mixing and dumping into the drum. Multiple units could be used in the glovebox to increase throughput.
- *No modifications required to the glovebox.* Equipment can be loaded into the glovebox via the daughter drum or the parent drum for set up. Zeolite can be introduced through the daughter drum. Although it is possible to use an augur to feed zeolite through the glovebox side or top, this would require modifying the glovebox.
- *Product quality is verifiable.* The blended product can be examined visually to ensure it has been well mixed before adding to the daughter drum.
- *Maximizes drum volume utilization.* The full drum volume can be utilized because the product quality is independent of drum utilization or drum weight.

4.2.3 Implementing Drum Blending for Nitrated Salt Waste Streams

A second alternative is to use the drum as the container for blending. As shown in Figure 3, an insert is placed in the daughter drum before bagging the daughter drum to the glovebox. The insert has baffles to aid in blending. Zeolite is placed in the insert before bagging the drum on to the glovebox. Salt/Swheat is then weighed and placed in the drum once it is bagged onto the glovebox. A top is secured to the insert, and the drum is then bagged off, covered, and placed in a drum tumbler for mixing (Figure 2).

Developing an insert that will improve blending will be important to achieving a well-blended product. Surrogate testing will provide insight into the effectiveness of this approach.

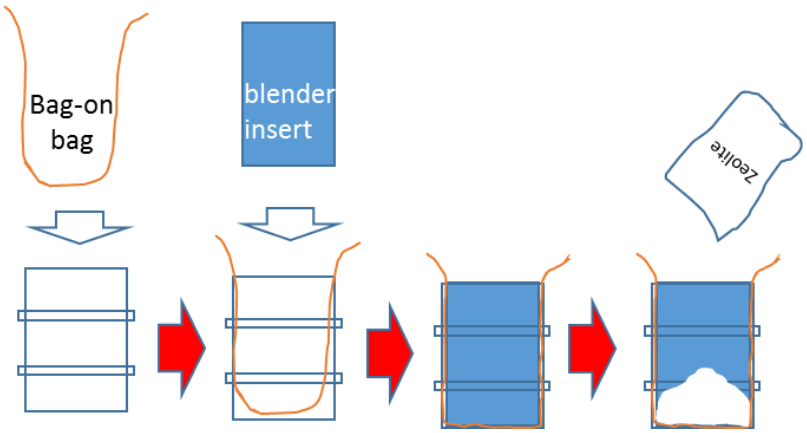

Figure 3 Preparation of a daughter drum for drum blending using a drum tumbler

Challenges to Implementing Drum Blending

- *Developing an insert to improve mixing performance.* Commercial blenders called "drum blenders" are used to blend ingredients in a number of industries. These are fixed units with a drum designed to fold and mix ingredients. The blenders have baffles and internal ribs that aid in mixing. Developing an insert that could be used with a drum roller or a drum tumbler may allow for batching ingredients into the drum and then mixing the contents after the drum is removed from the glovebox in a drum tumbler or drum roller.
- *Verification testing.* The product would be blended after the drum is closed. Testing would be required to verify the process effectively blends the zeolite and salt waste. RTR evaluations during testing may be effective at verifying blending performance and may provide a means of verification during processing.
- *Drum volume utilization.* To allow for mixing, the drum can only be partially filled, resulting in more daughter drums. For estimating purposes, a 60% fill volume was used. This resulted in an additional 46 RNS and 26 UNS daughter drums compared with filling the drum to 50 gallons.
- *Facility floor space.* The use of a drum tumbler requires availability of additional floor space. The system requires a space of about 8 feet × 10 feet.

Benefits to Implementing Drum Blending

- *Simple process.* Requires only that a preset volume of salt/Swheat be added to the drum. The drum is then removed and mixed via a drum tumbler or drum roller.
- *ALARA.* This option is very fast because it requires only the operator to measure out a volume of salt and add it to the drum, thus minimizing the amount of time dealing with the waste stream.
- *No modifications required to the glovebox.* No equipment is required inside the glovebox, making the best use of available space for handling debris and salt waste.
- *Available glovebox floor space.* Since no equipment is required inside the glovebox, the entire box is available for handling debris waste.

4.3 Cementation

Cementation of the salt or salt/Swheat waste streams can be achieved by mixing with an agitator in the glovebox (similar to TA-55 salt waste line) or drum tumbling in a containment box exterior to the glovebox (similar to RLW cementation process). In either case, the salt needs to be dissolved and pH adjusted before cementation takes place. This will likely require a tank or possibly a daughter drum for dissolution. Once the salt is dissolved, it can be pH-adjusted using a base solution such as sodium hydroxide. This operation can be thought of as a four-step process:

- Weighing of the salt/Swheat matrix or absorbed liquid/zeolite and zeolite components
- Dissolution of the salt/Swheat matrix and free liquids
- Adjusting the pH of the mixture
- Cementation of the mixture

The two cementation operations examined (TA-55 and RLW) do not include salt dissolution. The TA-55 cementation operation includes a pH adjustment followed by cementation and the RLW operation is just the cementation step.

A set of laboratory-scale cementation tests was completed by Robert Wingo using a blend of surrogate nitrate salts and Swheat. The salt/Swheat blend was pulped in water, the pH adjusted, and the blend cemented. Cementation was performed using an agitator and a roller. The cement made with the agitator was more homogeneous and stronger than the rolled product (from a qualitative perspective), although both were effective in removing the liquid. The 2-gallon monoliths are shown in Figure 4.

Figure 4 Comparison of cement made with agitation and via rolling "tumble"

One observation noted during the dissolution phase was the presence of gas formation before the pH for solutions that sat for extended periods (more than a week) was adjusted. However, no such gas

generation was observed for mixtures pH adjusted with caustic to precipitate the metals (pH of 9 to12). This was likely the result of biotic activity that was suppressed at high pH (and ionic strength).

4.3.1 TA-55 Salt Solution Cementation Process

The TA-55 nitrate salt waste line utilizes the daughter drum for mixing cement into the pH-adjusted solution. The intent is to prepare drums of monolithic concrete that have 150 to 200 ^{239}Pu FGE. The drum has a plastic insert to protect the bag-on bag from the processing operation. A total of 125 liters (33 gallons) of salt solution is mixed with approximately 25 liters (7 gallons) of 9-molar sodium hydroxide solution in the plastic insert to adjust the pH and precipitate metals. Once the solution has been pH adjusted to between 9 and 11.5, cement is metered into the drum while the solution is being agitated. The cement is stored outside the building in a large hopper and is batched into an inside hopper, which is then metered into the glovebox through the glovebox wall. A variable-speed mixer that can be raised or lowered via linear rails is used to blend the cement into the solution. The process recipe calls for approximately 300 pounds of cement. This results in a water-to-cement ratio of about 1:1 (which is very high), creating a soupy type of texture. The daughter drum rests on a scale to verify the amounts of material being added to the process. After mixing, the impeller is raised and cleaned off.

The glovebox is configured with two systems capable of preparing two drums. Figure 5 shows the cementation box at TA-55. The box has a height of about 12 feet to allow for raising and lowering the impeller shaft into the drum. The agitator is a variable speed Lightnin AJ350 and has dual impellers and 3.5-horsepower (hp) motor using 230-V three-phase power. After the cement is mixed, it is allowed to set for 2 days before it is bagged off. This set time allows for verification of the mix and time for the drum to cool.

Figure 5 Cementation glovebox operation at TA-55

The pH adjustment and the cementation process are both exothermic and generate heat. Drum heating has been noticed by the operators at TA-55, at the RLW facility and during processing cemented drums at the Dual Axis Radiologic Hydrodynamic Test Vessel Preparation Building. The TA-55 cementation process exhibits the following exothermic heating:

- Approximately 3500 kilocalories are generated during the strong acid/base reaction (pH adjustment) using 30 liters of 9-molar sodium hydroxide (270 moles of OH^-). This raises the 150-liter solution approximately 23°C.

- Type II Portland cement exhibits a heat of hydration of about 80 calories per gram typically over a 7-day period. The heat of hydration of cement will generate a concrete temperature rise of about 10°F (5.5°C) to 15°F (8°C) per 100 pounds of cement per cubic yard of concrete.[6]

The associated temperature rise with pH adjustment and hydration of Portland cement may be important considering the components in the waste may be heat sensitive, although the dissolution of the salts are likely to mitigate this sensitivity. Alternatives for cooling the drum or controlling the process rate may be considered to control temperature changes in the cemented waste. Reduced heat cements are available and should be evaluated if cementation is used[7].

4.3.2 RLW Waste Cementation Process

The RM-60 waste sludge stream at the RLW facility is cemented for final disposition. The waste stream contains precipitated hydroxides and oxides from pH adjustment related to water treatment. The waste feed is collected in a batch tank that holds 22 gallons of solution. A 55-gallon drum is loaded with 3 bags (280 pounds) of Portland cement (Type IV) and 2.5 gallons of sodium silicate, and the lid is installed and secured. The drum is then placed in a Morse Drum Tumbler, which is enclosed inside a high-efficiency particulate arresting (HEPA) filter ventilated containment box as shown in Figure 6. The 22 gallons of RM-60 sludge solution is gravity fed from the holding batch tank directly into the drum through the large bung in the drum lid. The bung is then tightened, the door to the box enclosure is closed, and the drum is tumbled for 20 minutes. After tumbling is complete, the door is opened and the large bung is removed with a rag covering the bung to relieve any pressure and open the drum to avoid pressurization during setting as the drum heats up. Finally a one-half cup of waste lock is added to absorb any free liquid that may weep out.

Figure 6 RLW drum tumbler and containment box

The recipe used for this operation is roughly 0.65 water-to-cement ratio, which is lower than the ratio used at TA-55 (1:1). The RLW recipe produces a drier, more viscous mix. It should be possible to design a

[6] Concrete Technology Today, Volume 18/Number 2, July 1997

[7] Concrete Technology Today, Volume 18/Number 2, July 1997

bung that could be vented to a pipe for HEPA filtering before the bung is removed to mitigate the “burp” associated with pressure build up during tumbling and to ensure it is handled in a controlled manner.

4.4 Implementing Cementation for Nitrated Salt Waste Streams

Two approaches are proposed for implementing cementation.

1. *Cementing inside the glovebox using a sacrificial agitator.* Salt dissolution, pH adjustment, and cementation are performed in a daughter drum, simulating the TA-55 process approach. The daughter drum requires a bag-on bag and an insert to accommodate agitation.
2. *Cementing outside the glovebox using a drum tumbler housed in a containment box.* Salt dissolution and pH adjustment are performed in a permanent daughter drum. The pH-adjusted solution is pumped from the glovebox to a drum for mixing similar to the RLW approach.

4.4.1 Cementation Recipe

Cementation tests completed by Robert Wingo[8] to evaluate the effectiveness of “grouting” RNS waste provide some guidance on a possible cementation recipe. A surrogate RNS waste was produced in the laboratory using nitrate, chloride and sulfate salts, oxalic and nitric acids, and Swheat. Figure 7 shows the surrogate mixture of salt/Swheat mixed with water.

Figure 7 Mixture of nitrate salt, Swheat, and water before cementation

The mixture was pH adjusted to 9 and mixed with type II Portland cement. The final recipe for the cemented product is as follows:

- Volumetric ratio of Swheat-to-nitrate salt mixture 3:1
- Equivalent-mass ratio of Swheat to nitrate salt mixture 1:1

[8] Notes from Cementation Tests, Robert Wingo

- Mass ratio of water to dry Swheat 3.5:1 (after pH adjustment)
- Moles of NaOH used per kg salt 3 (120 g)
- Equivalent mass water to cement ratio 0.65:1
- Ratio of cement to salt 5.2:1

The cemented product is shown in Figure 8. It was blended using a mixer, and the product was very homogenous and the Swheat well distributed throughout the matrix. For the period observed, no dewatering was observed.

Figure 8 Cut specimen of cemented surrogate nitrate salt/Swheat

A proposed recipe for cementing the RNS and UNS is shown below (Tables 1 and 2). Two changes were made to the laboratory recipe. The amount of water used per unit of Swheat was increased to ensure the resulting slurry would mix and pump. The 3.5:1 ratio of water to Swheat was increased to 4:1. Also water to cement ratio was increased from 0.6 to 0.75 to produce a lower-viscosity mixture that could be more easily mixed by agitator or drum tumbler. It is not clear if all of the water is available for wetting the cement, something further testing can clarify. The recipe used by TA-55 calls for a 1:1 ratio of water to cement. The recipe for mixing RNS waste starts with 28 gallons while the drum tumbling recipe starts with 20 gallons to keep the volume in the drum at 60% to aid in mixing during tumbling.

Table 1
Cementing Recipe for RNS Waste (60 Parent Drums)

Ingredient	Agitator (114 drums)	Tumbling (141 drums)
Water	28 gal. (235 lb)	20 gal. (168 lb)
Salt/SWheat	118 lb	84 lb
NaOH Soln*	3 gal.	2 gal.
Cement	325 lb	242 lb

Table 2
Cementing Recipe for UNS Waste (29 Parent Drums)

Ingredient	Agitator (68 drums)	Tumbling (95 drums)
Water	28 gal. (235 lb)	20 gal. (168 lb)
Salt	125 lb	88 lb
NaOH Soln*	4 gal.	4 gal.
Cement	340 lb	242 lb

*9-molar concentration.

The UNS recipe calls for more nitrate salt because the Swheat is not available to soak up the water before cementation. Based upon these recipes, the number of cemented RNS daughter drums is expected to be 114 when using a mixer and 141 when using a drum tumbler. The number of cemented UNS daughter drums from the aboveground UNS drums is expected to be 81 if a mixer is used and 95 if a drum tumbler is used.

4.4.2 Cementing Nitrated Salt Waste in Daughter Drums in the Glovebox

To cement in the glovebox, the daughter drum must be prepared for the process. The daughter drum requires a bag-on bag and an insert before bagging onto the glovebox. The insert provides a hard surface to contain the monolith and protect the bag during processing. Since most candidate gloveboxes do not have enough height to lift the agitator out of the drum, a sacrificial agitator is inserted into the drum before bag-on. The agitator will be inserted into the drive after bagging on the daughter drum and then removed after processing is complete. The steps are shown in Figure 9.

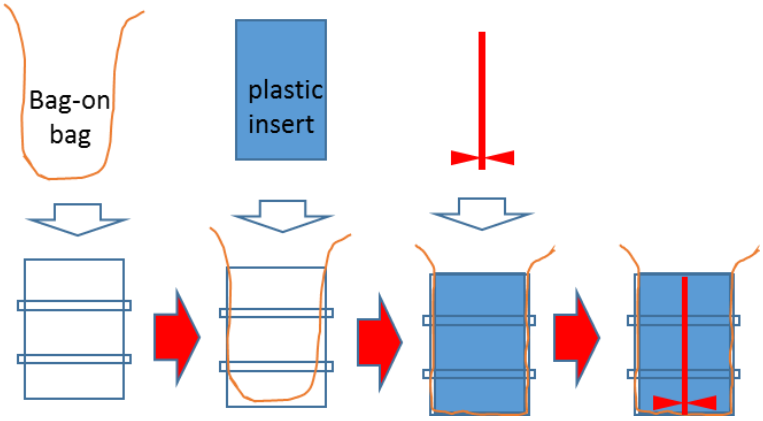

Figure 9 Drum preparation for pH adjustment and cement addition in glovebox

After the daughter drum is in place and the agitator is inserted into the drive, water is added to the drum and salt can be dissolved in water. Once the cement addition is complete, the agitator is removed and placed into the cement mix. The daughter drum is removed from the glovebox and allowed to cure. A general configuration of the equipment is shown in Figure 10.

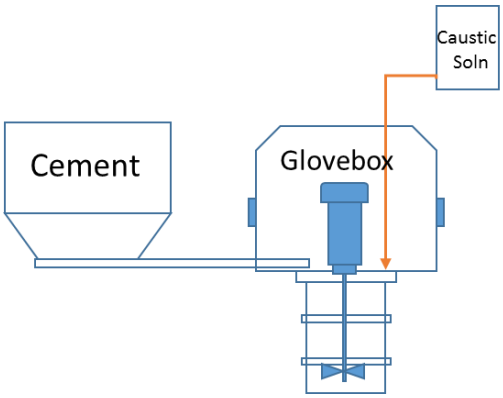

Figure 10 Glovebox cementation configuration

Challenges to Implementing Cementation via Mixing in the Glovebox

- *Glovebox modifications/design for agitator use.* The agitator at TA-55 is set in a glovebox with enough headroom to allow the mixer to raise and lower. Either (1) a box with more headroom needs to be selected or (2) the impeller/shaft may be used and sacrificed as part of the drum monolith. This would require a sacrificial impeller/shaft for each drum of waste. The agitator drive can be affixed to the box floor, the impeller and shaft introduced with the daughter drum, and the shaft installed into the drive before mixing and then removed and pushed into the monolith after mixing. Installation in a contaminated glovebox will be more challenging. A Lightnin I Series fixed mount 3-hp Type Q is similar to the system used by TA-55.

Figure 11 Lightnin Type Q drive to locate inside glovebox

An engineering review will be required to verify the unit (89 pounds) can be properly secured and the box credited-safety function is not compromised.

- *Ingredient introduction.* Introduction of caustic for pH adjustment and the addition of cement will require bringing these streams into the box through the top or side of the glovebox. This could be a challenge to retrofit for “hot” gloveboxes. It will likely include bulk transfer systems located outside the box and piped into the box for metering. Measurement of the drum weight or a predetermined volume could be used to ensure the proper recipe is achieved.
- *Throughput.* At TA-55, the daughter drum is allowed to cure for at least 2 days to ensure a proper set and no weeping of water from the mix. This process could significantly reduce the waste drum processing rate. A long cure time is not necessarily required, but bagging off the cement once mixed and before curing would require puncturing the plastic bag to allow for venting of hot gases

from the drum during curing. The addition of an absorbent above the cement may provide insurance for any weeping.

- *Box floor space.* The mixing equipment and cement delivery system will reduce available box space for handling debris waste.

Benefits to Implementing Cementation via Mixing in the Glovebox

- *Hazard mitigation.* Salt dissolution mitigates the potential of a fire from the oxidizer in the glovebox by immediately wetting the salt/Swheat mixture and dissolving the nitrate salts (although the liquid may still retain the oxidizer characteristic at that time). Wetting and cementation provide an added "comfort value" since cementation is widely accepted as a treatment process.
- *Single location.* Cementing in the glovebox eliminates the need to take the drum to another location to cement. Dissolution, pH adjustment, and cementation are done in a "one-stop" process, and the daughter drum is ready for disposal once curing is complete.
- *Verifiable product quality.* The cemented product can be examined visually to ensure it has been well mixed before the drum is removed.
- *Maximize drum volume utilization.* More drum volume can be utilized since the product quality is dependent upon mixing with an agitator and the entire drum volume can be utilized.

4.4.3 Cementing the Nitrated Salt Outside the Glovebox

To simulate the cementation process used by RLW, an exterior HEPA-filtered containment box is used to enclose the cementation operation. Dissolution and pH adjustment are carried out in the glovebox and then pumped to an exterior drum for cementation. The daughter drum becomes a permanent container used to dissolve the salts and adjust the solution pH. Dissolution can be achieved by adding water to a permanent daughter drum and dissolving the salt in the drum. A fixed mixer or a recycle pump is used for agitation during dissolution and pH adjustment. Caustic is pumped into the box and controlled via pH. Once the solution is ready for cementation, it is pumped to a holding tank or directly to the drum for cementing. The drum is located in a separate enclosure housing the drum tumbler and the drum. Figure 12 shows the configuration for mixing exterior to the glovebox.

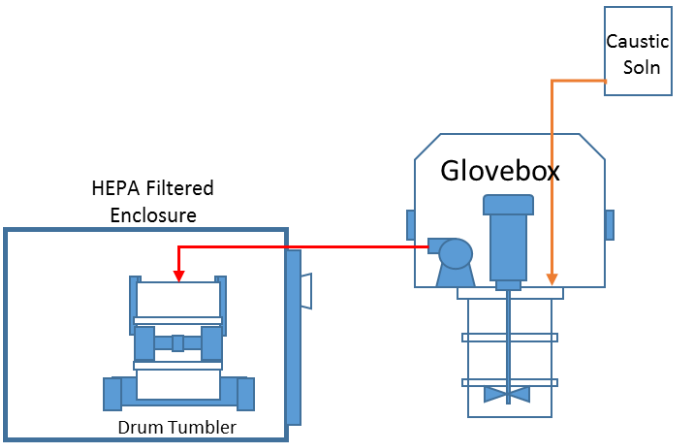

Figure 12 Cementation using a drum tumbler

Challenges to Cementing Using a Drum Tumbler

- *Verifying cementation effectiveness.* Unlike using a mixer with open access to the drum contents, it is difficult to verify the mix in the drum is adequate when using a drum tumbler. Mix quality will have to be thoroughly tested to ensure an adequate recipe and mix time are achieved. A bag or insert is unnecessary for this operation, so it is possible to preload baffling into the drum to improve mixing effectiveness. RTR evaluations during testing may be effective at verifying blending performance and may be a means of verification during processing.
- *Glovebox modifications.* Caustic addition and delivering the waste slurry to the drum will require piping through the glovebox.
- *Venting, post-cementation.* During tumbling (20 minutes), a cement-and-water mixture will likely heat the contents, including the gases, and will expand in the drum. After tumbling, the bung is removed and this is typically accompanied by a release of internal pressure as the bung is unscrewed from the lid. Using a bung with a valve should allow for a controlled release of pressure and for directing the gas to a HEPA filter for discharge.
- *Pumping.* Pumping the slurry will require a positive displacement pump that is capable of handling the repulped and pH-adjusted slurry.
- *Daughter drums.* For the drum tumbler to be effective, the daughter drum contents are kept below 60% to aid in mixing. This results in additional cemented daughter drums and in an estimated additional 27 RNS and 27 UNS daughter drums compared with mixing inside the glovebox.

Benefits to Cementing Using a Drum Tumbler

- *Hazard mitigation.* Salt dissolution mitigates the potential of a fire from the oxidizer in the glovebox by immediately wetting the salt/Swheat mixture and dissolving the nitrate salts (although the liquid may still retain the oxidizer characteristic at that time). Wetting and cementation provide an added “comfort value” since cementation is widely accepted as a treatment process.
- *Simplified glovebox operation.* The operations inside the glovebox only include dissolution of the salts and pH adjustment. Cementation is done externally so mixing and cement addition is not required inside the glovebox. This provides more room for waste handling and reduces the complexity of the processing in the glovebox.
- *Reduced bag-on operations.* The daughter drums are those that are exterior to the glovebox for cementation. Therefore, no daughter drum bag-ons required for cementation, only for debris waste.
- *Ease of cement addition.* Cement can be added to the drum before adding the salt/Swheat solution.
- *Production rate.* Reducing the operations in the glovebox will expedite processing. Daughter drums are concreted exterior to the glovebox, so bag-on operations for daughter drums are eliminated.

4.5 Processing Debris Waste

The RNS and UNS waste drums contain debris waste that is comingled with, and is assumed to be contaminated with, the salt (UNS) or the salt/Swheat (RNS). Adding organic debris waste to the daughter drums with the salt (UNS) or salt/Swheat (RNS) is not allowed (compatibility). There is no guidance

currently on a path forward for treating transuranic (TRU) D001 debris waste (if it carries the characteristic).

The generator is required to validate that the debris waste is either not TRU (D001 mixed low-level waste [MLLW] can be shipped off-site for treatment) or is not ignitable, or mitigate the characteristic, if it is ignitable. For TRU debris waste, this would require implementing a mitigation approach and verifying that the mitigation resulted in eliminating the D001 characteristic.

4.5.1 RNS Debris Waste

Each RNS drum was configured with an outer bag and a cardboard liner inside the outer bag. The salt waste was blended with Swheat using the stub-out bag and included horsetail remnants from the parent or daughter drum bag-on. The horsetail or bag was then placed in the daughter drum with the salt/Swheat.

Debris waste from the RNS drums also includes items that were placed in the drum along with the salt/Swheat. The items found in the drum typically include the following:

- plastic bag horsetails from bag-on process
- metal objects such as 30-gallon drum, lids, cans, hardware
- lead liners
- gloves
- cardboard liners
- cans
- cut-up plastic bottles (e.g., Spilfyter Kolorsafe containers)

Review of the RTR indicates the items are located throughout the salt/Swheat matrix. Typically, the lead is found on top of the last daughter drum derived from the parent.

Five containers contain free liquids. Two of these are POCs, and the liquid is found at the bottom of the pipe component. In the other three drums, the liquid is found in the creases of the plastic bags in amounts of about 10 milliliters.

The type and location of the debris waste suggests that the debris waste will be contaminated with the salt/Swheat stream. The fact that there is no free liquids, except for small amounts, indicates the contamination will be dry or moist but not overly wet.

The debris waste, once repackaged back into the parent drum, could meet low-level waste (LLW) requirements. It may be possible to clean the debris waste, place it back into the parent drum, and meet the 100 nano-curie/gram LLW limit. This would eliminate the WIPP requirement to treat debris waste. Estimates based upon drum information presented in Appendixes A and B provide insight into the maximum amount of salt/Swheat waste that could remain on the debris waste placed back in the parent drum. Table 3 values assume the radioactive contamination is well mixed among the salt/Swheat material, including that remaining on the debris. Table 3 groups the drums according to the amount of salt/Swheat that could report back into the parent drum and still meet the 100 nano-curie/gram LLW limit.

Table 3
Estimated Maximum Mass of Salt Waste Allowable in Parent Drum to Meet LLW Limit

Type of Waste	Less than 10 g	10 g–20 g	20 g–50 g	50 g–100 g	Over 100 g
RNS	1 drum	3 drums	26 drums	17 drums	13 drums
UNS	0 drums	0 drums	1 drums	2 drums	26 drums

Debris can be washed or soaked in the glovebox with water to remove as much salt or Swheat as possible. The resulting liquid can be used in the blending process and absorbed or cemented depending upon the treatment process. The washed debris is replaced into the parent drum. Finally, it would be beneficial to add 100 pounds of zeolite to the parent drum to ensure any salt solution remaining is absorbed and there are no free liquids. Zeolite has been shown to be an effective means to remove the D001 characteristic.

It will be worthwhile to test surrogate, cardboard, and plastics contaminated with nitrate salt to determine if they are a D001 waste. For this assessment, it is assumed the RNS debris waste will be washed and returned to the parent drum along with 100 pounds of zeolite to ensure any salt solution remaining is absorbed and there are no free liquids. The debris drum will then be evaluated to determine if it is LLW or TRU waste.

4.5.2 UNS Debris Waste

The UNS drums contain lead liners and plastic liners to protect the drum. The salt waste stream is packaged in plastic bags or cans and placed in the plastic liner. The liquid has leaked out of the packages (degradation of plastic over time) in many cases and is on the bottom of the drum and contained by the plastic liner. Other debris is not typically found in these drums. The amount of material that can remain in the parent drum and maintain the LLW criteria is typically more than in the RNS waste stream, as shown in Table 3.

For this assessment, it is assumed the UNS debris waste will be washed/soaked and returned to the parent drum along with 100 pounds of zeolite similar to the RNs approach. The debris drum will then be evaluated to determine if it is LLW or TRU waste.

4.6 Resource Conservation and Recovery Act Requirements

Regardless which process is selected for UNS drums, RNS drums, and debris waste, a Resource Conservations and Recovery (RCRA) permit will be required for any treatment effort. WCRRF and various units at Area G, including 231, 375 and 412, are currently permitted for storage but not for the treatment options considered in this report for RNS and UNS waste. There are three levels of RCRA permit modification: Class 1, 2, and 3. Class 1 can be obtained within 120 days of application. Class 2 allows authorization for construction as early as 60 days, and temporary authorization for activities can be provided within 120 days of application for a period of 180 days. Class 3 authorization is likely to require a year just to get a response from NMED. NMED could also choose to initiate a compliance order to remediate the nitrated wastes, which would likely be more expeditious. The current planning basis assumes that a Class 2 or 3 permit will be required to stand up nitrate salt processing.

Another option is a research development and demonstration RD&D permit. An RD&D permit may be issued by the EPA administrator (or an authorized representative) to a facility that proposes to utilize an innovative and experimental hazardous waste treatment technology or process for which permit

standards have not been promulgated [40 Code of Federal Regulations (CFR) 270.65(a)]. With RD&D permits, the responsible regulatory agency may expedite the permitting process by modifying or waiving the standard RCRA permit application and issuance procedures specified in 40 CFR Parts 124 and 270. Operation of an experimental unit under a RD&D permit is limited to 1 year, unless the permit is renewed before the end of its term. Renewal can occur up to three times, but as with the term of the original RD&D permit, each renewal period is limited to no more than 1 year [40 CFR 270.65(d)].

5.0 REMEDIATION/REPACKAGING SYSTEM OPTIONS

A review of the options available for remediating and repackaging the nitrated salt streams (RNS, UNS, and belowgrade) are presented herein. It is assumed that the system will be employed to process the nitrate salt waste streams using either blending or cementation as described in Section 4. The repackaging systems examined include the following:

- existing on-site systems: WCRRF glovebox and MOVER
- MORK (a mobile, modular system at SRS)
- existing gloveboxes that would require modification
- fabrication of a new glovebox
- relocation of the WCRRF glovebox to TA-54

Each system is evaluated against the following:

- ability to support blending or cementation processing
- remediating and repackaging the various nitrated salt drums at LANL
- accommodating remediation and repackaging drums at another location
- providing capability for legacy drum repackaging operations
- complexity to implement

5.1 Glovebox at WCRRF

Located at TA-50 Building 0069, the WCRRF glovebox was used to repackage a large number of TRU waste drums including drums containing nitrate salts. The WCRRF building is a Hazard Category 2 nuclear facility that meets Performance Category (PC) 2. (Hazard Category 2 is defined as a facility that can exceed the threshold quantities of radionuclide identified in DOE-STD-1027-92 for Category 2 Nuclear Facilities. For plutonium-239 that is 900 grams or 56 curies.) The MAR limit for WCRRF is currently 800 ECPE-Ci waste and 1800 PE-Ci total as specified under the current TSRs [ABD-WFM-006, R.2.1, Technical Safety Requirements (TSRs) for Waste Characterization Reduction and Repackaging Facility (WCRRF)]. These limits are well above the MAR inventory for the RNS-bearing waste drums and the estimated highest MAR drum in the belowground inventory that may be subject to future retrieval.

The current WCRRF Basis for Interim Operation (BIO) does not evaluate the treatment or “processing” of TRU waste drums containing oxidizers. Hence, since RNS has been designated as ignitable (D001), a change to the WCRRF BIO and TSRs will be required to allow for processing of RNS waste in the WCRRF glovebox. Additional BIO/TSR changes would also be required for using the drum tumbling option, glovebox modifications, and new processes to incorporate the proposed cooling control for RNS-bearing waste drums.

The floor plan for WCRRF is shown in Figure 13. Two airlock rooms (103 and 104) are located in front of the nuclear operations area Room 102. The glovebox is highlighted in red in Room 102. A second walk-in glovebox is shown in Room 102. This walk-in box glovebox (GBE) is currently not in use and not identified for use in the WCRRF BIO.

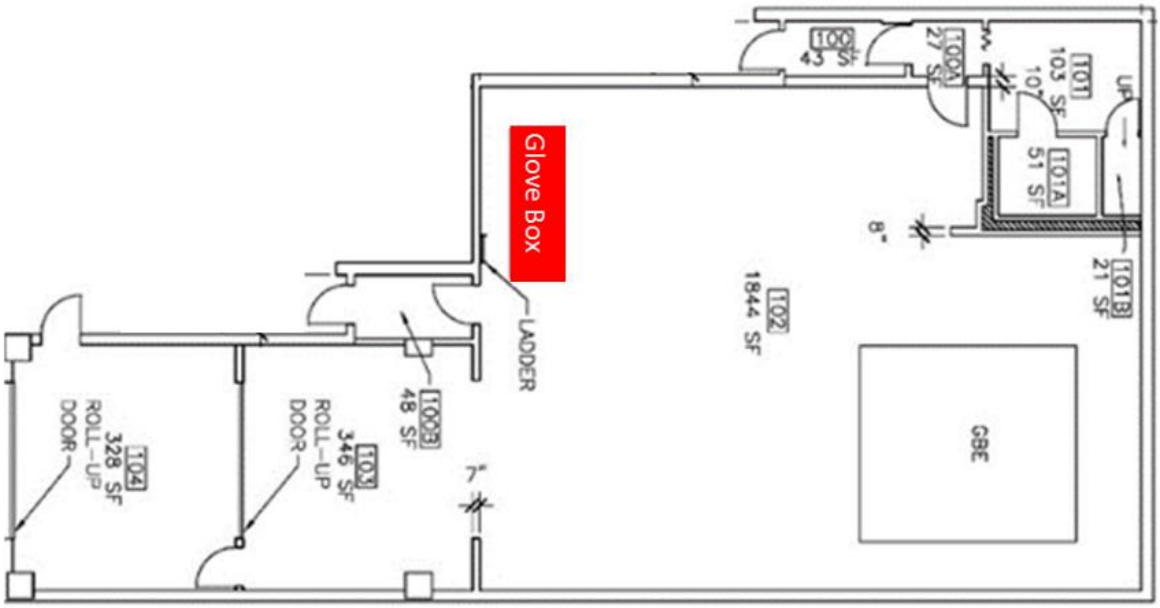

Figure 13 WCRRF floor plan

The WCRRF glovebox, shown in Figures 14 and 15, was designed in 1989 and installed in the early 1990s. It has two 55-gallon daughter drum bag-out ports, a 30-gallon daughter drum bag-out, and a single 55-gallon drum waste bag-on port. The box is 11 feet long, 3 feet wide, and 30 inches high. The box has seven work stations, three on the front side and four on the back. The waste drum is attached straight on from the front side of the glovebox and accessed from the back of the box. A liquid catch basin is located below the parent bag-on port to collect liquid from the parent drum. It is equipped with a water fire sprinkler for fire suppression. Ventilation for the glovebox is pulled in from the room and exhausted through HEPA filters on the glovebox and then through facility HEPA filters. Access to the box is tight from both the back side and the back end.

The WCRRF glovebox is a credited Safety Significant system located inside a Hazard Category 2 nuclear facility that can accommodate 800 PE-Ci of MAR. The glovebox protects the worker and the collocated worker from airborne contamination and reduces radiation exposure to the worker. The associated fire suppression system helps mitigate the potential impact of fires inside the glovebox, thereby protecting the worker. The WCRRF glovebox has been used successfully for repackaging waste for over 20 years, and the allowable MAR limit provides for processing and repackaging all expected LANL legacy waste drums in inventory above or belowgrade.

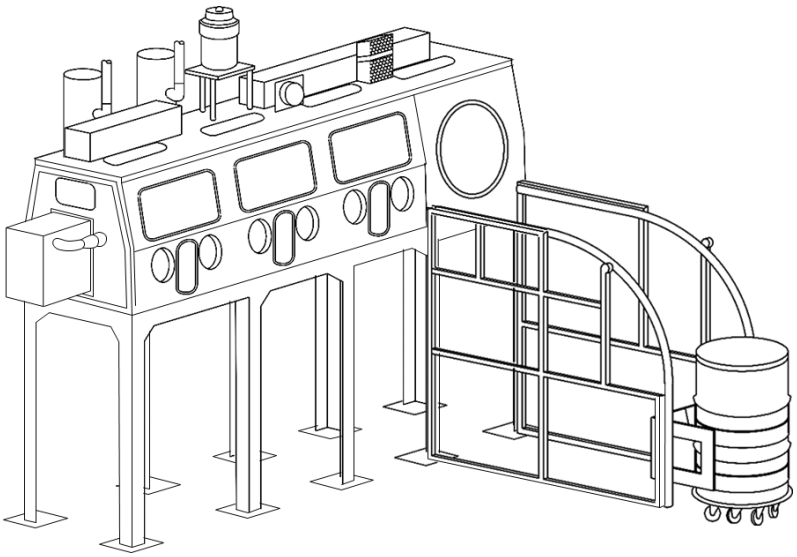

Figure 14 WCRRF glovebox isometric

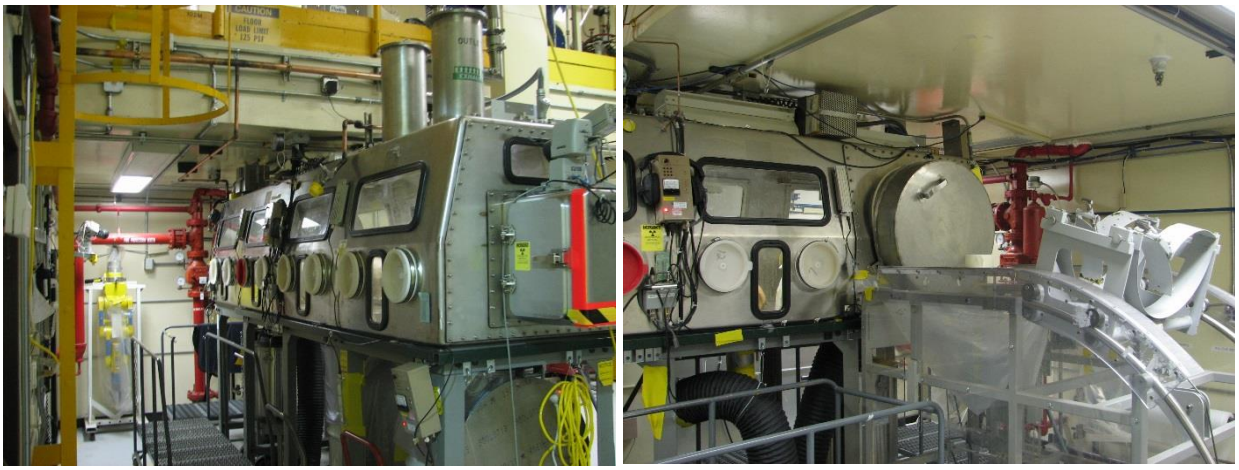

Figure 15 Photos of the WCRRF glovebox

5.1.1 WCRRF Evaluation

Positives

- The glovebox is configured, in place, and operational. No design, fabrication, or modifications are required to operate unless cement or zeolite needs to be delivered into the box.
- The WCRRF glovebox has been used effectively for 20 years on a wide variety of waste streams, including plutonium-238.
- The WCRRF MAR will accept all RNS, UNS, and belowgrade nitrate salt drums as well as any remaining legacy drums belowgrade (which are estimated to range to over 600 PE-Ci).
- The glovebox is located in a Hazard Category 2 facility, providing protection to the public for potential accidents. The facility has Safety Significant protections for the worker, allowing operations to proceed without workers continually wearing respirators.

- WCRRF has a BIO in place so most accident scenarios have already been evaluated, and appropriate TSRs are in place for safe operation. The review and update of the WCRRF BIO for processing D001 and D002 waste should be the less complicated and faster than options that include new equipment in new locations.
- WCRRF currently has National Environment Policy Act (NEPA) coverage in place.
- The configuration of the WCRRF glovebox has two salient features that assist with drum repackaging: (1) the side parent drum bag-on location and (2) the liquid collection basin. The parent bag-on port is the preferred location because it allows for direct viewing and access of the drum from the opposite side, and a work station on the end of the box also allows for accessing the lid and bolt for removal of the lid. The liquid collection basin will contain the free liquid often found in drums, making it easier to process.

Challenges

- WCRRF BIO modifications are required for processing material with D001 and D002 codes and for any needed glovebox or equipment changes.
- Use of WCRRF glovebox requires relocation of drums from Area G to WCRRF. This requires transport over road that would not be associated with a system located at TA-54.
- A refrigerator for maintaining drum cooling may be needed at or near WCRRF to ensure drums are properly cooled when processing begins to maximize operational time. Drum storage at WCRRF is described in the WCRRF BIO and managed with TSRs. This aspect of the BIO and TSRs would need to be modified for use of the WCRRF glovebox.
- Modifications to the facility and/or the glovebox for cementation at WCRRF would be required. The glovebox is contaminated and may need decontamination before any modifications are made. Either (1) a mixer and cement delivery system needs to be installed in the glovebox for cementing inside the glovebox or (2)a drum tumbler needs to be installed in the WCRRF glovebox room for mixing outside the glovebox.

Zeolite Blending

Zeolite addition can be readily accomodated within the WCRRF glovebox. Blending can be achieved using blenders loaded into place via the bag-out ports. Zeolite can be introduced into the box using the daughter drums loaded with bags of zeolite, or a zeolite addition system could be added to augur material into the box. Both daughter drum ports can be utilized for blending in either batch or drum processing.

Cementation

The WCRRF glovebox can accommodate cementation using a sacrificial agitator for in-box mixing or a containment box to house a drum tumbler. The glovebox and the facility require modifications for (1) delivering caustic solution for pH adjustment, (2) storage and delivery of cement, and (3) installation of an agitator drive. Modifications to the glovebox may be difficult because the glovebox is contaminated.

If a drum tumbler is found to be an acceptable cementation approach, then glovebox modifications are reduced and cement bulk storage and delivery to the glovebox are unnecessary.

For either cementation option, the process of designing glovebox and facility modifications and making the modifications will take time and add to the overall schedule.

Debris Waste

There is limited room to handle debris waste in the WCRRF glovebox. Removal, storage, and handling the debris during processing of the salt may be tight, with limited space to store the debris before it is placed back in the parent drum. Using water will require a means to introduce and use water for washing the debris waste.

RNS Drums Processing

All drums should be acceptable for processing once WCRRF BIO modifications are in place for operations. All RNS drums have MAR well below 800 ECPE-Ci and 1800 PE-Ci

UNS Drums Processing

All drums should be acceptable for processing once BIO modifications are in place for processing. All UNS drums have MAR well below 800 ECPE-Ci and 1800 PE-Ci. Most UNS drums have liquid and will require a means to collect, contain, and absorb the liquid. The glovebox has a collection reservoir.

Belowgrade Drums

All drums should be acceptable for processing once BIO modifications are in place for processing. All belowgrade drums have MAR well below 800 ECPE-Ci and 1800 PE-Ci.

WCS Drums Processing

No WCS drums will be allowed to be shipped off-site from the WCS facility.

Legacy Waste—Future Capability

WCRRF safety basis currently allows for the characterization and repackaging of all belowgrade drums based upon the known drum curie content. Other constituents in belowgrade drums may not be allowed at WCRRF without a Safety Basis change.

5.1.2 WCRRF Path Forward

Currently, WCRRF is in cold stand-by mode. To restart operations in the glovebox, the following steps are expected:

- Select nitrate salt process, select process-specific equipment, and develop procedures
- Identify glovebox and facility-related modifications to accommodate process option and initiate design effort
- Modify the WCRRF BIO, update the TSRs, and get approval to process a waste stream that contains an oxidizer and a corrosive in the WCRRF glovebox and make necessary facility/glovebox modifications
- Initiate RCRA permit modification or receive NMED order
- Implement needed facility or glovebox modifications

- Install a refrigerator at WCRRF for storing drums
- Implement BIO changes and conduct appropriate Readiness Assessments

5.2 MOVER

The MOVER system is a glovebox contained in a Type-A transportainer. MOVER is shown in Figure 16. The transportainer is a 40-foot-long qualified U.S. Department of Transportation (DOT) 7A Type-A container capable of highway transportation and designed to be located inside or outside, typically in a Hazard Category 2 facility. The system is meant to set and connect to existing building utilities. Generally, setup requires leveling the container, direct connecting to the building power with a simple plug, and, in some cases, connecting to the building compressed air or vacuum. MOVER has its own ventilation blower and the exhaust can be connected to a building's HEPA system but is not necessary. The module is designed to be self-sufficient and to handle risks posed by waste material fed to the glovebox. Typically, a facility safety analysis report or BIO can be structured to allow for the siting of the unit.

Figure 16 MOVER before deployment to Argonne East

MOVER was deployed to Argonne East in June 2001, during which time approximately 400 drums were processed for shipment to WIPP. The unit was operated by Central Characterization Project (CCP) of Washington Group International. Waste processing was completed in May 2003. The system was decontaminated and readied for redeployment to Lawrence Livermore National Laboratory (LLNL). Radiological work at LLNL began in April 2004.

On August 19, 2004, a radiological release occurred in the MOVER during bag-out operations. A complete listing of the root causes, contributing causes, and judgments of need (JONs) for the incident are included in Appendix E. Most of these deal with methods used to bag the parent drum onto the glovebox. A Price-Anderson Amendments Act (PAAA) Enforcements investigation of the MOVER radiological uptakes followed and resulted in proposed civil penalties to Washington TRU Solutions and LLNL. Violations included safety basis, work process, design and design basis documentation, and quality improvement violations for Washington TRU Solutions and as ALARA, radiological monitoring and

work process violations for LLNL. MOVER operations were suspended after the incident, and the system was returned to LANL where it has resided since.

The general configuration of MOVER is shown in Figure 17. The facility has three rooms: a control room, a receiving room, and an operations room with a glovebox.

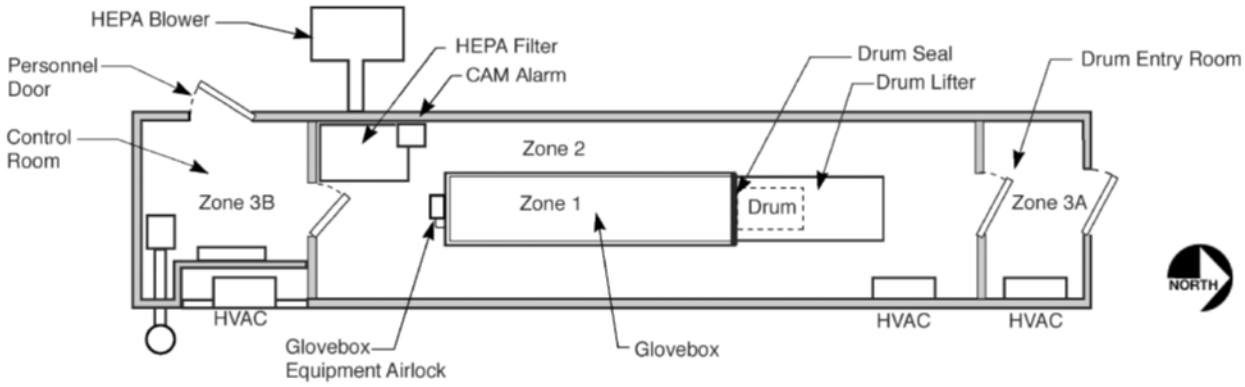

Figure 17 MOVER general configuration

Ventilation is pulled through HEPA filters in the side of the transportainer into Zone 2 (or the glovebox work area) and then into the glovebox (Zone 1) and out through a testable HEPA filter. The ventilation blower is staged outside. The control room, drum entry room, and the general work area all have an independent heating, ventilation, and air conditioning (HVAC) system that recirculate room air.

The drum entry room is located at one end of the trailer. This room provides space for four standard 55-gallon drums on transport dollies. Typically, only one drum is characterized in this process and placed in the airlock for testing each day.

Doors between each section isolate each room and are kept closed during glovebox operations to maintain negative pressure in the unit. Air-flow direction is maintained so the air flows from areas of low contamination to areas of potentially higher contamination before it is exhausted through the HEPA ventilation system.

Exterior doors are provided at each end of the trailer. A flashing light next to the exterior doors to the MOVER is lit if the continuous air monitoring (CAM) sounds. Exterior doors have a handle that can be locked when the MOVER is unattended. Radiological and warning postings signs are provided at each door entrance point in accordance with Environment, Safety, and Health protocols.

Once operators unlock the exterior doors (i.e., one at the drum entry room end and the other at the control room), the keys are removed and controlled by the operator. This precludes locking the doors while operations are ongoing.

The MOVER structure is classified as a Type II structure per National Fire Protection Association 220, Standard on Types of Building Construction. Its interior walls were constructed as double-walled for contamination purposes, with sealed and polished stainless-steel interior for ease of decontamination. The MOVER can be transported on public roads without special escort. The outside walls of the MOVER are constructed of carbon steel. The walls are insulated with cellulose, which is manufactured under Consumer Product Safety Commission performance criteria mandating fire standards. The insulation has a flame spread rate of 20 and smoke development rate of 5. Acceptable levels for a Class 1, flame

spread rate are less than 25. The interior and exterior of the MOVER are nonflammable metal with steel stud construction. All electrical systems were designed to the National Electrical Code.

The glovebox, shown in Figure 18, is of similar design to the WCRRF glovebox with two significant exceptions:

- The drum on port is on the end of the box
- The box has one 55-gallon daughter drum and one 30-gallon daughter drum bag-out port

The MOVER glovebox is 12 feet long, 2.75 feet high, and 3 feet wide at the work area.The ventilation system provides 16 airchanges per hour and maintains a flow of 25 cubic feet per minute, which provides 125-feet-per-minute face velocity if a window or glove opening is compromised. The blower is capable of twice the normal operating flow.

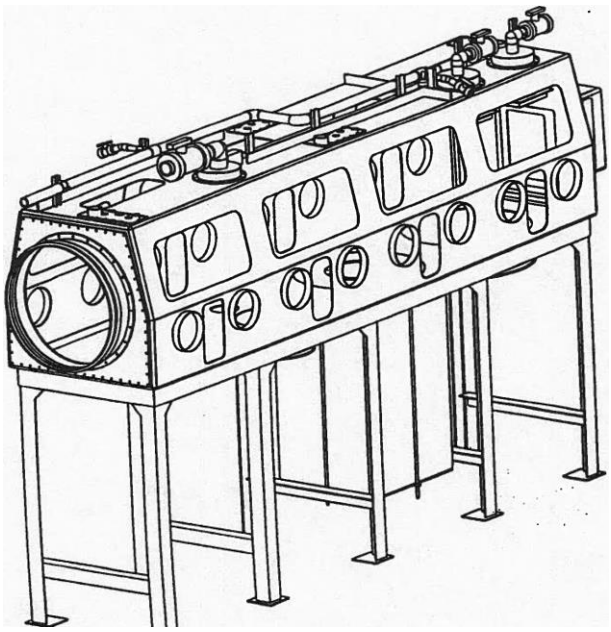

Figure 18 MOVER Glovebox

Working platforms are positioned on each side of the glovebox and are approximately 16 inches above the floor level. One step is required to access the working platforms. The platforms are hinged to the outside wall of the 7A Container and remain in the up position until used. In the down position, the platforms rest on pieces of angle iron welded to the glovebox feet (upright legs). Drums of characterized waste must be moved into and out from under the glovebox by lifting the section of the platform in the travel path of the drum.

MOVER could be located outside or inside a dome, preferably near the waste storage at the 231 or 375 dome, making access very easy and eliminating the need to transport waste for remediation.

5.2.1 MOVER Evaluation

Positives

- MOVER is very simple to set up and initiate operation, requiring only power.
- MOVER can be located at TA-54 close to the waste drums, thus eliminating the need to transport drums from TA-54 for remediation and processing.
- MOVER is mobile and can be relocated to other locations.

Challenges

- MOVER has not been used since 2004. It will require decontamination and resolution of those items identified by the PAAA investigation. Most of these relate to the parent drum bag-on approach and should be relatively easy to resolve.
- Necessary PAAA and JON issues should be resolved before restart.
- MOVER only has one 55-gallon daughter drum port.
- It is not known what MAR limit would be allowed in MOVER; however, it should be acceptable for the 18 ECPE-Ci if located inside either the 231 or 375 dome near the waste storage.
- MOVER reputation suffers from the LLNL event.
- It is unclear how much MAR MOVER would be allowed to house during operations and may limit its use on drums that contain higher amounts, especially legacy waste.
- Moving drums into and out of MOVER is slow as the elevated floor sections have to be lifted out of the way for each movement.

Zeolite Blending

Zeolite addition can be accomodated in the MOVER glovebox. Blending can be achieved using a blender loaded into place via the bag-out port. Zeolite can be introduced into the box using the daughter drum loaded with bags of zeolite. Blending the loaded drum in a drum tumbler located outside MOVER after filling would also be accomodated by MOVER.

Cementation

MOVER will complicate the cementation option as the addition of caustic and cement will need to be fed through the transportainer and into the glovebox. The box could be configured to accommodate cementation using a sacrificial agitator for in-box mixing.

As with blending the cement in a drum tumbler, the pH-adjusted solution would have to be pumped through the glovebox and the transportainer shell to an exterior drum tumbler enclosure.

If a drum tumbler is found to be an acceptable cementation approach, then glovebox modifications are reduced and cement bulk storage and delivery to the glovebox is unnecessary.

Debris Waste

MOVER has room on the back end of the box for handling debris waste. This may require manuvering around blending or mixing equipment. There is room for washing the debris. Water-supply issues will have to be resolved to accomdate washing the debris.

RNS Drums Processing

It is unclear what ECPE-Ci or PE-Ci limitations may be required for MOVER. MOVER would likely be allowed to process the RNS and UNS drums if located in the 231 or 375 dome.

UNS Drums Processing

It is unclear what ECPE-Ci or PE-Ci limitations may be required for MOVER. MOVER would likely be allowed to process the RNS and UNS drums if located at the 231 or 375 dome. Most UNS drums have liquid and will require a means to collect, contain, and absorb the liquid. The glovebox does not have a collection reservoir, and a basin will need to be added for this stream.

Belowgrade Nitrate Salt Drum

It is unclear what ECPE-Ci or PE-Ci limitations may be required for MOVER. MOVER would likely be allowed to process all the belowgrade nitrate salt drums, except the 10 that exceed Hazard Category 2 levels.

WCS Drums Processing

MOVER can be relocated and operated at other sites. It can move over the public roads as a Type A container. WCS would have to evaluate the MOVER and determine if operation is allowable.

Legacy Waste—Future Capability

MOVER may be capable of a limited role for handling low MAR waste for repackaging located at Area G. It is unclear what MAR would be allowed once the PAAA issues are addressed. It is likely the MAR would be above the 18 ECPE-Ci because the Type-A container provides a credited confinement.

5.2.2 MOVER Path Forward

Currently, the MOVER is shuttered and in need of maintenance and improvements to resolve PAAA and JON requirements. To restart operations in the glovebox, the following steps are expected:

- Select nitrate salt process and develop process-specific equipment and procedures (blending or cementing using a drum tumbler is preferred)
- Perform a readiness evaluation on the MOVER and its systems
- Prepare a list of maintenance, repair and PAAA and JON changes that will bring the MOVER in to operational readiness
- Identify a location to set MOVER for operation

- Update the Area G BIO and receive approval to process a waste stream that carries the D001 and D002 charactersitics in the MOVER glovebox to allow removal of these characterisitics (prohibited under the WIPP WAC).
- Initiate RCRA permit modification or receive NMED order
- Relocate MOVER for maintenance and upgrade work
- Initiate MOVER modifications and required TA-54 facility modifications
- Evaluate against NEPA requirements

5.3 MORK

MORK is a modular container that houses two repackaging gloveboxes.

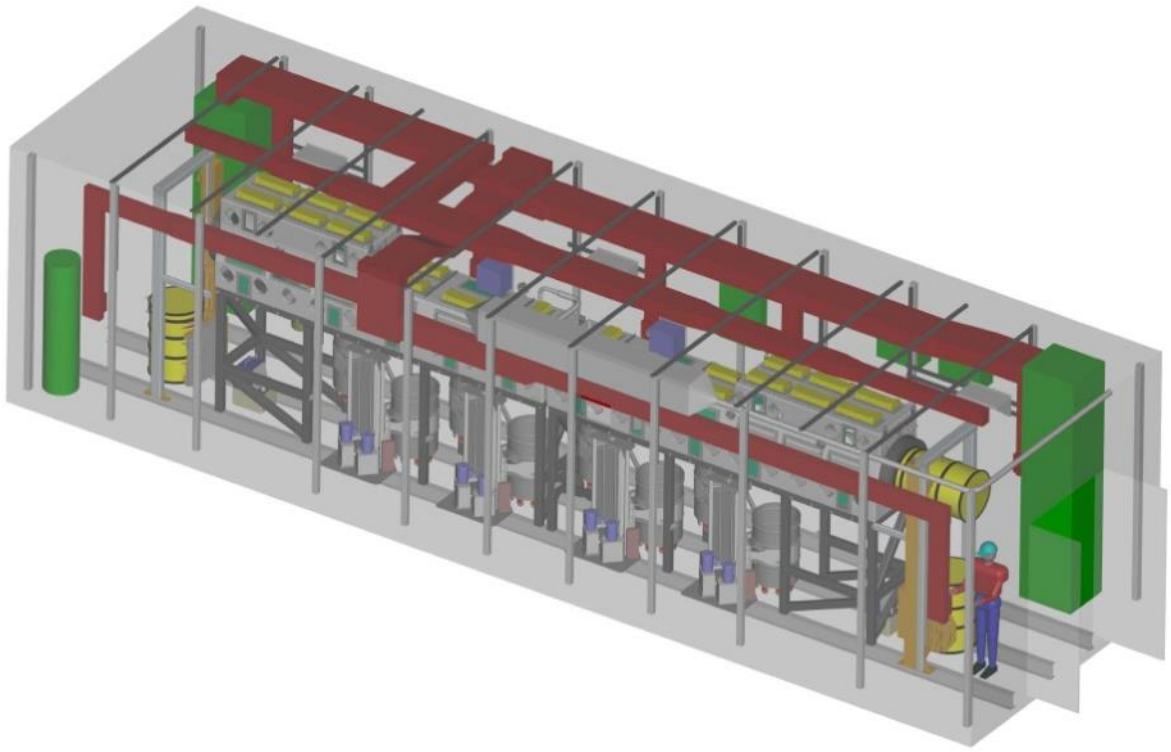

Figure 19 MORK Isometric Drawing

In 2004, at the request of Carlsbad Field Office, LANL completed the design of a set of mobile and modular nuclear facilities that could be used to process waste streams for packaging and shipment to WIPP. Two designs were completed in parallel: a Hazard Category 2 PC-2 design for deployment to SRS and a Hazard Category 2 PC-3 design for use at LANL. The SRS modular units (MUs) were built and deployed to SRS for use in repackaging drums for WIPP. The LANL design was completed, including all of the necessary documentation (construction designs, system design description, design criteria, functional and operating requirements, management level (ML) determinations, codes and standards, and preliminary detailed safety analyses (PDSA), to submit to DOE for approval. Figure 20 is a drawing of the nuclear-rated transportainer shell in which operations equipment is configured, and Figure 21 shows photos of the shell during construction.

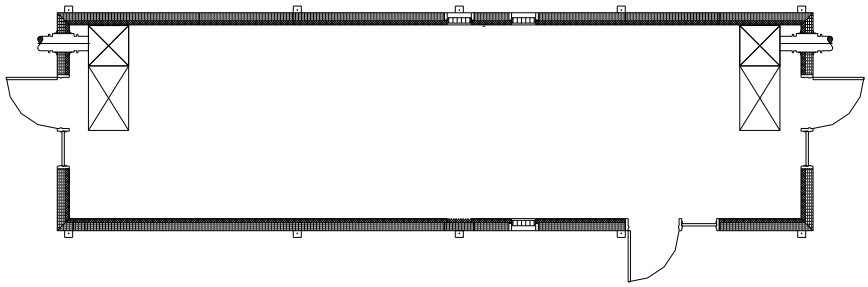

Figure 21 Nuclear transportainer shell drawing

Figure 21 Photos of the nuclear transportainer shell during construction

5.3.1 Transportainer

The mobile modular concept takes advantage of a standardized Hazard Category 2 nuclear transportainer shell that can be used to house nuclear operations and is mobile. The transportainer can be outfitted with equipment that performs functions necessary to meet mission objectives, in this case repackaging waste for shipment to WIPP. The transportainer is standardized to minimize fabrication costs and is self-sufficient. The shell is 47 feet long and 14 feet wide, capable of being moved over road, and connectable to other modules.

Transportainer internal dimensions are 12 feet wide by 11 feet high and 45 feet 6 inches long. Walls and ceilings are approximately 4 inches thick. Floors are approximately 6 inches thick. The framework is made entirely of metal. Transportainers are insulated with noncombustible insulation, and the inside (ceiling, walls, roof) is lined with 16-gauge stainless steel, which provides a radioactive material release barrier that can readily be decontaminated. All stainless-steel seams are sealed. The transportainers are equipped with fusible linked dampers to cover the HEPA filter penetrations. This provides complete isolation of the transportainer in the event of a fire. Transportainers must meet PC-3 criteria for LANL natural phenomena hazard events.

Transportainers have one exterior door opening at each end and two door openings on each side. Doors not in use can be sealed with a blind plate. All doors open to the outside and provide a tight seal. Exterior doors, panels, and frames are stainless-sheet steel and insulated with an R-value of 10. They are SDI-100, Grade III, extra heavy-duty, Model 2, minimum 16-gauge faces, insulated fire-rated B-label. Closed top and bottom edges of exterior doors are integral parts of door construction or by addition of minimum 16-gauge inverted steel channels.

Frames, concealed stiffeners, reinforcement, edge channels, louvers, and moldings are from either cold-rolled or hot-rolled stainless steel. Frames are a minimum of 16-gauge cold-rolled stainless steel. They are designed with mitered and welded corners. Door silencers are drilled to receive three silencers on strike jambs of single doorframes and two silencers on heads of frames with pairs of doors.

Penetrations through the exterior walls include ductwork, electrical conduit, and personnel doors. All transportainer penetrations, such as for ventilation and exhaust system ducts, pipes, and conduits, are sealed. Pipes, ducts, and valves are weld-type or flanged when it is not possible to weld such components.

The transportainer when equipped with process equipment becomes an MU. The MU equipped with two gloveboxes is called a MORK. Figure 22 shows the layout of a MORK with two gloveboxes for repackaging. MORK can be used to visually examine waste, retrieve prohibited items, or divide waste that exceeds radioactive limits for transportation for WIPP acceptance. MORK contains two 16-foot-long gloveboxes.

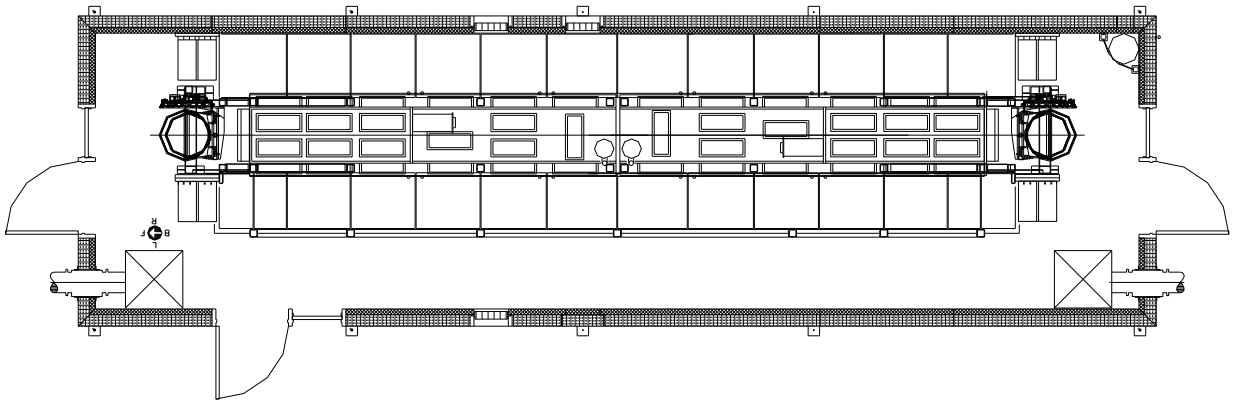

Figure 22 MORK floor plan with two gloveboxes

The MORK is self-sufficient and does not require other MUs to operate; however, it can attach to other MUs via a spool, allowing movement between MUs. The parent waste drum on ports for the gloveboxes is located at each end of the transportainer. Each end of the transportainer has a door and can be mated to a spool for access to other transportainers or to a receiving unit.

Each MU can be connected to other MUs—nuclear or nonnuclear—allowing for multiple functions, command and control, or increasing capacity. Figure 22 shows a configuration of four transportainers, two MORKs, a Command Operations Unit, and a drums storage/headspace gas unit connected via a spool as well as three receiving units also connected via a spool. This configuration is one of many possible with MUs.

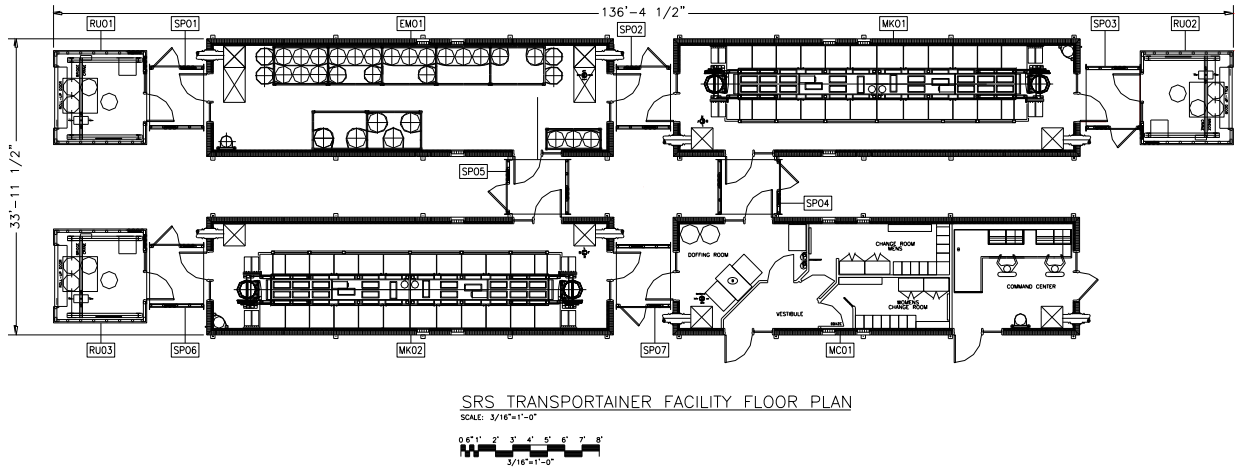

Figure 23 Mobile system facility configuration

The MAR for an MU is set at 2000 PE-Ci (32,160 plutonium-equivalent gram [PE-g]) because this limit provides a reasonable level for hot operations. Similarly, a single waste container, most often a drum, is limited to 1000 PE-Ci (16,080 PE-g). Based on operations, points of entry for the waste drums, and planned connection of the various MUs, the maximum MAR available for release is limited to the MAR of one MORK and is set at 2000 PE-Ci (32,160 PE-g).

The HVAC system for an MU is mounted on a concrete slab external to the MU. Major components of the system include supply fan, air filter, evaporator coil, hermetic compressor, condenser coil, and operating controls. The supply fan is a forward-curved centrifugal type, mounted with a V-belt drive, and an isolated high-efficiency motor. The air inlet filters are made of 2-inch-thick glass fiber disposable media in metal frames with 60% efficiency.

A concrete mounting pad is an integral safety feature included in the design of the MORK. The concrete mounting pad is identified as the key safety control to protect the off-site individual from a natural phenomenon event. A stable platform for the MORK transportainer to prevent loss of radioactive material is critical to meet PC-3. The safety function of the concrete mounting pad is to provide a stable platform that will be able to survive a seismic event. This, in turn, allows the MORK transportainer to maintain structural integrity and confinement before, during, and after a natural phenomenon event.

5.3.2 MORK Glovebox

The general dimensions for each MORK glovebox are 40 inches wide by 48 inches tall, by 16 feet long. Five workstations are on each side as shown in Figure 24. Each glovebox has the following features:

- glovebox windows made of leaded glass;
- inerting gas supplied to the glovebox interior to prevent fires;
- a tray to collect free liquids from the parent drum located under the parent drum port mouth;
- five stations on each side, one bag-on port for parent drums located on the vertical end face and designed for both 55-gallon and 85-gallon overpack waste drum, and four bag-out ports for 55-gallon daughter drums on the underside;
- a trolley to lower objects weighing up to 500 pounds into a daughter drum;

- a high-purity Germanium counter located on the underside to measure the amount of radioactive material;
- electrical connections for tools;
- internal covers for bag-out ports not in use;
- seismically designed stand;
- external covers for all ports to protect the plastic bag stub during maintenance, extended shutdowns, and transportation;
- fire detection, fire suppression (FM-200), and fire alarm systems;
- fusible link dampers on intake and exhaust of ventilation system; and
- an oxygen monitor.

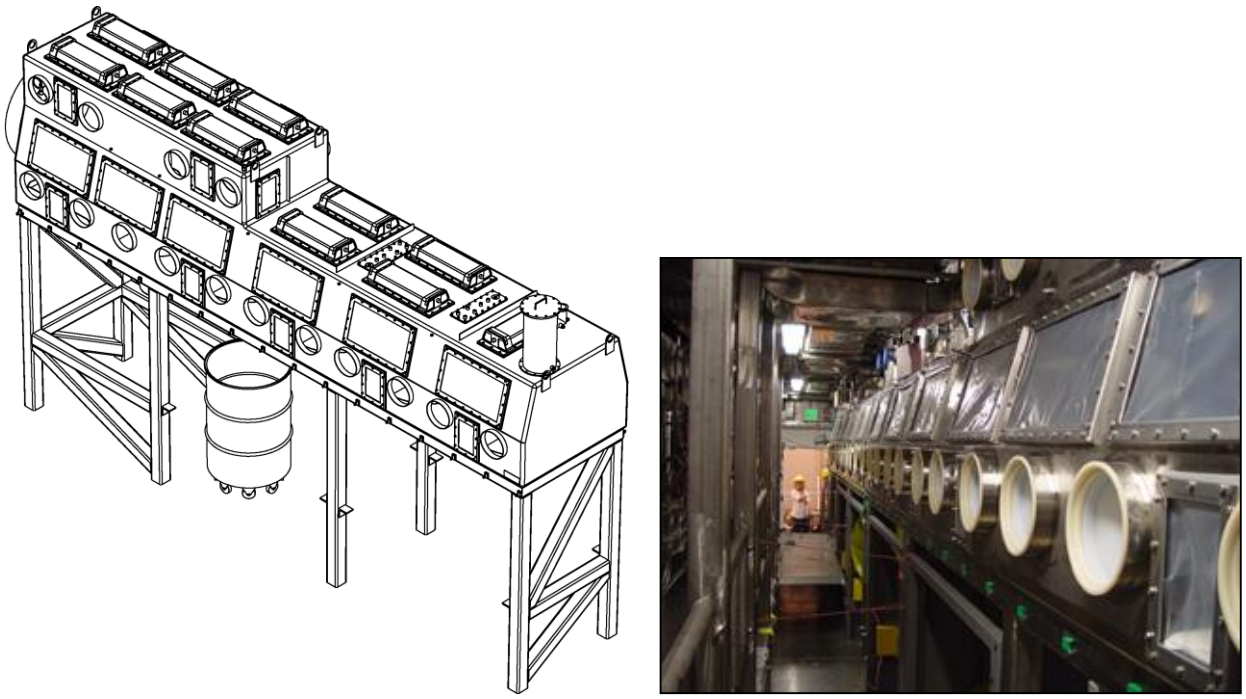

Figure 24 Isometeric drawing of MORK glovebox and picture of gloveboxes in SRS MORK

Waste containers that are 85 gallon or 55 gallon and weigh up to 1000 pounds can be bagged on to each glovebox for repackaging into four 55-gallon daughter drums. Drum lifts are provided for the waste drums and the daughter drums. The glovebox and the lifts are seismically designed and shown in Figure 25.

Figure 25 Daughter drum and waste drum lifts for MORK

5.3.3 SRS MORK

In 2004, the Carlsbad Field Office sent a set of MUs to SRS to assist with the repackaging of waste for shipment to WIPP. The MUs were fabricated in Carlsbad by Washington TRU Solutions via funding from the Carlsbad Field Office. Siting of the MUs at SRS required that the MORK meet Hazard Category 2 and PC-2 because the location was about 1 mile from the site boundary. The system configured at SRS includes a MORK, two spools, a receiving unit, and a control MU as well as the HVAC system. The system was placed inside a “RUB,” a fabric dome similar to those at Area G. The layout of this system is shown in Figure 26.

Figure 26 Configuration of modular units at SRS

The SRS system (referred to as the MRS) was installed, successfully started up, and processed approximately 750 drums. Most of the operations was repackaging of legacy waste drums. These drums contained americium-241, plutonium-239, and plutonium-238 isotopes. No major problems were encountered with the operation of the system. Drum movement was noted to be tight (which was expected and was reflected in the design). HEPA filter change out was also noted to be difficult. However, the system performed well, and the legacy waste destined for repackaging was processed and sent to WIPP.

The system was shut down in 2009 after the legacy waste was repackaged and sent to WIPP. The system was then put into “lock down.” Currently, SRS no longer uses these MUs and is contemplating salvaging them. The ventilation system has been shut down and the MORK was shuttered to allow for no ventilation. Phone discussions with Lee Fox, Deputy Director of Solid Waste Management at SRS, confirmed the site has no plans to use the MUs and is open to the possibility of relocating the system.

The glovebox contains contamination and will require decontamination before relocation to meet DOT requirements. Discussions with LANL Transportation representatives concluded that the best alternative is to transport MORK as a Surface Contaminated Object (SCO). SCO exists in two phases ACO-I and SCO-II. LANL Transportation believes the MORK would meet the SCO-II container requirements:

SCO-II: A solid object on which the limits for **SCO-I** are exceeded and on which:

(i) The non-fixed contamination on the accessible surface averaged over 300 cm^2 (or the area of the surface if less than 300 cm^2) does not exceed 400 Bq/cm^2 (24,000 disintegrations per minute [dpm]) for beta and gamma and low toxicity alpha emitters, or 40 Bq/cm^2 (2,400 dpm) for all other alpha emitters;

(ii) The fixed contamination on the accessible surface averaged over 300 cm^2 (or the area of the surface if less than 300 cm^2) does not exceed 8 × 105 Bq/cm^2 (4×10^7 dpm) for beta and gamma and low-toxicity alpha emitters, or 8 × 104 Bq/cm^2 (4×10^8 dpm) for all other alpha emitters; and

(iii) The non-fixed contamination plus the fixed contamination on the inaccessible surface averaged over 300 cm^2 (or the area of the surface if less than 300 cm^2) does not exceed 8 × 105 Bq/cm^2) (4×10^7 dpm) for beta and gamma and low-toxicity alpha emitters, or 8 × 104 Bq/cm^2 (4×10^6 dpm) for all other alpha emitters.

5.3.4 MORK Siting at LANL

To maintain the PC rating and, in turn, maximize the amount of MAR allowance, a suitable foundation and location must be available to set MORK. The concrete mounting pad provides a stable platform that will be able to withstand a PC-3 seismic event and prevent the transportainer from toppling. Based on the analysis, the transportainer concrete mounting pad and tie down hardware are designated as safety class. The concrete mounting pad cannot be placed above any of the previously used pits or underground storage locations.

Locations in Area G are limited for siting MORK because nearly all the existing buildings and most of the property sits above underground storage locations. A large area in the center of Area G is a drainage area and is not considered for siting. Dome 33 is one building that is not above a waste storage area and it is covered by an existing RCRA permit. Dome 33 currently has a mission for venting legacy drums for processing. It is unclear if the existing pad would meet seismic requirements. Another location in Area G (proposed in 2004 for siting MORK) is west of Pit 38 and it is not covered by a RCRA permit. Dome 33 and the area west of Pit 38 are outlined in red in Figure 27.

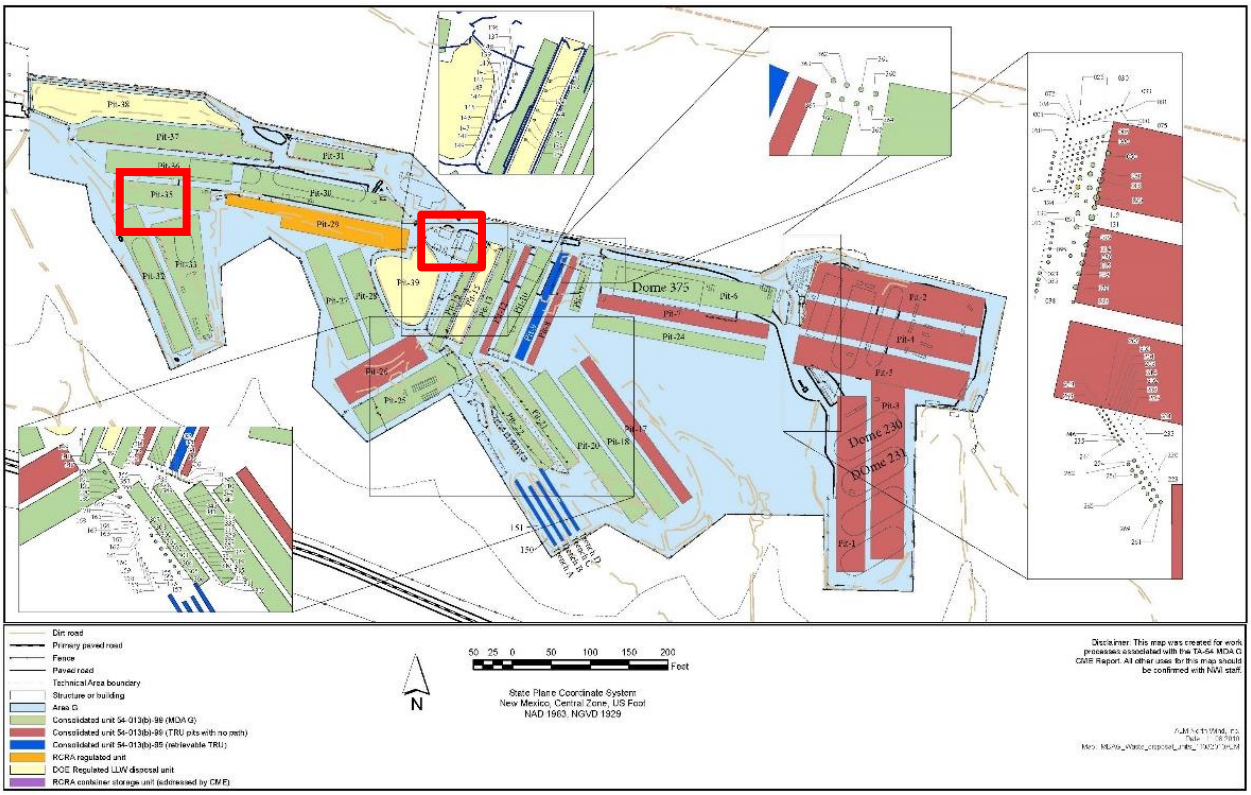

Figure 27 Area G Map showing possible MORK siting locations

Benefits are associated with locating MORK outside Area G. The R-56 pad in Zone 4 is located close to Area G and is in an area that could be configured to house MORK. Locating MORK next to Area G enables repackaging and remediation within Area G, thus minimizing operational impacts. However, this location does not benefit from having the Area G BIO or RCRA permit and would require a documented safety analysis (DSA) and RCRA Class 3 application permit to operate. This impacts cost and schedule for this option but provides a longer-term solution for legacy drums that could replace WCRRF.

Siting MORK at Area G or R-56 will require providing a seismic pad to maintain the Safety Class performance of the transportainer. Power and weather protection would be additional requirements and costs associated with siting MORK. While MORK could be located outside, it is not preferable for drum operations in the winter or during poor weather (although a dome or Perma-Con® surrounding MORK could be constructed).

5.3.5 MORK Evaluation

Positives

- MORK is designed for 1000 PE-Ci/drum and 2000 PE-Ci/MORK. This will accommodate all legacy drums, including the high PE-Ci drums containing plutonium-238 waste stored in Trenches A–D.
- MORK was and can be operated as a Hazard Category 2 nuclear facility.
- MORK operated effectively at SRS, processing approximately 750 drums including plutonium-238 waste.

- MORK has two gloveboxes each with four daughter drum ports, enabling faster processing of the waste if both boxes are used simultaneously.
- MORK can be relocated.
- Locating MORK at Area G is close to the drums, thus eliminating the need for off-site drum movement

Challenges

- MORK has not been used since 2009. Systems will need to be examined and assessed. It is likely maintenance will need to be performed to bring systems up.
- MORK will require decontamination to relocate to LANL. Deconatmination will require the support of SRS and must meet SCO-II levels. Contamination includes plutonium-238.
- Transporting MORK will require preparing the internal systems for transportation, including bracing the glovebox and ventilation ducting.
- MORK may require special siting to meet seismic requirements that may include the installation of a concrete slab.
- TA-54 BIO modification or a new DSA will be required depending upon its location for MORK to operate.
- Locating MORK at TA-54 will require utilities to run the HVAC, ventilation, lighting, and controls.
- A Class 3 RCRA permit application will likely be required for locating MORK in a location not already covered by a RCRA permit.

Zeolite Blending

Zeolite addition can be accomodated in the MORK glovebox. Blending can be achieved using a blender loaded into place via the bag-out port. Zeolite can be introduced into the box using the daughter drums loaded with bags of zeolite. Drums can also be preloaded with zeolite and tumbled after the salt is added to the drum. Use of a drum tumbler will require a location outside the MORK.

Cementation

MORK will complicate the cementation option because the addition of caustic and cement will need to be fed through the transportainer and into the glovebox. The box could be configured to accommodate cementation using a sacrificial agitator for in-box mixing.

Similarly, for blending the cement in a drum tumbler, the pH-adjusted solution would have to be pumped through the glovebox and the transportainer shell to an exterior located drum tumbler enclosure.

If a drum tumbler is found to be an acceptable cementation approach, then glovebox modifications are reduced and cement bulk storage and delivery to the glovebox is unnecessary.

RNS Drums Processing

The MORK is expected to handle all of the RNS drums since it is designed for 1000 PE-Ci/drum. Having four daughter drum bag-on ports makes the MORK versatile in providing multiple blending stations as well as providing space for debris issues.

UNS Drums Processing

The MORK is expected to handle all of the UNS drums because it is designed for 1000 PE-Ci/drum. Most UNS drums have liquid and MORK has a collection reservoir for liquids.

Belowgrade Nitrate Salt Drum

The MORK is expected to handle all of the belowgrade nitrate salt drums because it is designed for 1000 PE-Ci/drum.

WCS Drums Processing

MORK can be relocated and operated at other sites. Moving MORK requires decontamination of the glovebox to acceptable SCO levels.

Legacy Waste—Future Capability

The MORK is expected to handle all LANL legacy waste drums because it is designed for 1000 PE-Ci/drum. MORK has two gloveboxes each with four daughter drum bag-on ports. This should provide excellent remediation and processing capability.

5.3.6 MORK Path Forward

Relocation of the SRS system to LANL will require the following path forward:

- *Approval from SRS to relocate.* SRS (via Lee Fox) has indicated it does not intend to use MORK and is considering decontamination and demolition. It is amenable to supporting a LANL effort to decontaminate and relocate MORK.
- *Verification that MORK can be moved if the glovebox meets SCO-II levels.* Initial discussions with LANL Transportation representatives indicate that MORK could be transported if the glovebox is decontaminated to meet SCO-II levels
- *Verification that MORK glovebox can be decontaminated down to a level that meet SCO-II levels.* Initial indications with LANL decontamination subject matter experts (SMEs) indicate the MORK glovebox could be decontaminated to meet SCO-II requirements. A better judgment can be made once the current MORK glovebox contamination levels are analyzed.
- *Walkdown and evaluation of system at SRS by LANL SMEs.* Relocation of MORK will require a concerted effort by a range of SMEs at LANL. An initial review and visit to SRS by engineering, Facilities Operation Division (FOD) and AB SMEs are recommended to ensure the system can be relocated to LANL and deployed successfully. This review and evaluation will provide a more accurate determination of the effort, cost, and schedule required.
- Approval to use at LANL
    - *Identify a siting location.* The original design (2004) called for the MU facility to be PC-3 and located at the west end of TA-54. Two potential sites were identified: one west of Pit 38, and the other southeast of former Building TA-54-281. Based upon the waste to be processed and the MAR the facility must house, the need for seismic performance may be reduced, especially if the intent is to remediate only the RNS and UNS waste streams. A longer-term strategy for using the MORK as a Hazard Category 2 facility to

process additional legacy waste may require meeting more stringent mounting requirements and an alternate location.

- *Identify concrete mounting pad requirements.* As with the MUs, the concrete pad must meet PC-3 criteria for LANL natural phenomena hazard events. The MUs must be anchored to a concrete mounting pad. The concrete mounting pad design must have sufficient length and width to support and anchor the MUs. Based upon the waste to be processed and the MAR the facility must house, the need for seismic performance may be reduced and siting requirement related to the concrete pad relaxed. Handling the RNS and UNS and most of the belowgrade nitrate salt waste, of which only nine drums exceed 18 ECPE-Ci, may be acceptable without significant concrete pad requirements.
- *Identify the RCRA and NEPA requirements for the siting location.* Either a new permit will be required or an existing permit may need to be modified.
- *Identify AB requirements for use.* Verify LANL can obtain DOE approval to relocate and use MORK based upon siting location and intended usage plans. Identify what siting requirements would be imposed.

- *Decontaminate glovebox to levels that meet SCO-II.* Coordinate with SRS to decontaminate the MORK glovebox with SRS. Preferable option is to provide funding if necessary to SRS for decontamination services.
- Initiate AB efforts to adjust AB for using MORK
- Initiate planning to decontaminate and relocate MORK to LANL

5.4 Glovebox Located at Area G

Four gloveboxes are considered as candidates for installation at Area G. Two gloveboxes are in storage at Area G. They were obtained from sources within the LANL. Both have some level of pedigree but would likely not meet all the requirements for a credited system providing a safety significant function. Both will require some additional modifications to become operational and useful for this effort. The third glovebox is the MORK glovebox. The MORK-type glovebox would have to be fabricated. It is considered because (1) it has a complete design package, (2) the design is for use in a Hazard Category 2 facility to withstand PC-3 seismic events, and (3) the box includes four daughter drums ports. Finally, relocating the WCRRF glovebox is considered because it is a credited glovebox and has demonstrated that it can be an effective repackaging system.

Benefits are associated with having a repackaging capability at Area G. The current repackaging glovebox is located at TA-50 and requires transportation and storage of waste to and from WCRRF to repackage drums. Transportation impacts schedule, adds to cost, and increases coordination complexity. Additionally, operating a remote Hazard Category 2 facility requires additional maintenance, operations personnel, and operating cost. However, WCRRF TSRs allow for 800 ECPE-Ci and 1800 PE-Ci, providing a location to repackage any legacy drum on-site at LANL.

At a maximum, the current Area G TSRs allow for 18 ECPE-Ci of MAR in process and 18 ECPE-Ci of MAR in storage where SSSR is taking place. Nine RNS drums exceed 18 ECPE-Ci, *assuming all the contained MAR is now considered combustible*. All the aboveground UNS drums have less than 18 PE-Ci. It may be possible to receive approval to process a drum containing 36 ECPE-Ci if during sort, segregate, size reduction and repackaging (SSSR) operations no MAR is stored in the building. Only 1 RNS and 26 belowgrade drums exceed 36 PE-Ci. Therefore, it may be possible to repackage 141 of the 168 known LANL RNS, UNS, and belowgrade nitrate salt drums in a glovebox located within Area G.

A glovebox located within Area G could also be used after completing the nitrate salt waste stream for limited combustible PE-Ci drums. Approximately 3900 remaining legacy drums have less than 18 PE-Ci and are candidate drums for repackaging in a glovebox at Area G. Processing higher MAR drums will likely require systems such as WCRRF or MORK that has credited systems in place and can handle higher MAR.

Currently, three locations are considered for housing a glovebox for repackaging operations: the Perma-Cons® in Domes 375 and 231 and the enclosure at Building 412. There are competing priorities for these locations for the longer term, especially the two Perma-Cons®. This may limit the duration and long-term viability of maintaining a repackaging capability in the Perma-Cons®. Building 412 requires additional effort because the ventilation system requires attention to bring it up and to verify operation requirements; it has never been demonstrated to be nuclear capable.

5.4.1 Area G Glovebox Requirements

Fabrication or modification of available gloveboxes is an option that may be effective for the remediation of the RNS and UNS waste streams. It is unclear what level of rigor would be required for installing a glovebox at Area G. Typically, a glovebox is a credited system under safety basis to limit MAR dispersion and to protect workers.

The current Area G TSRs allow SSSR activities that are below 18 ECPE-Ci to be carried out in a glovebag at Building 412. A glovebag does not provide credited protection as a Safety Significant–rated glovebox does. The Area G BIO identifies training, fire watch, and separation distances as mitigating controls. It is unlikely that these administrative controls will be credited in future hazard analyses. Therefore, it is expected that any system employed to handle the UNS and RNS waste stream will likely require a glovebox that is Safety Significant and can act as a primary control to limit exposures to the worker. The glovebox/drum lift system

1. Provides confinement to potential airborne radioactive material, preventing release of radioactive material from the glovebox to working areas;
2. Attenuates the level of penetrating radiation to the work area;
3. Provides a stable platform for the waste container; and
4. Prevents embers from being entrained in the exhaust ventilation (fire screen) and HEPA system.

Similarly, a fire suppression system in the glovebox will also act as a Safety Significant system to control and limit the size of fire within the glovebox and mitigate the impact of the fire on the glovebox confinement (i.e. gloves).

A safety significant component (SSC) protecting the worker requires the requisite design, fabrication, installation, maintenance, and quality assurance (QA) to meet the SSC designation. The added formality and documentation ensure the glovebox can be credited to protect the worker during operation and will perform its intended preventive or mitigating function.

Schedule considerations for repackaging RNS drums may not allow for the design, fabrication, installation, and readiness activities associated with a new credited glovebox system at Area G. Discussions with Merrick suggest design may take 9 months to a year and fabrication an additional 9 months to a year. This would mean that a credited glovebox may not be available for 18 to 24 months. Alternate options are (1) qualifying an existing glovebox (with potential design/QA vulnerabilities) as a safety control for worker protection, or (2) accepting the use of an existing glovebox as defense-in-depth

but requiring additional protection for the workers as determined by the Radiation Protection Program. This would mean the glovebox was performing a radiation-control function (not Safety Class).

Therefore, for this option, the glovebox is assumed to be a non-credited system but capable of handling the RNS and UNS drums containing less than 18 ECPE-Ci. The need for a gap analyses to bring it into compliance for Safety Significant credit and the associated qualification efforts will not be considered. The glovebox will be evaluated for its ability to support remediation and repackaging efforts.

5.4.2 Glovebox 1121

Glovebox 1121 is tall box with an open floor plan and is shown in Figure 28. The box has 8 stations around the base and 24 elevated stations. The box is 8 feet long, 7 feet high, 5 feet wide at the bottom, and 4.5 feet wide above the large viewing windows. Currently, it has no glass, gloves, or base. It also does not have any parent or daughter drum bag-on adapters.

Figure 28 Glovebox 1121

The large floor plan in this box provides adequate space for handling waste, blending operations, and cementation operations. The high ceiling will allow for installing agitators for cementation that could be raised and lowered. Space exists for at least three daughter drum ports. Two daughter drum ports could be used to process salt waste and the third for debris waste. Large blenders could be installed, thereby reducing the number of batches to fill a drum. A catch basin could also be installed to collect free liquids when parent drums are opened. Figure 29 shows a possible floor plan for the glovebox with three daughter drum ports, a collection basin, and a parent drum bagged on port, shown in red in the figure.

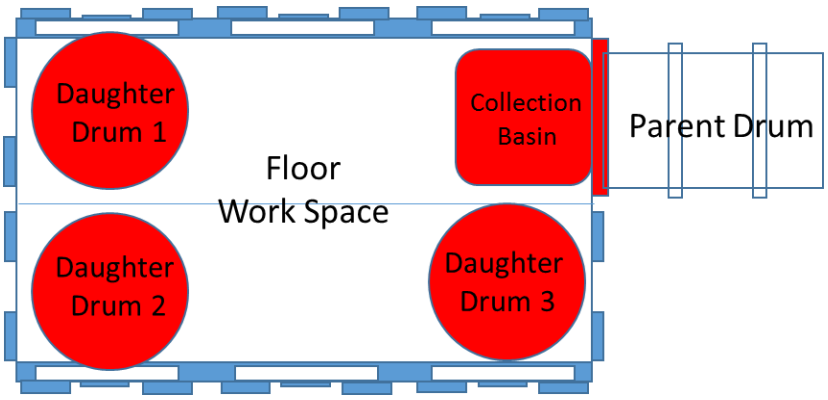

Figure 29 Floor plan option for Glovebox 1121

Significant design changes would be required to modify and qualify this glovebox for use. These would include the following:

- installing daughter drum bag-on ports
- installing parent drum bag-on port
- installing a collection basin
- covering many of the upper glove and window ports
- installing HEPA filter and ventilation ports
- installing a fire-suppression system
- modifying the base support for repackaging
- designing and fabricating a seismic stand
- providing lighting

The extensive design changes and modifications may be better achieved by designing a new box. Before initiating a redesign, it would be advisable to compare the time and cost of modifying versus starting new. The time to design and modify the glovebox for repackaging operations is estimated to be 12 to 18 months.

5.4.2.1 Glovebox 1121 Evaluation

Positives

- Large floor space is available in the glovebox to add daughter drum bag-on ports and a collection basin and still have room for dealing with debris.
- The high ceiling could accommodate mixer or more and larger blending equipment.
- A new unused and uncontaminated glovebox will be easier to set up with processing equipment.
- A glovebox at Area G is close to the drums and eliminates the need for off-site drum movements.
- Setting the glovebox in a Perma-Con® or at Building 412 provides a large working space around the glovebox, allowing for easy access to drum tumbler and cement or zeolite delivery equipment.

Challenges

- Design and modifications could take over a year to complete.
- The glovebox would not be a credited system and may not be acceptable for use at Area G.
- Operators my have to wear respirators during operations because the glovebox is not a credited system.
- The TA-54 BIO will require modification for using the glovebox for D001 and D002 operations.

Zeolite Blending

Zeolite addition can be accomodated in the glovebox. Blending can be achieved using large blenders loaded into place before commissioning. Zeolite can be introduced into the box using the daughter drums loaded with bags of zeolite. The open floor plan in the Perma-Con® would also allow for auguring zeolite into the glovebox. Easy to locate a drum tumbler near the glovebox if setting up in a Perma-Con®.

Cementation

The glovebox can be modified to support cementation either in the glovebox or outside the glovebox. Necessary provisions can be made to install mixers for mixing in the glovebox or pumps to feed a drum tumbler. The glovebox could also be configured to direct free liquids to the cementation or dissolution drum.

RNS Drums Processing

All but nine RNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. If 36 ECPE-Ci is allowed without drum storage, then all but one RNS drums could be processed in the glovebox. The larger glovebox floor space would allow for storing and handling debris waste associated with the RNS drums.

UNS Drums Processing

All UNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. Installing a collection basin would aid in collecting or diverting and treating free liquids found in the UNS drums.

Debris Waste

The 1121 Glovebox would provide for the best configuration to collect and wash the debris waste. The glovebox has available floor space to handle and store as well as wash debris removed from RNS drums.

Belowgrade Nitrate Salt Drum

Six belowgrade nitrate salt drums cannot be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G (assuming all radiological material is considered combustible). If 36 ECPE-Ci is allowed without drum storage, then four UNS drums exceed the limitation.

WCS Drums Processing

A similar glovebox (plus Perma-Con®) could be fabricated and located at WCS for RNS processing.

Legacy Waste—Future Capability

Gloveboxes located at Area G would likely be limited to the 18 ECPE-Ci limitation. There are approximately 3900 remaining legacy drums that contain less than 18 ECPE-Ci, but approximately 500 drums exceed this value.

5.4.3 Glovebox 412

Glovebox 412 is a more typical repackaging configured glovebox. It has limited space for remediation and waste handling and has only one daughter drum bag-on port. It was modified in 2014 to be used in the glovebag area for SSSR operations. The modifications were identified as “Defense-in-Depth” and included installing the daughter and parent drum bag-on ports, a support stand connected to a large steel plate, and piping for a water sprinkler system. The glovebox is intended to connect to the facility ventilation and fire protection systems and can be moved when needed.

Figure 30 Glovebox 412

A second daughter drum port could be added as well as a collection basin for free liquids. The limited work area in the glovebox would probably not allow for both. A collection basin and an area to handle debris waste are a higher priority than a second daughter drum port. The floor plan for Glovebox 412 is shown in Figure 31.

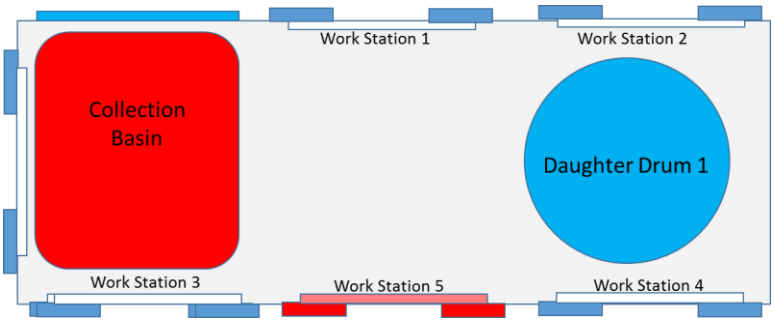

Figure 31 Glovebox 412 proposed floor plan.

Design and modifications would be required to modify and qualify this glovebox for use. These would include the following:

- installing collection basin
- installing HEPA filter and ventilation ports
- installing a fire suppression system
- providing lighting
- installing an extra work station

5.4.3.1 Glovebox 412 Evaluation

Positives

- Minimal modifications are required for this glovebox.
- A new unused and uncontaminated glovebox will be easier to set up with processing equipment.
- A glovebox at Area G is close to the drums, thus eliminating the need for off-site drum movements.
- Setting the glovebox in a Perma-Con® or at Building 412 provides a large working space around the glovebox, allowing for easy access to a drum tumbler and cement or zeolite delivery equipment.
- The parent bag-on port is the preferred location because it allows for direct viewing and access of the drum from the opposite side, and a work station on the end of the box also allows for accessing the lid and bolt for removal of the lid.

Challenges

- Limited glovebox working floor space and one daughter drum will limit throughput.
- Design and modifications could take up to a year to complete.
- The glovebox may not be a credited system and may not be acceptable for use at Area G.
- Operators my have to wear respirators during operations.
- The TA-54 BIO will require modification for using the glovebox for D001 and D002 operations.

Zeolite Blending

Zeolite addition can be accomodated in the glovebox. Blending can be achieved using a large blender loaded in place before commissioning. Zeolite can be introduced into the box using the daughter drum loaded with bags of zeolite. The open floor plan in the Perma-Con® would also allow for auguring zeolite into the glovebox. A drum tumbler could easily be locatednear the glovebox if it is set up in a Perma-Con®.

Cementation

The glovebox can be modified to support cementation either in the glovebox or outside the glovebox. Necessary provisions can be made to install mixers in the glovebox or pumps to feed a drum tumbler. The glovebox could also be configured to direct free liquids to the cementation or dissolution drum.

RNS Drums Processing

All but nine RNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. If 36 ECPE-Ci is allowed without drum storage, then all but one RNS drums could be processed in the glovebox. Limited floor space makes it difficiult to store and handle debris waste associated with the RNS drums.

UNS Drums Processing

All UNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. Installing a collection basin would aid in collecting or diverting and treating free liquids found in the UNS drums.

Debris Waste

The glovebox has limited floor space to handle, store, and wash debris removed from RNS drums.

Belowgrade Nitrate Salt Drum

Six belowgrade nitrate salt drums cannot be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. If 36 ECPE-Ci is allowed without drum storage, then four UNS drums exceed the limitation.

WCS Drums Processing

A similar glovebox could be fabricated and located at WCS for RNS processing.

Legacy Waste—Future Capability

Gloveboxes located in a Perma-Con® at Area G would likely be limited to the 18 ECPE-Ci limitation. Approximately 3900 remaining legacy drums contain less than 18 PE-Ci, but approximately 500 drums exceed this value.

5.4.4 MORK-Type Glovebox

A completed design is available for the MORK-type glovebox shown in Figure 23. This design could reduce the time to get a credited glovebox fabricated by eliminating or greatly reducing the design time. The glovebox is described in Section 5.3.2. The system provides a Safety Significant design with four daughter drum bag-on ports and can handle a 1000-pound, 85-gallon parent drum. However, the Safety Significant design may not provide for accepting additional MAR if it is located in a facility at TA-54 unless the Safety Basis is increased above the current 36 ECPE-Ci.

A review of the current design should be performed to identify any gaps that may be present from changes in requirements for gloveboxes since the MORK glovebox design was completed (2004). This gap analysis, along with possible design adjustments, would take 3 to 6 months. Fabrication time for building a MORK-type glovebox is expected to be about 1 year, assuming the Safety Significant pedigree

is required. A MORK glovebox could be operational within 2 years (including time associated with design verification, purchasing, contracting, fabrication, installation, and start-up). This system would provide a credited, robust capability for processing and repackaging.

5.4.4.1 MORK-Type Glovebox Evaluation

Positives

- The design is complete.
- It provides Safety Class credit for worker safety.
- A new, unused, and uncontaminated glovebox will be easier to set up with processing equipment.
- A glovebox at Area G is close to the drums, thus eliminating the need for off-site drum movements.
- Setting the glovebox in a Perma-Con® or at Building 412 provides a large working space around the glovebox, allowing for easy access to drum tumbler and cement or zeolite delivery equipment.
- The MORK glovebox has four daughter drum bag-on ports and a free liquid collection reservoir

Challenges

- The design review and modifications could take 2 years.
- The TA-54 BIO will require modification for using the glovebox for D001 and D002 operations.
- Locating at 231 would impact demands for that space and may limit long-term use

Zeolite Blending

Zeolite addition can be accomodated in the glovebox. Blending can be achieved using large blenders loaded into place before commissioning. Zeolite can be introduced into the box using the daughter drum loaded with bags of zeolite. The open floor plan in the Perma-Con® would also allow for auguring zeolite into the glovebox. It would be easy to locate a drum tumbler near the glovebox, if setting up in a Perma-Con®.

Cementation

The glovebox can be modified to support cementation either in the glovebox or outside the glovebox. Necessary provisions can be made to install mixers for mixing in the glovebox or pumps added to feed a drum tumbler. The glovebox could also be configured to direct free liquids to the cementation or dissolution drum.

RNS Drums Processing

All but nine RNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. If 36 ECPE-Ci is allowed without drum storage, then all but one RNS drums could be processed in the glovebox.

UNS Drums Processing

All UNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. Installing a collection basin would aid in collecting or diverting and treating free liquids found in the UNS drums.

Debris Waste

The glovebox has four daughter drum ports and space for handling debris waste.

Belowgrade Nitrate Salt Drum

Six belowgrade nitrate salt drums cannot be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. If 36 ECPE-Ci is allowed without drum storage, then four UNS drums exceed the limitation.

WCS Drums Processing

A similar glovebox could be fabricated or relocated at WCS for RNS processing.

Legacy Waste—Future Capability

Gloveboxes located in a Perma-Con® at Area G would likely be limited to the 18 ECPE-Ci limitation. Approximately 3900 remaining legacy drums contain less than 18 ECPE-Ci, but approximately 500 drums exceed this value.

5.4.5 WCRRF Glovebox

Removing and relocating the WCRRF glovebox to Area G provides a ready glovebox that is credited. The glovebox is described in Section 5.1. This is a quick way to get an operating glovebox in an Area G space. No modifications or design work is required. The glovebox could be decontaminated, secured, and relocated for installation in a Perma-Con® or Building 412. The box has been proven in operation for over 20 years and is effective for repackaging operations. Removal and relocation could be accomplished in 6 months to a year. Removing the glovebox would eliminate the capability to handle drums with more than 18 ECPE-Ci at WCRRF.

Positives

- No design, fabrication or modifications required to operate unless cement or zeolite delvery into the box is added.
- The WCRRF glovebox has been used effectively for 20 years on a wide variety of waste streams, including plutonium-238.
- The WCRRF glovebox would act as a safety significant system
- The configuration of the WCRRF glovebox has two salient features that assist with drum repackaging: (1)the side parent drum bag-on location and (2) the liquid collection basin. The parent bag-on port is the preferred location because it allows for direct viewing and access of the drum from the opposite side; a work station on the end of the box also allows for accessing the lid and bolt to remove the lid. The liquid collection basin contains the free liquid often found in drums.

- Glovebox could be located at TA-54 close to the drums, thus eliminating the need for off-site drum movement
- Setting up in a Perma-Con® provides a large working space and allows for easy access to drum tumbler and cement or zeolite delivery equipment

Challenges

- Removing the glovebox from WCRRF eliminates the only existing location to handle drums with elevated MAR.
- The glovebox must be decontaminated and removed from WCRRF.
- The glovebox is contaminated and may need decontamination before any modifications can be made. Either (1) a mixer and cement delivery system needs to be installed in the glovebox for cementing inside the glovebox or (2) a drum tumbler needs to be installed in the WCRRF glovebox room for mixing outside the glovebox.

Zeolite Blending

Zeolite addition can be accomodated in the WCRRF glovebox. Blending can be achieved using blenders loaded into place via the bag-out ports. Zeolite can be introduced into the box using the daughter drums loaded with bags of zeolite or a zeolite addition system could be added to auger material into the box. Both daughter drum ports can be utilized for blending for either batch or drum loading.

Cementation

The WCRRF glovebox can accommodate cementation using a sacrificial agitator for in-box mixing or a containment box to house a drum tumbler. The glovebox requires modifications for (1) delivering caustic solution for pH adjustment, (2) storage and delivery of cement, and (3) installation of an agitator drive. Modifications to the glovebox may be difficult since the glovebox is contaminated.

If a drum tumbler is found to be an acceptable cementation approach, then glovebox modifications are reduced and cement bulk storage and delivery to the glovebox is unnecessary.

Debris Waste

There is limited room to handle debris waste in the WCRRF glovebox. Removal, storage, and handling the debris during processing of the salt may be tight, with limited space to store the debris before placing back in the parent drum. Rinsing with water will require a means to introduce and use water for washing the debris waste.

RNS Drums Processing

All but nine RNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. If 36 ECPE-Ci is allowed without drum storage, then all but one RNS drums could be processed in the glovebox. Limited floor space makes it difficiult to store and handle debris waste associated with the RNS drums.

UNS Drums Processing

All UNS drums can be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. Most UNS drums have liquid and the WCRRF glovebox has a collection reservoir for liquids.

Belowgrade Nitrate Salt Drum

Six belowgrade nitrate salt drums cannot be handled under the existing 18 ECPE-Ci TSR limit for SSSR at Area G. If 36 ECPE-Ci is allowed without drum storage, then four UNS drums exceed the limitation.

WCS Drums Processing

The glovebox could be decontaminated and relocated (within a Perma-Con®) at WCS for RNS processing.

Legacy Waste—Future Capability

Gloveboxes located in a Perma-Con® at Area G would likely be limited to the 18 ECPE-Ci limitation. Approximately 3900 remaining legacy drums contain less than 18 ECPE-Ci but approximately 500 drums exceed this value.

6.0 OPTIONS ASSESSMENT

6.1 Nitrate Waste

1. Nine RNS drums contain more than 18 PE-Ci. The RNS waste drums have little or no free liquids, except for two POCs each containing approximately 2 liters, based on RTR evaluations. The waste is bulk loaded into a cardboard liner and debris is randomly placed in the salt/Swheat. The debris waste does not have a clear path forward if the debris is determined to be TRU and D001. The intent is to clean the debris of as much Swheat and salt as possible to meet LLW levels and/or conduct testing to demonstrate the debris is not D001. This could be difficult and time-consuming. A treatment approach may be required for this stream if it is considered D001 and contains TRU levels of contamination.

2. The UNS aboveground drums all contain less than 18 PE-Ci. Typically, the UNS drums have free liquids (up to 15 gallons), indicating the salt is very wet. Accommodations should be in place to collect the free liquids when the drums are opened and absorbing the liquid with zeolite. The salt is packaged in plastic bags but they may have broken open. There is less debris in these drums but it is likely more contaminated with salt solution. The moist texture and composition will likely provide a different remediation challenge compared with the RNS drums.

3. The UNS belowgrade drums are less well characterized. Approximately 6 exceed 18 ECPE-Ci and 31 exceed 18 PE-Ci. It is assumed they are similar to the aboveground UNS drums, although the composition and chemical makeup may be significantly different.

6.2 Nitrate Waste Process Options

1. The preferred option is drum blending with zeolite—if it can be proven to be effective and safe. Blending using a drum tumbler would be the easiest operationally and would be the fastest and have the least impact to operators from an ALARA perspective. This option can be handled by any of the available gloveboxes and would not require a complex zeolite delivery system. Zeolite could be added to the drum before bagging on to the glovebox. This approach results in the most daughter drums (178 RNS blended daughter drums and 99 UNS blended daughter drums) because the drums are only filled to 60% for purposes of optimal blending. This approach would have to be tested and proven to show that the resulting mix is well blended. The UNS stream will be the most difficult to effectively blend because it will be the wettest and least friable.

2. Batch blending zeolite is the second choice as it requires installing a system in the glovebox and extends the amount of time operators must spend processing waste. The process is simple, requiring only one operation: blending. All the gloveboxes would be capable of supporting this approach without modification. Zeolite addition could be accomplished using the daughter drum although an augur-type delivery system would be preferable. Less testing and verification are required for this option as the blended product is visually confirmed. It will produce an expected 132 RNS blended daughter drums and 73 UNS blended daughter drums.

3. Cementation using a drum tumbler is a more complex operation, requiring dissolution of the salt, pH adjustment, pumping, and drum tumbling. This method of cementation removes the cementing process from the glovebox reducing the complexity associated with delivering cement to the glovebox and would be the preferred cementation approach. Issues associated with achieving a reliable mix and incorporating the Swheat into the matrix as well as the heating associated with hydration of the cement will need to be addressed. As with drum blending, it would have to be tested and proven to show the resulting mix is well blended. The additional complexity and quality issues make this a less desirable approach. The MOVER and MORK would be the most difficult to employ this approach because of the tight quarters in the transportainer and the need to breach both the glovebox and the transportainer wall to move solution. A glovebox located in an open area like a Perma-Con® would be the best choice for this process option. This approach is expected to produce 141 RNS blended daughter drums and 95 UNS blended daughter drums.

4. Cementation in the glovebox is the most complex and not well accommodated by any of the gloveboxes, except Glovebox 1121. The limited box height, the need to deliver the cement to the glovebox, and the number of steps involved make it the least desirable option. It will require the most modifications and has the greatest chance for operational upsets that could impact operations. The approach will provide a well-mixed and remediated product and produces the least number of daughter drums, an expected 114 RNS blended daughter drums and 68 UNS blended daughter drums.

6.3 Nitrate Salt Remediation System Options

Seven system options were identified for implementing the blending or cementation processing approaches. The 7 options included using WCRRF as is, 2 modular systems (MOVER and MORK) that would be located at TA-54, and 4 glovebox options to be located either in a Perma-Con® or at Building 412. Consideration was also given to the usefulness of the system to repackage legacy waste after nitrate salt drums were completed. An initial evaluation was performed relating to 12 criteria. Each criterion was

rated from 1 to 5, with 5 being better. The rating breakdown for each criterion is included in Appendix F. The criteria are as follows.

Amenable to installing process equipment and operating:

- **Blending:** Complexity and difficulty to configure for blending operations and performing blending for RNS and UNS waste drums.
- **Cementing:** Complexity and difficulty to configure for cementation operations and performing cementation for RNS and UNS waste drums
- **Debris Waste:** Complexity and difficulty to configure for debris waste cleanup and performing debris operations for RNS and UNS waste drums

Ability to handle waste streams:

- **RNS and UNS Drums**: Percentage of RNS and UNS drums the option will be able to process because of physical and AB-related limitations
- **Legacy Drums:** Percentage of legacy drums the option will be able to process because of physical and AB-related limitations

Permitting Issues:

- **AB Issues:** Number and complexity of issues and adjustments required for preparing and getting AB approval.
- **RCRA Issues:** Number and complexity of issues and adjustments required for RCRA permitting approval.

Physical Adaptation Requirements:

- **Fabrication:** Modifications to the system before installing or using for repackaging.
- **Installation:** Complexity related to installing the system for operation.

Project Impacts

- **Schedule:** Impact to schedule related to the options requirements to become operational
- **Regulatory/Public Acceptance:** Effectiveness and reliability of the approach/system

Table 4 provides the ratings for each of the system options discussed. The best option is to utilize WCRRF. The next two best options are installation of a glovebox in a Perma-Con® at Area G using one of the available gloveboxes. These two options are rated higher, based on the assumption that the gloveboxes would only be used as a non-credited glovebox and could not provide worker protection as a Safety Significant system. If the box were to be credited, nine drums exceed 18 EC-PE, and the ratings would be lower-impacting fabrication, installation, schedule, and cost.

Table 4 System Options Evaluation Table

	Blending	Cementation	Debris Waste	RNS & UNS Drums	Legacy Drums	AB Issues	RCRA Issues	Installation	Fabrication	Regulatory/Public Acceptance	Operation	Schedule	**Total**
WCRRF	4	3	4	5	5	4	3	4	5	3	3	4	47
MOVER	3	1	2	5	4	4	1	4	4	2	3	3	36
MORK	3	2	5	5	5	2	1	2	3	4	4	1	37
GB 1121	5	5	5	3	3	3	2	3	2	2	4	3	40
GB 412	4	4	2	3	3	3	2	3	4	2	4	4	38
MORK GB	4	4	4	3	3	4	2	3	1	3	5	2	38
WCRRF GB	4	3	4	3	3	4	1	2	3	2	4	4	37

1. The WCRRF glovebox provides the least risk, adequate flexibility, and (likely) the fastest path to remediating the nitrate salt drums. The glovebox is configured to accommodate blending with zeolite but is less amenable to supporting cementation, especially cementing within the glovebox. The WCRRF BIO is already in place, and updating to allow for nitrate salt processing should not be overly onerous because similar operations have been performed at WCRRF, although not with the same hazards. The infrastructure is in place and has been well tested for the last 20 years. Transporting the waste and potentially refrigerating it at WCRRF are negative aspects of this option because they introduce additional cost, safety concerns, and coordination difficulties. However, the reliability of an available proven and operating glovebox approved for 800 ECPE-Ci, compared with modifying or relocating systems that have no operating record and no current AB, makes WCRRF the best choice for the short term to handle the nitrate waste streams. The schedule required to bring WCRRF online for nitrate salt processing is better understood, more predictable, and less likely to be adversely impacted compared with other options.

   WCRRF can be utilized until a new system is configured, tested, and approved for use in TA-54. This ensures a repackaging capability is always available and mitigates being held hostage to schedule problems that may be associated with initiating a new system. MORK, the only other system that can handle more than 18 ECPE-Ci, has some hurdles that must be overcome, including decontamination, transportation, and siting to meet seismic requirements. These risks, along with the time it may take to relocate, set up, and obtain permission to operate, are significant to both success as well as schedule. Maintaining the WCRRF glovebox operation ensures a viable capability until an alternate system can be approved, installed, tested, and brought online.

2. Installing a glovebox at Area G for supporting nitrate waste repackaging and for supporting remaining LANL legacy waste could provide added flexibility and be a relatively inexpensive option to augment repackaging. Two issues need to be resolved for this option: (1) the necessity to provide a Safety Significant glovebox for worker protection and (2) the allowable ECPE-Ci for any drum in process. These are both AB issues that should be resolved before moving forward on this option. They will impact the choice of glovebox as well as design and

fabrication/modification requirements. The flexibility to configure a new glovebox for drum repackaging and locate it in an open floor plan like a Perma-Con® room is an attractive option for a large subset (~3900 drums) of legacy waste. The existing Perma-Cons® are used for other waste-processing operations that would be impacted if a glovebox is housed in an existing Perma-Con®.

6.3.1 Legacy Waste Repackaging System Options at TA-54

Two options for drums containing more than 18 ECPE-Ci are WCRRF glovebox and MORK. An estimated 470 legacy drums at Area G contain more than 18 PE-Ci. MORK provides a tested and robust capability for repackaging and remediation. Locating the SRS MORK at TA-54 would be a long-term solution for handling the remaining legacy waste at LANL. Relocating the SRS MORK requires resolving a number of issues, including

- Verifying MORK can transport SCO-II material over public roads,
- Decontaminating the glovebox to meet SCO-II levels,
- Transporting MORK to LANL without compromising the systems,
- Siting MORK to meet seismic requirements, and
- Repairing and reactivating MORK.

If these issues cannot be resolved, then LANL could have a MORK built for deployment at TA-54. The design for fabricating a new MORK exists and could be used, and the AB documentation exists. Until an alternate system that can handle MAR in excess of 18 ECPE-Ci is up and running, WCRRF should be maintained and operational.

6.4 Recommendations

1. *Initiate nitrate salt waste stream process development.* Begin testing equipment for blending zeolite with nitrate salt waste streams. Building 39 at Area L can be set up to begin equipment evaluation and testing using surrogate waste. Purchase a drum tumbler, drum roller, and batch blenders for evaluation and process development.

2. *Decide on a system for carrying out nitrate salt repackaging.* Identify which glovebox system will be utilized for nitrate salt repackaging to tailor the process, equipment, and operating procedures to the system. Focus AB efforts for addressing the processing of D001 waste and identify any restrictions or operating requirements that may impact processing operations.

3. *Determine if debris waste associated with nitrate waste steams is D001.* Initiate tests to determine if debris waste separated from the RNS or UNS drums is D001. TRU debris waste that is D001 cannot be sent to WIPP unless the D001 characteristic is removed. Use the results from surrogate testing to drive the handling and processing of this waste stream after separating from the salt waste.

4. *Resolve AB, RCRA, and NEPA issues associated with repackaging nitrate salt drums and for developing new capability at Area G.* Four AB issues need clarification:

    - Area G BIO issues for safeing and denesting the waste from the overpacked SWBs

- WCRRF TSRs that may be required for remediating and repackaging RNS and UNS waste considered D001 and D002
- Necessity of a credited Safety Significant glovebox for waste repackaging at Area G
- Determination of AB requirements to relocate MORK: if a PDSA is required or a major modification can be used for relocating MORK to Area G

4. *Visit the SRS site to obtain more information on MORK.* Relocation of MORK will require a concerted effort by a range of SMEs at LANL. An initial review and visit to SRS by engineering, operations, FOD, and AB SMEs is suggested to ensure the system can be relocated to LANL and deployed successfully. This review and evaluation will provide a more accurate determination of the effort, cost, and schedule required.

APPENDIX A REMEDIATED NITRATE SALT DRUMS

Remediated Nitrate Salt Drums														
PKG_ID	ParentID	Waste Stream	Container Type	Gross Wt (lbs)	PECi	EC PECi	FGE	TOTAL DOSE	Drum Utiilzation (vol%)	Debris (vol%)	Hogeneous Solids (vol%)	Lead/Metal (kg)	Non-Metal (kg)	Salt/Swheat Matrix (kg)
68408	S842463	MHD01	55-gallon	205	0.4	3.7	18.9	10.5	50	60	40	25.0	6.0	29.1
68430	S833846	MIN02	POC	383	11.2	9.8	33.1	1.5	70	5	95	0.0	2.0	26.9
68507	S853279	MIN02	POC	353	4.8	4.2	36.6	0.9	50	5	95	0.0	2.0	13.4
68540	S842181	MHD01	55-gallon	85	0.3	0.3	0.4	13.5	20	85	15	0.1	4.0	1.0
68553	S842181	MHD01	55-gallon	118	1.9	1.6	2.8	33.5	45	50	50	1.2	6.0	13.8
68567	S816837	MHD01	55-gallon	202	1.3	1.1	4.6	7.5	40	65	35	31.6	7.0	20.0
68624	S824184	MIN02	55-gallon	152	0.9	0.8	10.6	38.5	40	25	75	0.0	7.0	28.6
68631	S825810	MIN02	55-gallon	214	1.5	1.3	2.3	70.5	45	15	85	20.0	3.0	40.6
68638	S825810	MHD01	55-gallon	85	0.4	0.4	0.5	14.5	25	90	10	0.0	4.0	1.1
68648	S855139	MIN02	55-gallon	269	16.7	14.7	62.4	28	55	15	85	23.0	5.5	60.1
68665	S853492	MIN02	55-gallon	372	10.1	8.9	47.1	17.5	85	15	85	13.0	7.0	115.6
68685	S855793	MIN02	55-gallon	391	8.5	7.5	21.9	26.7	85	20	80	25.2	6.0	112.9
69013	S870213	MIN02	POC	349	0.7	0.6	3.6	1.5	50	5	95	0.0	2.0	11.4
69015	S851418	MIN02	POC	358	3.0	2.6	9.3	1.5	60	5	95	0.5	4.0	12.9
69036	S873554	MHD01	55-gallon	175	10.3	9.1	22.0	31.3	50	80	20	33.4	4.2	8.5
69076	S852530	MIN02	55-gallon	384	10.0	8.8	56.0	60.8	100	5	95	5.0	6.0	130.1
69079	S901114	MIN02	55-gallon	402	21.1	18.6	58.3	70.5	75	10	90	7.8	6.1	134.7
69183	S870478	MIN02	55-gallon	427	15.1	13.3	46.8	50.5	100	10	90	3.0	12.0	145.1
69208	S851772	MIN02	55-gallon	507	39.1	34.4	157.4	82	95	10	90	10.0	2.0	184.1
69280	S841251	MIN02	55-gallon	236	18.1	15.9	69.3	22.8	40	15	85	6.0	4.0	64.6
69298	S841251	MIN02	55-gallon	448	23.2	20.4	122.4	46.5	95	10	90	5.0	4.1	160.5
69361	S892963	MIN02	55-gallon	170	4.9	4.3	17.3	30.5	35	5	95	0.1	2.0	41.5
69445	S823229	MIN02	55-gallon	366	5.1	4.5	57.6	31	75	15	85	15.0	6.0	110.6
69490	S892963	MIN02	55-gallon	371	18.9	16.6	76.0	60.5	95	5	95	3.0	8.4	123.2
69491	S891387	MIN02	55-gallon	330	12.1	10.6	14.4	60.5	85	5	95	5.1	6.0	105.0
69519	S816768	MIN02	POC	416	17.2	15.0	18.7	2.1	75	5	95	7.0	0.0	36.9
69520	S813471	MIN02	POC	344	5.1	4.4	2.1	2	65	40	60	0.0	2.0	9.4
69548	S851416	MIN02	55-gallon	207	0.9	0.8	2.4	7.5	45	10	90	23.0	2.0	35.6
69553	S841627	MIN02	55-gallon	227	12.5	10.8	61.7	29	50	10	90	5.0	2.0	62.6
69559	S832148	MIN02	55-gallon	367	14.0	12.3	22.4	60.5	70	5	95	30.0	2.0	100.6
69568	S825664	MIN02	55-gallon	248	4.5	1.1	5.2	10.5	95	60	40	0.1	6.0	33.0
69595	69090	MIN02	55-gallon	189	8.8	7.7	57.3	14.5	65	20	80	4.0	3.0	45.1
69598	S793450	MIN02	POC	349	2.0	1.8	0.3	1.5	45	5	95	0.1	2.0	11.3
69604	S816768	MIN02	POC	413	11.6	10.1	4.3	1.6	95	5	95	0.0	0.0	42.4
69615	S843673	MIN02	55-gallon	285	13.3	11.6	85.6	60.9	75	15	85	23.0	0.0	73.1
69616	S841627	MIN02	55-gallon	437	9.8	8.5	62.1	35.5	90	10	90	26.0	3.0	135.6
69618	S818412	MIN02	55-gallon	373	4.1	3.6	3.6	20.5	75	20	80	23.0	7.0	106.1
69620	S816768	MIN02	55-gallon	376	20.4	17.8	9.1	51.3	90	20	80	23.0	2.0	112.1
69630	S843672	MIN02	55-gallon	437	20.4	17.8	74.2	28.6	95	10	90	5.0	5.0	155.1
69633	S851418	MIN02	55-gallon	417	20.2	17.6	59.0	36.5	90	10	90	10.0	4.0	142.1
69634	S851416	MIN02	55-gallon	501	11.0	9.5	35.1	25.5	95	5	95	10.0	0.0	183.6
69635	S851418	MIN02	55-gallon	240	4.4	3.8	17.3	40.5	60	5	95	0.5	5.0	70.1
69636	S843672	MIN02	55-gallon	209	15.1	13.1	50.2	16.8	65	15	85	5.0	6.0	50.6
69637	S813471	MIN02	55-gallon	301	6.7	5.8	5.2	26.5	75	20	80	5.0	3.5	94.6
69638	S822679	MIN02	55-gallon	331	8.1	7.1	5.9	43.5	70	5	95	0.0	5.0	111.6

Remediated Nitrate Salt Drums														
PKG_ID	ParentID	Waste Stream	Container Type	Gross Wt (lbs)	PECi	EC PECi	FGE	TOTAL DOSE	Drum Utiilzation (vol%)	Debris (vol%)	Hogeneous Solids (vol%)	Lead/Metal (kg)	Non-Metal (kg)	Salt/Swheat Matrix (kg)
69639	S843673	MIN02	55-gallon	362	4.4	3.8	31.5	8.5	95	10	90	15.0	3.0	113.1
69641	S813471	MIN02	55-gallon	323	9.1	7.9	5.1	75.5	70	10	90	15.1	5.0	92.5
69642	S818412	MIN02	55-gallon	215	4.9	4.3	3.3	14.5	55	15	85	8.0	0.0	56.1
69644	S793450	MIN02	55-gallon	510	9.3	8.1	4.2	30.5	100	5	95	8.1	7.0	183.0
69645	S822679	MIN02	55-gallon	352	16.1	14.0	17.2	22.5	55	15	85	20.0	6.0	100.6
87823	S864332	MIN02	POC	376	0.9	0.1	10.4	0.65	95	30	70	0.0	2.0	54.0
87825	S864332	MIN02	POC	378	2.1	0.2	22.8	0.95	95	70	30	3.1	2.0	20.8
87826	S864332	MHD01	POC	392	0.9	0.2	10.4	1.2	95	90	10	0.0	25.3	10.0
87827	S864332	MHD01	POC	342	0.9	0.2	10.4	0.75	95	95	5	0.0	9.3	3.0
92459	90316	MIN02	55-gallon	247	2.0	0.5	9.6	30	90	30	70	12.0	12.0	54.6
92472	90317	MHD01	55-gallon	228	3.3	2.8	11.9	50.3	65	60	40	19.5	16.0	34.6
92669	90900	MHD01	55-gallon	250	2.0	1.8	8.6	12.3	85	55	45	28.0	16.0	36.1
93605	S824541	MIN02	55-gallon	348	14.8	13.0	21.3	50.7	75	20	80	10.5	4.0	110.5
94068	S851852	CIN01	55-gallon	398	29.0	0.2	173.2	81.3	80	15	85	12.0	4.0	131.1
94227	S813475	MHD01	55-gallon	208	1.3	0.0	3.5	11.7	100	75	25	42.0	3.0	16.1

APPENDIX B UNREMEDIATED NITRATE SALT DRUMS

Unremediated Nitrate Salt Drums														
PKG_ID	Waste Stream	EPA Codes	Container Type	Gross Wt (lb)	PECi	EC PECi	FGE	Total Dose	Liners	Liner wt. (kg)	Pb/Metal (kg)	Nonmetal (kg)	Water vol gal	Salt wt. (kg)
S864213	CIN01	1,2,7,8,9	85/55 gal	434	5.5	0.0	61.1	8	Rigid/Lead	15	12	5.0	5	117
S862888	CIN01	1,2,7,8,9	85/55 gal	373	1.8	0.0	19.4	2	Lead	12	12	3.0	3	90
S853714	CIN01	1,2,7,8,9	85/55 gal	457	6.9	0.0	76.7	4	Rigid/Lead	15	12	4.0	3	136
S842446	CIN01	1,7,8,9	85/55 gal	572	4.9	0.0	44.7	5	Rigid/Lead	15	12	0.0	0	100
S825879	CIN01	1,2,7,8,9	85/55 gal	352	4.7	0.0	52.1	5	Rigid/Lead	15	12	0.5	1	51
S825878	CIN01	1,7,8,9	85/55 gal	358	16.7	0.1	186.3	2	Rigid/Lead	15	12	0.0	0	88
S823184	CIN01	1,2,7,8,9	85/55 gal	443	14.2	0.1	158.6	2	Rigid/Lead	15	12	5.0	5	128
S823124	CIN01	1,7,8,9	85/55 gal	578	2.7	0.0	30.2	1	Rigid/Lead	15	12	0.0	0	159
S822844	CIN01	1,2,7,8,9	85/55 gal	481	8.6	0.1	95.6	2	Rigid/Lead	15	12	15.0	15	147
S822713	CIN01	1,2,7,8,9	85/55 gal	521	1.3	0.0	11.0	120	Rigid/Lead	15	12	5.0	5	114
S822599	CIN01	1,2,7,8,9	85/55 gal	435	5.2	0.0	58.5	7	Rigid/Lead	15	12	4.0	4	124
S818435	CIN01	1,2,7,8,9	85 gal	351	1.8	0.0	20.3	2	Rigid/Lead	15	12	5.0	5	92
S816810	CIN01	1,2,7,8,9	85/55 gal	511	2.1	0.0	22.3	1	Rigid/Lead	15	12	5.0	5	133
S816434	CIN01	1,2,7,8,9	85 gal	476	3.5	0.0	38.9	2	Rigid/Lead	15	12	1.0	1	150
S813545	CIN01	1,2,7,8,9	85/55 gal	419	1.0	0.0	11.5	0	Rigid/Lead	15	12	4.0	4	79
S813385	CIN01	1,2,7,8,9	85 gal	442	4.1	0.0	45.8	11	Rigid/Lead	15	12	1.0	1	104
S805289	CIN01	1,2,7,8,9	85/55 gal	470	0.3	0.0	0.2	2	Rigid/Lead	15	12	3.0	3	140
S805051	CIN01	1,2,7,8,9	85 gal	397	0.6	0.0	6.2	1	Rigid/Lead	15	12	2.0	2	112
S804995	CIN01	1,2,7,8,9	85/55 gal	415	1.1	0.0	12.3	2	Rigid/Lead	15	12	2.0	2	115
S804948	CIN01	1,2,7,8,9	85/55 gal	414	0.5	0.0	4.3	0	Rigid/Lead	15	12	2.0	2	101
S803078	CIN01	1,2,7,8,9	85/55 gal	382	1.7	0.0	15.3	1	Rigid/Lead	15	12	3.0	3	101
S802833	CIN01	1,2,7,8,9	85/55 gal	417	0.4	0.0	4.2	3	Lead	12	12	5.0	5	113
S802739	CIN01	1,2,7,8,9	85/55 gal	407	0.6	0.0	6.0	1	Rigid/Lead	15	12	2.0	2	106
S801676	CIN01	1,2,7,8,9	85/55 gal	421	1.2	0.0	11.3	2	Rigid/Lead	15	12	2.0	2	112
S793724	CIN01	1,2,7,8,9	85/55 gal	365	1.4	0.0	9.5	2	Rigid/Lead	15	12	2.0	2	89
70072	CIN01	1,2,7,8,9	85/55 gal	490	4.7	0.0	49.6	11.3	No Liners	15	12	5.0	5	128
70069	MIN02	1,2,7,8,9	85/55 gal	464	0.6	0.5	5.4		Rigid/Lead	15	12	0.5	1	102
69907	MIN02	1,2,7,8,9	85/55 gal	429	14.4	12.5	149.1	9	Rigid/Lead	15	12	5.0	5	100
69904	MIN02	1,2,7,8,9	85/55 gal	509	10.3	8.9	114.7	5.25	Rigid/Lead	15	12	0.5	1	151

APPENDIX C BELOWGRADE POSSIBLE SALT DRUM CONTENT INFORMATION

Belowgrade Nitrate Salt Drums								
PKG_ID	Waste Code	Location	Container Type	Gross Wt (lb)	PECi	EC PECi	FGE	Total Dose
S790007	25	Pit 9	55 gal	247	6.7	0.0	70.3	5
S790008	25	Pit 9	55 gal	255	15.3	0.1	171.1	8
S790009	25	Pit 9	55 gal	290	7.3	0.0	81.7	0
S790010	25	Pit 9	55 gal	168	11.2	0.1	117.3	6
S790011	25	Pit 9	55 gal	33	1.4	0.0	16.1	1
S790012	25	Pit 9	55 gal	67	1.6	0.0	17.9	1
S790013	25	Pit 9	55 gal	58	4.1	0.0	35.4	1
S790039	25	Pit 9	55 gal	213	19.5	0.1	98.7	10
S790061	25	Pit 9	55 gal	130	3.4	0.0	31.6	0
S790070	25	Pit 9	55 gal	256	75.5	0.5	13.8	3
S790071	25	Pit 9	55 gal	251	2.3	0.0	16.1	2
S790085	25	Pit 9	55 gal	246	6.2	0.0	69.9	0
S790087	25	Pit 9	55 gal	139	54.2	0.3	155.5	0
S790098	25	Pit 9	55 gal	210	15.2	0.1	121.8	0
S791736	25	Pit 9	55 gal	152	15.5	0.1	171.7	2
S791737	25	Pit 9	55 gal	115	0.2	0.0	2.0	1
S791751	25	Pit 9	55 gal	336	29.8	0.2	39.2	8
S791752	25	Pit 9	55 gal	433	1.2	0.0	11.1	1
S791754	25	Pit 9	55 gal	255	4.1	0.0	44.6	2
S791923	25	Pit 9	55 gal	262	0.0	0.0	0.0	0
S791924	25	Pit 9	55 gal	290	2.6	0.0	20.3	1
S791925	25	Pit 9	55 gal	361	4.2	0.0	29.7	2
S791926	25	Pit 9	55 gal	346	0.7	0.0	5.5	2
S791927	25	Pit 9	55 gal	373	0.8	0.0	5.6	1
S791934	25	Pit 9	55 gal	375	1.2	0.0	10.2	2
S791944	25	Pit 9	55 gal	376	1.4	0.0	11.7	2
S791947	25	Pit 9	55 gal	84	3.5	0.0	38.7	3
S791948	25	Pit 9	55 gal	410	0.4	0.0	3.5	1
S791950	25	Pit 9	55 gal	448	0.5	0.0	3.8	1
S791952	25	Pit 9	55 gal	220	7.6	0.0	58.9	1
S793025	25	Pit 9	55 gal	186	8.0	0.0	59.2	0
S793063	25	Pit 9	55 gal	241	25.9	0.2	96.0	3
S793110	25	Pit 9	55 gal	381	1.6	0.0	12.8	1
S793113	25	Pit 9	55 gal	191	13.2	0.1	134.0	2
S793125	25	Pit 9	55 gal	174	37.8	0.2	176.6	10
S793143	25	Pit 9	55 gal	423	2.3	0.0	15.8	2
S793152	25	Pit 9	55 gal	148	0.2	0.0	1.1	2
S793159	25	Pit 9	55 gal	241	31.8	0.2	187.2	40
S793172	25	Pit 9	55 gal	143	11.4	0.1	98.4	4
S793178	25	Pit 9	55 gal	125	26.6	0.2	158.8	4
S793180	25	Pit 9	55 gal	125	0.1	0.0	0.9	2
S793190	25	Pit 9	55 gal	138	40.5	0.3	159.2	8

Belowgrade Nitrate Salt Drums								
PKG_ID	Waste Code	Location	Container Type	Gross Wt (lb)	PECi	EC PECi	FGE	Total Dose
S793194	25	Pit 9	55 gal	358	1.1	0.0	9.1	2
S793196	25	Pit 9	55 gal	317	65.5	0.4	146.2	15
S793204	25	Pit 9	55 gal	240	5.2	0.0	45.2	5
S793212	25	Pit 9	55 gal	161	2.0	0.0	12.5	3
S793216	25	Pit 9	55 gal	217	10.0	0.1	134.5	10
S793219	25	Pit 9	55 gal	328	2.7	0.0	18.8	3
S793220	25	Pit 9	55 gal	276	36.4	0.2	84.9	7
S793244	25	Pit 9	55 gal	122	23.1	0.1	171.4	15
S793250	25	Pit 9	55 gal	214	34.3	0.2	88.8	50
S793276	25	Pit 9	55 gal	199	26.0	0.2	51.0	100
S793279	25	Pit 9	55 gal	334	11.3	0.1	85.1	10
S793292	25	Pit 9	55 gal	393	2.0	0.0	14.8	5
S793404	25	Pit 9	55 gal	157	53.6	0.3	164.5	2
S793410	25	Pit 9	55 gal	96	6.4	0.0	53.8	4
S793411	25	Pit 9	55 gal	95	1.3	0.0	9.0	6
S793429	25	Pit 9	55 gal	121	49.3	49.3	150.5	13
S793443	25	Pit 9	55 gal	170	16.1	0.1	184.1	1
S793451	25	Pit 9	55 gal	238	6.4	0.0	71.7	2
S793455	25	Pit 9	55 gal	289	19.2	0.1	33.9	10
S793475	25	Pit 9	55 gal	221	20.1	0.1	86.4	8
S793490	25	Pit 9	55 gal	358	2.2	0.0	16.3	1
S793706	25	Pit 9	55 gal	390	2.2	0.0	9.2	1
S793707	25	Trench OC	55 gal	386	0.7	0.0	4.9	1
S793768	25	Trench OC	30 gal cask	70	120.9	120.9	2.2	15
S793769	25	Trench OC	30 gal cask	55	14.5	14.5	3.1	1
S802571	25	Trench OC	30 gal cask	78	110.5	110.5	2.0	1
S803091	25	Trench OC	30 gal cask	64	29.9	29.9	0.6	2
S816426	25	Trench OC	30 gal cask	37	122.2	122.2	2.3	15
S822540	25	Trench 0D	30 gal cask	35	26.0	26.0	0.5	2
S822560	25	Trench 0D	30 gal cask	47	200.1	1.2	3.7	8
S824154	25	Trench 0D	30 gal cask	69	180.7	1.1	3.3	4
S832274	25	Trench 0A	30 gal cask	76	92.3	0.6	1.7	5
S842188	25	Trench 0D	30 gal cask	59	222.2	1.4	4.1	18
S844290	25	Trench 0D	30 gal cask	41	46.8	0.3	0.9	6
S845076	25	Trench 0D	30 gal cask	59	210.5	1.3	3.9	16
S846104	25	Trench 0A	30 gal cask	70	144.3	0.9	2.7	12
S846105	25	Trench 0D	30 gal cask	55	166.4	1.0	3.1	8

APPENDIX D WASTE CONTROL SPECIALISTS, LLC, NITRATE SALT DRUM INFORMATION

WCS Nitrate Salt Drums													
PKG_ID	Waste Stream	Contnr Type	Gross Wt (lb)	PECi	EC PECi	FGE	Total Dose	Vol Util. (%)	Liner wt. (kg)	Metal (kg)	Nonmetal (kg)	Container (kg)	Salt wt. (kg)
68311	MIN02	55-GAL	306	5.4	4.8	32.0	14.5	75	5.7	0.1	6.3	28	99
68313	MIN02	55-GAL	308	14.3	12.6	112.3	12.5	65	5.7	19	4	28	84
68314	MIN02	55-GAL	206	1.8	1.6	10.0	10.5	50	5.7	0	6.1	28	54
68315	MIN02	55-GAL	359	27.7	24.4	186.0	61.2	75	5.7	25	4	28	101
68325	MIN02	POC	376	11.8	10.4	54.2	2.5	55	36.1	0	0	109	25
68341	MIN02	POC	377	13.1	11.5	62.0	2.6	80	36.1	0.5	0	109	25
68342	MIN02	POC	334	2.6	2.3	15.0	1	35	36.1	0	1	109	5
68347	MIN02	POC	372	6.7	5.9	13.2	1.1	80	36.1	0	4	109	20
68350	MIN02	POC	388	11.8	10.4	34.4	1.7	85	36.1	0	0	109	31
68396	MHD01	POC	330	3.7	0.9	15.6	1	45	36.1	2.7	2.2	109	
68425	MIN02	POC	386	2.9	2.5	0.7	1.5	80	36.1	0.3	0	109	29
68426	MIN02	POC	347	15.5	13.6	147.1	1.5	50	36.1	0	1	109	12
68428	MIN02	POC	380	6.7	5.9	9.4	1.5	85	36.1	0	2	109	25
68429	MIN02	POC	396	4.2	3.7	9.7	1.5	90	36.1	0	0	109	34
68431	MIN02	POC	362	12.3	10.7	45.3	1.8	70	36.1	0	4	109	15
68432	MIN02	POC	337	0.3	0.3	2.3	1.5	30	36.1	0	1	109	7
68433	MIN02	POC	351	1.0	0.9	9.0	0.8	40	36.1	0	1	109	13
68449	MIN02	55-GAL	429	13.1	11.5	68.0	40.5	90	5.7	20	4	28	138
68508	MIN02	POC	398	7.9	6.9	35.8	1.5	95	36.1	0	0	109	35
68509	MIN02	POC	370	5.5	4.9	14.2	1	75	36.1	0	0	109	22
68543	MIN02	55-GAL	358	8.4	7.3	46.7	25.5	85	5.7	6	2	28	121
68546	MIN02	55-GAL	415	47.4	41.7	146.6	72	85	5.7	10	1	28	144
68580	MIN02	55-GAL	465	4.5	3.9	11.3	8.5	90	5.7	20	11	28	147
68583	MIN02	POC	382	5.7	5.0	2.1	2	75	36.1	0	0	109	29
68584	MIN02	POC	361	1.5	1.3	16.2	0.9	50	36.1	0	2	109	17
68617	MIN02	POC	341	2.5	2.2	28.9	0.8	50	36.1	0.3	0	109	10
68619	MIN02	POC	392	4.6	4.0	35.3	0.9	75	36.1	0	0	109	32
68620	MIN02	POC	403	0.9	0.8	10.6	1.2	75	36.1	0	2	109	35
68625	MIN02	55-GAL	295	2.0	1.8	22.7	52	50	5.7	7	4	28	89
68627	MIN02	55-GAL	357	0.3	0.3	2.3	30.5	85	5.7	35	8	28	86
68628	MIN02	55-GAL	370	0.9	0.8	10.6	70.5	80	5.7	8	4	28	123
68632	MIN02	55-GAL	342	2.5	2.2	28.8	60.5	80	5.7	6.5	4.2	28	111
68656	MIN02	55-GAL	461	34.4	30.3	160.1	51	95	5.7	5	21	28	150
68661	MIN02	55-GAL	309	3.2	2.8	16.5	18.5	65	5.7	5	7	28	94
68676	MIN02	55-GAL	473	6.0	5.2	4.2	18.5	100	5.7	6	6	28	169
68679	MIN02	55-GAL	507	18.8	16.5	35.6	20.5	100	5.7	5	4	28	187
68681	MIN02	55-GAL	374	11.3	10.0	78.2	35.5	80	5.7	2	1	28	133
68686	MIN02	55-GAL	367	9.0	7.9	43.7	50.5	75	5.7	10	6	28	117
69014	MIN02	POC	352	3.6	3.2	11.8	1.1	70	36.1	0	2	109	12
69033	MIN02	55-GAL	262	13.4	11.7	30.5	60.5	65	5.7	10	1	28	75
69034	MIN02	55-GAL	303	11.4	10.0	43.4	100.5	75	5.7	2.5	4	28	97
69041	MIN02	55-GAL	444	12.2	10.8	33.9	16.5	85	5.7	38	0	28	130
69043	MIN02	55-GAL	420	11.3	9.9	78.8	40.5	90	5.7	0	6	28	151

WCS Nitrate Salt Drums													
PKG_ID	Waste Stream	Contnr Type	Gross Wt (lb)	PECi	EC PECi	FGE	Total Dose	Vol Util. (%)	Liner wt. (kg)	Metal (kg)	Nonmetal (kg)	Container (kg)	Salt wt. (kg)
69045	MIN02	55-GAL	288	9.0	7.9	83.3	12.5	80	5.7	20	6	28	72
69060	MIN02	55-GAL	210	11.8	10.4	68.8	85.8	50	5.7	0.2	5	28	56
69061	MIN02	55-GAL	256	12.3	10.8	70.9	15.6	50	5.7	20	2	28	61
69063	MIN02	55-GAL	350	14.4	12.7	102.9	37.5	80	5.7	2	7	28	117
69064	MIN02	55-GAL	334	6.1	5.4	34.0	32.5	80	5.7	10	9	28	99
69066	MIN02	55-GAL	379	15.2	13.4	107.0	20.5	90	5.7	20	4	28	115
69067	MIN02	55-GAL	246	17.0	15.0	107.8	60.9	50	5.7	10.1	2	28	66
69068	MIN02	55-GAL	244	2.3	2.0	11.8	18.5	75	5.7	0.1	9.5	28	68
69069	MIN02	55-GAL	227	3.0	2.7	19.1	16.5	50	5.7	10.1	6.5	28	53
69073	MIN02	55-GAL	256	13.6	12.0	63.0	38.8	75	5.7	5	6	28	72
69074	MIN02	55-GAL	229	11.2	9.9	95.8	16.8	55	5.7	3	2	28	66
69077	MIN02	55-GAL	294	1.3	1.1	9.0	5.5	85	5.7	27	6	28	67
69080	MIN02	55-GAL	295	6.1	5.4	19.9	50.5	75	5.7	16	3	28	81
69081	MIN02	55-GAL	191	18.2	16.0	118.6	87	50	5.7	5	2	28	47
69083	MIN02	55-GAL	431	10.7	9.4	57.5	28.5	95	5.7	12	8	28	142
69085	MIN02	POC	346	6.4	5.7	47.7	1.5	50	36.1	0.5	2	109	9
69087	MIN02	POC	382	9.8	8.6	68.6	1.5	90	36.1	3	0	109	25
69091	MIN02	55-GAL	268	9.2	8.1	32.1	37.5	65	5.7	0.5	2.5	28	85
69094	MIN02	55-GAL	416	9.6	8.5	7.3	70.5	55	5.7	0	6	28	82
69097	MIN02	55-GAL	415	11.3	9.9	92.4	25.5	100	5.7	24	6	28	123
69099	MIN02	55-GAL	235	14.5	12.7	65.6	48.8	40	5.7	0	2	28	72
69102	MIN02	POC	348	3.8	3.3	20.2	0.9	50	36.1	0	2	109	11
69103	MIN02	POC	375	7.9	7.0	65.7	1.5	85	36.1	0.5	2	109	22
69105	MIN02	POC	425	4.2	3.7	19.5	1.5	70	36.1	1.5	0	109	18
69154	MIN02	55-GAL	134	1.7	1.5	9.9	7.5	25	5.7	10	2	28	16
69158	MIN02	55-GAL	182	19.4	17.0	90.2	50.5	40	5.7	19.5	2	28	28
69159	MIN02	55-GAL	457	26.3	23.1	97.4	71.5	95	5.7	23	6	28	144
69161	MIN02	55-GAL	370	5.1	4.5	8.0	18.5	85	5.7	0	8	28	126
69162	MIN02	55-GAL	339	7.9	7.0	35.3	27.5	85	5.7	5.2	6.1	28	109
69163	MIN02	55-GAL	414	8.3	7.3	30.6	36.8	95	5.7	0	6	28	148
69177	MIN02	55-GAL	279	2.9	2.6	9.8	15.5	70	5.7	0.1	6	28	95
69179	MIN02	55-GAL	384	9.9	8.7	79.1	20.5	95	5.7	5.2	6	28	129
69180	MIN02	55-GAL	337	31.8	28.0	137.0	51.5	70	5.7	8	4	28	108
69181	MIN02	55-GAL	213	2.2	1.9	13.9	10.5	55	5.7	0.5	4	28	59
69182	MIN02	55-GAL	252	8.5	7.5	52.5	34	60	5.7	5.1	4	28	72
69185	MIN02	POC	381	1.9	1.7	5.0	0.9	85	36.1	0	2	109	25
69187	MIN02	POC	378	6.1	5.4	20.1	1.5	65	36.1	0	2	109	24
69188	MIN02	POC	382	4.1	3.6	15.9	1.5	80	36.1	0	2	109	26
69189	MIN02	POC	343	2.3	2.0	11.0	1.5	60	36.1	0	2	109	8
69191	MIN02	POC	393	6.4	5.6	8.8	1.2	75	36.1	0	2	109	31
69192	MIN02	POC	342	3.1	2.7	21.8	1.5	55	36.1	0	1	109	9
69193	MIN02	55-GAL	332	8.3	7.3	47.9	30.5	75	5.7	0	9	28	108
69194	MIN02	55-GAL	248	0.4	0.3	1.4	3.7	65	5.7	2	8.5	28	69
69195	MIN02	55-GAL	265	11.9	10.5	52.8	17.5	55	5.7	35	0	28	52
69196	MIN02	55-GAL	115	2.1	1.8	5.8	25.5	40	5.7	0	5.1	28	14

WCS Nitrate Salt Drums													
PKG_ID	Waste Stream	Contnr Type	Gross Wt (lb)	PECi	EC PECi	FGE	Total Dose	Vol Util. (%)	Liner wt. (kg)	Metal (kg)	Nonmetal (kg)	Container (kg)	Salt wt. (kg)
69209	MIN02	55-GAL	532	43.3	38.1	93.1	70.5	95	5.7	25	5	28	178
69210	MIN02	55-GAL	288	3.2	2.8	6.6	10.5	55	5.7	15	4	28	78
69216	MIN02	55-GAL	232	10.9	9.5	85.0	32.8	60	5.7	0	2	28	50
69217	MIN02	55-GAL	305	10.0	8.8	17.0	29	75	5.7	23.3	4.2	28	78
69226	MIN02	55-GAL	300	20.8	18.3	100.6	46	60	5.7	5.5	7.2	28	90
69230	CIN01	55-GAL	349	6.4	6.4	31.9	10.5	95	5.7	1.5	0	28	123
69232	MIN02	55-GAL	383	4.0	0.5	44.2	15.5	95	5.7	16	0	28	127
69234	MIN02	55-GAL	347	2.9	2.6	9.3	11.5	80	5.7	1	7	28	116
69235	MIN02	55-GAL	265	1.0	0.1	11.2	15.5	60	5.7	5	2	28	80
69237	MIN02	55-GAL	383	12.9	11.3	55.2	45.5	80	5.7	18	0	28	122
69279	MIN02	55-GAL	385	28.2	24.8	102.4	80.9	70	5.7	6	4	28	132
69282	MIN02	55-GAL	354	12.7	11.1	71.4	50.5	80	5.7	5	4	28	118
69285	MIN02	55-GAL	287	13.9	12.2	93.1	91	60	5.7	8	5.5	28	83
69295	MIN02	55-GAL	317	3.0	2.6	6.0	21.5	100	5.7	10.1	8	28	93
69402	MIN02	55-GAL	390	5.2	4.5	57.6	32.5	90	5.7	6	8	28	129
69413	MIN02	55-GAL	293	4.1	3.6	19.3	7.5	65	5.7	5	2	28	93
69422	MIN02	55-GAL	253	10.1	8.9	4.4	23	50	5.7	10	0	28	72
69428	MIN02	55-GAL	385	11.4	10.0	4.4	32.5	80	5.7	0	8.3	28	133
69430	MIN02	55-GAL	419	3.9	3.4	41.7	17.7	90	5.7	1.5	4	28	151
69492	MIN02	55-GAL	352	1.2	1.1	13.8	14.5	75	5.7	0	8	28	119
69493	MIN02	55-GAL	375	18.4	16.2	63.0	31.2	80	5.7	12	8	28	116
69555	MIN02	55-GAL	375	27.1	23.9	70.3	80.5	90	5.7	15.1	1	28	121
69565	MIN02	55-GAL	315	24.5	21.3	144.4	28.9	80	5.7	23	3	28	84
92900	MHD01	55-GAL	283	6.3	1.6	20.5	32.6	80	5.7	23	12	28	60
94201	CIN01	55-GAL	503	24.4	0.2	124.3	49.9	100	5.7	0	11	28	182
94211	CIN01	POC	405	17.2	0.1	100.2	1.9	95	36.1	0	0	109	39

APPENDIX E MOVER INCIDENT ANALYSIS REPORT FINDINGS

Root Causes (RC)

RC 1: Lawrence Livermore National Laboratory's (LLNL's) initial evaluation and formal acceptance testing of the Vendor's confinement system (design, technique, and procedures) were less than adequate.

RC 2: LLNL's ongoing evaluation of the Vendor's confinement system (design, technique, and procedures) was less than adequate for the bag-in and bag-out operation involving LLNL transuranic (TRU) waste drums.

Contributing Causes (CC)

CC 1: The CCP/Los Alamos National Laboratory (LANL) initial design of glovebox drum port/bag interface, necessary to maintain the integrity of the seal when working with materials from LLNL drums. Specific supporting examples are as follows:

- The design of the retaining band was LTA and did not address performance specifications (e.g., torque, change-out frequency) for the band.
- The approach to establishing a seal at the drum ports was ineffective, including use of a retaining clamp on the ends of the internal O-ring of the bags, tape under the exterior retaining band, and lack of taping the end of the bag to the port.
- The design to achieve a wrinkle-free attachment of the bag around the full circumference of the drum port was LTA.

CC 2: LLNL's response to the National Nuclear Security Administration's/LSO's ORR comment on the Vendor's configuration management did not fully address the flow down of design intent and specifications, from a radiological control standpoint, to end users at LLNL.

CC 3: The Vendor's (CCP's) safety management of ongoing operations was LTA. Specific supporting examples are as follows:

- Communication to LLNL of previous MOVER (Mobile Visual Examination and Repackaging) operation experiences was not adequate. The full paper trail for exposure histories and airborne monitoring at all sites (Nevada Test Site, Argonne National Laboratory, LANL) where the MOVER had operated was not provided to LLNL by CCP.
- Changes to operations (addition of the bungee cord) to address emerging issues (low-level airborne contamination) were agreed to by LLNL and CCP project managers; however, CCP did not consider these to require procedure or design changes. As a result, these changes were not reviewed by the CCP safety organization. CCP did not seem inquisitive as to the safety implications of change or its need to conduct a safety review of the change. Rather, the main concern was if the change impacted the Waste Isolation Pilot Plant (WIPP) certification for the drums.

CC 4: Vendor's operational procedures did not include methods for recognizing and responding to changing conditions. Specific supporting examples are as follows:

- The approach and procedure for handling low-Ci and high-Ci drums in the glovebox were the same; consideration of implications of the change in drum activity and material form was LTA.
- Operators normalized events (low-vacuum alarms, elevated meter readings, and minor contaminations) and considered them minor nuisances.
- Identification of radiological hold points for out-of-normal conditions was LTA.

CC 5: LLNL's verification of the Vendor's quality assurance plan for the design and fabrication of the confinement system (design, technique, and procedures) was LTA.

CC 6: Communication of technical issues and operational problems up the LLNL and CCP line management systems was LTA.

Judgments of Need (JONs)

JON 1: LLNL needs to recommend to the Vendor that the seal between the bag and drum port needs to be redesigned to achieve an effective seal. This need includes, but is not limited to, recommending that the Vendor evaluate the current process against other, more effective interface-seal processes used in gloveboxes to seal the drum port/bag interface; modify its seal process based on its evaluation; evaluate the current drum port bags against other drum port bags; and provide an effective drum port/bag interface seal.

JON 2: LLNL needs to evaluate and revise, as necessary, its current ORR process to ensure that the adequacy of a subcontract's design data information and quality assurance (QA) plan/program are assessed as part of the ORR process to identify any gaps in the adequacy of a subcontract's design review for quality significant equipment.

JON 3: LLNL needs to review its process for using Vendor-supplied, quality-significant equipment in a nuclear facility to ensure the equipment is evaluated either through an LLNL design review or other adequate Vendor design review.

JON 4: LLNL needs to review and revise, as necessary, its processes for formal communication of the risks associated with Vendor-supplied, quality-significant equipment that has unverified design reviews to the cognizant approval authority.

JON 5: LLNL needs to formally communicate to the Vendor the safety management significance and importance of the Vendor providing historical operational information—involving Vendor-supplied, quality-significant equipment—to U.S. Department of Energy sites before the equipment is put to use.

JON 6: LLNL needs to formally communicate to the Vendor the safety management significance and importance of the Vendor reviewing site proposed process changes involving Vendor-supplied, quality-significant equipment for safety impacts relative to the initial design specifications and radiological control intent.

JON 7: LLNL needs to review and revise, as necessary, its current procedure review processes (contained in the Environment, Safety and Health manual) to ensure that hold points are assessed for their appropriateness and incorporated into procedures to proactively prevent out-of-control conditions from occurring, to allow management adequate time to evaluate areas of concern, and to render effective decisions to address the concerns.

JON 8: LLNL needs to issue a lessons learned document on the importance and process for effectively communicating technical issues, operational problems, safety concerns, and off-normal conditions to line management in a timely manner.

JON 9: LLNL needs to develop for submittal to DOE a lessons learned document on the issues identified in this Incident Analysis Report.

APPENDIX F NITRATE SALT SYSTEM ASSESSMENT SCORING BASIS

Blending
5 = No radiological issues for set up, system holds multiple blenders, or holds larger capacity blender and can access drum tumbler easily
4 = Moderate to set up in glovebox (GB) (contamination), holds multiple blenders or larger capacity blender, access drum tumbler easily
3 = Moderate to set up in GB (contamination)—single blender—can access drum tumbler easily
2 = Moderate to set up in GB (contamination)—single blender—drum tumbler outside facility
1 = Difficult to insert blender—single blender

Cementation
5 = No radiological issues for set up—multiple mixers—can feed drum tumbler easily
4 = No radiological issues for set up—single mixer—can access drum tumbler easily
3 = Moderate to set up in GB (contamination—single mixer—access to tumbler inside
2 = Moderate to set up in GB (contamination)—single mixer—drum tumbler outside facility
1 = Difficult to insert blender-single mixer—drum tumbler outside facility

Debris Waste
5 = Space for handling waste and treating debris, free liquid reservoir
4 = Moderate space for handling waste, free liquid reservoir
3 = Moderate space for handling waste, no free liquid reservoir
2 = Little space for debris waste, free liquid reservoir
1 = Little space for debris waste

Remediated Nitrate Salt (RNS) and Unremediated Nitrate Salt (UNS) Drums
5 = Capable of handling all drums
4 =
3 = 18 Equivalent Combustible Pu-Equivalent Curies (ECPE-Ci)
2 =
1 = Few RNS and UNS

Legacy Drums
5 = All drums
4 = Hazard Category 3 levels
3 = 18 ECPE-Ci
2 =
1 = Few RNS and UNS

Authorization Basis (AB) Issues
5 = Page change
4 =
3 =
2 =
1 = New DSA

Resource Conservation and Recovery Act (RCRA) Issues
5 = No modification required/RD&D Permit
4 = Class I modification
3 = Class II modification
2 =
1 = Class III modification

Installation
5 = Minimal to no modifications for startup
4 = Facility utility-related modifications to accept capability
3 = Facility structural, ventilation, and foundation modifications
2 = Facility structural, ventilation, and some site modifications
1 = Significant facility and site modifications

Fabrication
5 = No fabrication
4 = Minimal fabrication or maintenance upgrades
3 = Design and fabrication requiring equipment installation
2 = Design and fabrication requiring equipment installation and facility modifications
1 = Significant equipment fabrication and facility modifications

Regulatory/Public Acceptance
5 = Facility and process well configured for remediation operation with historical operating record and all permitting in place
4 = Facility and process well configured for remediation operations and minimal permitting required for operations
3 = Facility has track record for safe, compliant operations. New permitting required for operations.
2 = Facility systems and infrastructure meet required performance, not all drums permitted for processing, some new permitting required
1 = Facility systems and infrastructure meet required performance, not all drums permitted for processing, all new permitting required

Operation
5 = Drums easily accessible, operators easy access to box and systems, limited personal protective equipment (PPE), as low as reasonably achievable (ALARA) impact, high drum process rate, low complexity to operate systems
4 = Drum accessible, operator access, limited PPE, ALARA impact, drum process rate, low complexity
3 = Drum accessible, operator access, limited PPE, ALARA impact, drum process rate, low complexity
2 = Drum accessible, operator access, limited PPE, ALARA impact, drum process rate, low complexity
1 = Drum accessible, operator access, limited PPE, ALARA impact, drum process rate, low complexity

Schedule
5 = Page change for AB issues only to start and Class II RCRA
4 = Resolve AB issues, minimal fabrication and installation
3 = Resolve AB issues, moderate fabrication and installation
2 = Resolve AB issues, Class III RCRA, significant fabrication and installation, off-site coordination and effort
1 = New documented safety analysis for AB issues, Class III RCRA, significant fabrication, off-site coordination and effort

Appendix 8

Engineered Option Treatment of Remediated Nitrate Salts: Surrogate Batch-Blending Testing

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March 9, 2016

Engineered Option Treatment of Remediated Nitrate Salts: Surrogate Batch-Blending Testing

Los Alamos
NATIONAL LABORATORY
EST. 1943

LA-UR-16-21653

Prepared by Kurt R. Anast
Associate Directorate Environmental Management

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EXECUTIVE SUMMARY

This report provides results from batch-blending test work for remediated nitrate salt (RNS) treatment. Batch blending was identified as a preferred option for blending RNS and unremediated nitrate salt (UNS) material with zeolite to effectively safe the salt/Swheat material identified as ignitable (U.S. Environmental Protection Agency code D001). Blending with zeolite was the preferred remediation option identified in the Options Assessment Report[1] and was originally proposed as the best option for remediation by Clark and Funk in their report, Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes[2] and also found to be a preferred option in the Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing.[3]

This test work evaluated equipment and recipe alternatives to achieve effective blending of surrogate waste with zeolite. An 8-qt Kitchen Aid blender was used to blend zeolite and surrogate RNS waste to ensure a well-blended product could be prepared from a range of surrogate formulations. Surrogate blends of salt and Swheat were prepared with dyed Swheat and colored sodium chloride rock salt. These color-enhanced surrogates provided a means to visually evaluate the effectiveness of blending the surrogate with zeolite. The surrogate feed material for the test work was formulated to have a range of moisture and salt content to ensure the surrogates would bound the RNS waste characteristics expected to be found in the candidate drums.

Tests with salt combined with Waste Lock 770 were also carried out. Waste Lock 770 was combined with nitric acid in the laboratory and then blended with zeolite using the same process and blending operation used for surrogates formulated with Swheat. Comparable tests were performed with salt combined with water to identify any observable differences with the baseline nitric acid test, but none were observed. All further Waste Lock 770 testing was performed with water-saturated salt.

The 8-qt Kitchen Aid mixer (Model KSMC895ER) was found to effectively blend the entire range of surrogates RNS formulations with zeolite. Similar results were found with surrogates of salt and Waste Lock 770. The 8-qt Kitchen Aid model fits easily into the Waste Characterization Reduction and Repackaging Facility (WCRRF) glovebox, allows for two blenders to be used simultaneously, is relatively easy to install in the glovebox or remove if required, easy for one person to operate, and has a 2-gal volume mixing bowl. A mixing duration of 30 s to 1 min is adequate to get a well-blended zeolite and surrogate product. The resulting blend easily dumps out of the bowl when the side is tipped and tapped, resulting in 95% of the contents discharging. To minimize dose to the operator and to best contain the materials during blending, a slightly modified bowl with a cover will be fabricated for the RNS blending process. The bowl will have an expanded upper lip as well as a slip-on cover. These adjustments will ensure the material is enclosed during processing and will reduce possible dose to workers during the blending operation.

A recipe and process were identified based upon testing a range of surrogates and an examination of the resulting blend with zeolite. The batch-blending tests were designed to simulate the test work conducted at Southwest Research Institute (SWRI). The final blending recipe will be identified upon conclusion of SWRI nitrated surrogate testing. The intent is to verify that large-scale volume (6 qt) blending of colored Swheat, salt, and zeolite in ratios tested at SWRI will provide a well-blended and remediated product for

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[1] Robinson, B.A., and P.A. Stevens, December 16, 2015. “Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory, Revision 1,” Los Alamos National Laboratory document LA-UR-15-27180, Los Alamos, New Mexico.

[2] Clark, D.L., and D.J. Funk, February 17, 2015. “Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes,” Los Alamos National Laboratory document LA-UR-15-22393, Los Alamos, New Mexico.

[3] Anast, K.R., November 2015. “Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing,” Los Alamos National Laboratory document LA-UR-15-28900, Los Alamos, New Mexico.

the entire range of possible RNS physical waste compositions. The blending process was effective over the following ranges:

- Swheat moisture content: 25 wt% to 60 wt%
- RNS surrogate salt composition: 22 vol% to 50 vol%
- RNS surrogate-to-zeolite blend volume ratio: 1:2 to 1:4
- Water addition rate (RNS surrogate to water volume ratio): 1:1 to 1:0.5

Testing showed that a single recipe could be followed to provide (1) water to achieve a soupy mixture and dissolve salts in the RNS waste and (2) zeolite to absorb the RNS solution and remediate the Swheat material. The amount of zeolite will be confirmed with testing at SWRI on nitrated surrogate blends. The ratios of ingredients on a volume basis are

- 1 volume RNS waste (Swheat and nitrate salts),
- 0.65 volume water, and
- 2.5 volumes zeolite.

The recommended steps for the blending process and the recipe for the 8-qt Kitchen Aid mixer are as follows:

1. Add 1.15 qt warm water to bowl
2. Add 1.75 qt RNS waste
3. Blend on speed setting 4 for 3 min
4. Add 2.20 qt of zeolite
5. Blend on speed setting 8 for 10 s
6. Reduce speed to setting 3
7. Add 2.20 qt of zeolite
8. Blend for 30 s

This recipe includes the addition of water used to first mix with the RNS waste. The water acts to moisten the Swheat material and creates a soupy consistency that is easier to blend into the zeolite. It also provides a means to dissolve salts and aids in desorbing or wetting salts already contained in the Swheat. The zeolite addition rate provides sufficient zeolite for water absorption and remediating the nitrate salts and will be verified by SWRI tests. The recipe can be used for dry Swheat and salt, Swheat used to absorb free liquid found in the RNS parent drums, salt blended with Waste Lock 770, or a blend of these.

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Acronyms and Abbreviations

DOE	Department of Energy
EPA	Environmental Protection Agency (U.S.)
LANL	Los Alamos National Laboratory
LLW	low-level waste
MMLM	Merlin Melts Like Magic
NMED	New Mexico Environmental Department
RNS	remediated nitrate salts
SWB	standard waste boxes
SWRI	Southwest Research Institute
UNS	unremediated nitrate salts
WCRRF	Waste Characterization Reduction and Repackaging Facility
WIPP	Waste Isolation Pilot Plant
WL	Waste Lock

1.0 INTRODUCTION

1.1 Objective

The primary objective of conducting batch-blending tests is to identify processing equipment, an ingredient recipe, and the process steps for safing remediated nitrate salt (RNS) waste contained in 60 on-site drums at Los Alamos National Laboratory (LANL). The treated RNS and UNS waste will be packaged and shipped to the Waste Isolation Pilot Plant (WIPP) for disposal. Batch-blending tests on RNS surrogate material will be used to evaluate blending equipment and ingredient information for a processing recipe.

1.2 Background

This report documents the process development of Option 1 (modification of Option 1) for use in the treatment for the RNS and UNS waste containers at LANL. Modified Option 1 is the dissolution of the nitrate salts in water followed by blending of the drum waste (Swheat and nitrate salt) with zeolite. For the UNS waste, this consists of dissolving nitrate salts in water and then adding zeolite to absorb the salt solution. For RNS waste, the nitrate salt/Swheat material would be mixed first with water and then blended with zeolite to absorb the salt solution and remediate the Swheat material.

This engineered implementation of Option 1 treats the containerized material in a manner that renders the waste safe and suitable for transport and final disposal in the WIPP repository, under specifications listed in the WIPP Waste Acceptance Criteria (DOE/CBFO, 2013). LANL recognizes that the results must be thoroughly vetted with the New Mexico Environment Department (NMED) and that a modification to the LANL Hazardous Waste Facility Permit is a necessary step before implementation of this or any treatment option. Likewise, facility readiness and safety basis approvals must be received from the Department of Energy (DOE). This report presents LANL's process development and testing of modified Option 1, and documentation of the process for determining the recommended treatment option for the RNS and UNS wastes. The process will be followed using nitrated salts mixed with Swheat at Southwest Research Institute (SWRI) to prepare samples for confirmatory evaluation to ensure the zeolite blended product passes ignitability and spontaneous combustion tests.

After the release of radioactivity from the WIPP on February 14, 2014 and the subsequent recognition that the breached drum was a RNS waste drum processed at LANL (Drum 68660), LANL took a number of precautionary steps to protect workers, the public, and the environment. Drums stored at LANL continue to be maintained in isolated storage. Monitoring results are reported to the NMED under the LANL Nitrate Salt Bearing Waste Container Isolation Plan (Isolation Plan: LANL, 2014). Drums are currently stored under a High Efficiency Particulate Air filtration system and the temperature controls provided by the building, with active fire suppression systems. Monitoring of the drums consists of visual inspections, daily temperature measurements of the standard waste boxes (SWBs) containing the RNS waste drums, and periodic sampling and analysis of the headspace gases within these SWBs. This configuration of the RNS wastes at LANL represents the *initial state* for a subsequent treatment option being considered in this Addendum.

The report describes the process development of Option 1, one option of fourteen potential treatment options, recommended to permanently treat the combination of nitrate salt and Swheat drum waste. This report documents a small-scale, batch blending study conducted to ensure techniques are adequate to render the waste material treatable.

1.3 Engineered implementation of Option 1:

The focus of these tests is to identify the best equipment, process, and recipe to prepare a well-blended product that meets the waste acceptance criteria for disposal WIPP. The RNS drums that remain at LANL

include 60 identified drums, of which 56 were repackaged with an organic kitty litter and 4 were repackaged with Waste Lock 770, a solid, granular superabsorbent polymer. This cross-linked polyacrylate material swells and absorbs many times its weight in aqueous solutions. Waste Lock 770 has been engineered to absorb under pressure and has properties that make it ideally suited for the absorption and solidification of low-level waste (LLW) and other types of waste sludges. Swheat Scoop, primarily a wheat-based organic kitty litter, was added to the unremediated nitrate salt (UNS) waste during repackaging to absorb free liquids and remediate the ignitability characteristic of the nitrate salts. The resulting mixture was repackaged in daughter drums that became the RNS waste stream.

Swheat was found to increase the hazard associated with the UNS waste by creating a potential for exothermic chemical reactions[1]. After a release at WIPP from a stored LANL RNS drum containing Swheat, LANL initiated steps to isolate all remaining RNS waste drums located at LANL. The drums were overpacked in SWBs and placed in a Permacon, in Dome 375, at Area G, located in Technical Area 54. The RNS drums are being stored in a temperature-controlled environment to mitigate the oxidizing behavior of the waste in the drums. LANL designated all remaining RNS drums at LANL as “ignitable,” assigning U.S. Environmental Protection Agency (EPA) Hazardous Waste Number D001 after independent reactivity testing on surrogate samples containing Swheat and sodium nitrate salt[2]. Those drums containing free liquid have also been assigned an EPA code of D002 (corrosive).

An options assessment report and an engineering options assessment report[3] were prepared to evaluate various treatment options for the RNS and UNS waste streams to remove their hazardous characteristics, in response to a New Mexico Environment Department–issued Administrative Order. This assessment identified five candidate treatment options for remediation of both RNS and UNS drums at LANL. The preferred option was dry blending with zeolite as the primary unit operation for remediating the drums. This test work provides an evaluation of batch blending that could be effectively conducted inside a glovebox and identifies equipment, process steps, and a recipe for achieving a well-blended zeolite and RNS waste for disposal at WIPP.

Ongoing testing at SWRI with surrogate nitrate salt material will provide analytical verification of the zeolite-blended surrogates (SW-846, Test Method 1030, SW-846, Test Method 1050, UN DOT O.1 and O.2 oxidizer tests, and 9095B Paint Filter tests) that blending RNS waste with zeolite can (1) effectively deactivate the ignitable and corrosive characteristics of the RNS waste, (2) produce a product that no longer carries the D001 or D002 hazardous waste number designations, and (3) meet WIPP waste acceptance criteria. This report identifies how to achieve an acceptable blended product.

[1] Robinson, B.A., and P.A. Stevens, December 16, 2015. “Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory, Revision 1,” Los Alamos National Laboratory document LA-UR-15-27180, Los Alamos, New Mexico.

[2] Clark, D.L., and D.J. Funk, February 17, 2015. “Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes,” Los Alamos National Laboratory document LA-UR-15-22393, Los Alamos, New Mexico.

[3] Anast, K.R., November 2015. “Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing,” Los Alamos National Laboratory document LA-UR-15-28900, Los Alamos, New Mexico.

2.0 APPROACH

To evaluate the effectiveness of batch blending zeolite with RNS waste, the following steps were followed:

1. Prepare a surrogate RNS waste that can be tracked visually
2. Prepare a range of surrogate wastes that have moisture and salt contents that will bound the waste likely to be found in the 60 RNS drums
3. Conduct blending tests on varying recipes of waste, zeolite, and water
4. Determine the blending speed, duration of blend, volume of material, and ingredients that provide a well-blended zeolite RNS waste product and maximizes batch volume.

2.1 RNS Surrogate Formulation

Surrogate RNS waste was prepared from Swheat and sodium chloride rock salt. Figure 1 shows the zeolite, Swheat, rock salt, and dye used for all tests.

Figure 1. Samples of zeolite, Swheat, rock salt, and dye used for tests

Swheat was colored red with red food coloring by blending 1 volume of water, 1 capful of red food coloring, and 9 volumes of Swheat kitty litter. The resulting Swheat contained approximately 25% moisture and was a dry-appearing material. Figure 2 shows the Swheat after it was dyed red and combined with the rock salt. The red-dyed Swheat and purple rock salt were used in all batch-blending test work.

Figure 2. Sample of dyed Swheat and purple rock salt—surrogate RNS material

Rock salt that was dyed purple was purchased to prepare the RNS surrogate waste for batch-blending tests. The product used was Merlin Melts Like Magic (MMLM) and is shown in Figure 3.

Figure 3. Purple rock salt MMLM

Dyed Swheat and purple rock salt were then blended together in a Kitchen Aid blender by blending predetermined volumes of each for 1 min. Six blends were prepared and are detailed in Table 1 and shown in Figure 4.

RNS surrogate blends were prepared by blending various volumetric ratios of ingredients together to produce material that would bound the physical properties of the RNS waste likely to be found in the candidate RNS drums. Moisture content and salt loading are the two primary variables that were adjusted. A dry product simulates the salt/Swheat material, and the wet material simulates Swheat that absorbed liquid found in the UNS parent drum.

Table 1
RNS Surrogate Waste Formulations

Surrogate	Swheat:Water:Salt (vol. ratio)
Dry/Low Salt	9:1:2
Dry/High Salt	9:1:4.5
Moist/Low Salt	3:1:1
Moist/High Salt	3:1:1.5
Wet/Low Salt	2:1:0.5
Wet/High Salt	2:1:1

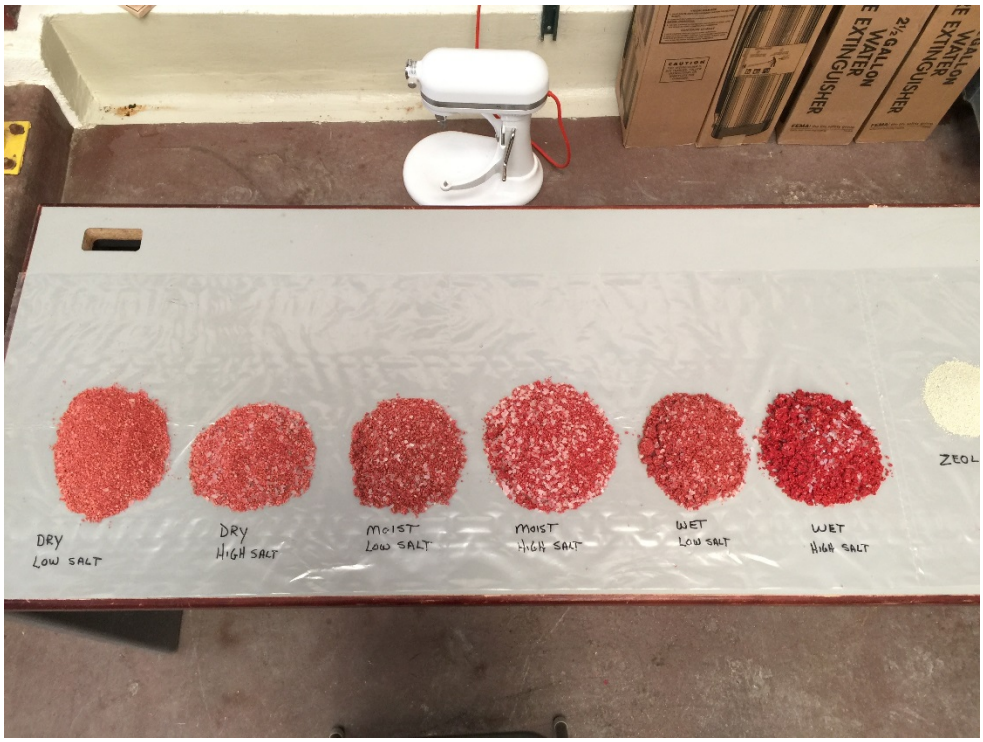

Figure 4. Surrogate waste formulations for zeolite-blend testing

2.2 RNS Processing Approach

Batch blending of RNS waste with zeolite occurs in a three-step process.

1. Blend RNS waste with water to achieve a soupy consistency that will more easily blend into zeolite and dissolve the available salt so it can be absorbed by zeolite
2. Add zeolite to the slurry to absorb any free liquid remediate the RNS waste
3. Blend until the ingredients are a homogenous mix

2.3 RNS Surrogate Blending Tests

The batch-blending tests were carried out using an 8-qt Kitchen Aid blender, the largest commercially available model that fits into the Waste Characterization Reduction and Repackaging Facility (WCRRF) glovebox and is easily lifted and set in place. The parts exposed to liquid are stainless steel, which is the material of choice[4] for nitric acid environment, which will accompany the RNS waste once dissolved. The blender is shown in Figure 5. The WCRRF glovebox will accommodate at least two blenders.

Figure 5. Kitchen Aid Blender

Surrogate RNS waste prepared with dyed Swheat and purple rock salt was blended with zeolite and water. A suite of tests was conducted to address a number of questions:

- The volume of water required (as a ratio of RNS waste surrogate) to achieve an acceptable consistency
- The blending time required to dissolve salt before zeolite is added
- The effectiveness of the blender at producing a homogenized product with Swheat well distributed throughout the zeolite matrix, independent of the RNS surrogate blend
- The speed of the blender and duration required for Step 1 and Step 3
- The ingredients for the recipe
- The ease of operation and manipulation for the glovebox configuration

2.3.1 Step 1: RNS Waste Dissolution Step

RNS waste dissolution includes combining water with RNS waste to achieve a soupy consistency to optimize blending the Swheat and salt into the zeolite and dissolve the salt. To evaluate this step, the

[4] Fontana, M.G., and N.D. Greene, 1967. Corrosion Engineering, McGraw-Hill Book Company, New York, New York.

dry/high-salt surrogate was used to determine the amount of water and blending time necessary to dissolve the salt. The surrogate salt, MMLM, contains 80% sodium chloride, with the remaining 20% a blend of potassium chloride, magnesium chloride, and calcium magnesium acetate. MMLM is less soluble than any of the major nitrate salts found in the RNS waste material. See Table 2.

The estimated volume of water required for salt dissolution per liter of waste is presented in Table 2. (Approximately 590 g of SWRI surrogate salt is found in a liter of surrogate RNS waste blended at a 2:1 volume ratio of Swheat to salt.) Table 2 compares the amount of water required to dissolve the salt found in 1 L of RNS waste at three different volume ratios of Swheat to salt. The amount of water required relates both to the solubility of the salt and the density because the Swheat:salt ratio is volumetric. Based upon the estimated ratio of each nitrate salt expected in the RNS drums[5], the water required for complete dissolution of the RNS salts is 0.79 L for a 2:1 volumetric Swheat:salt ratio and 0.64 L for a 3:1 Swheat:salt volumetric ratio.

Because nitrate salts require less water for dissolution compared with the surrogate sodium chloride, the tests will focus on both salt dissolution and consistency of the slurry. Therefore, the time required to dissolve sodium chloride in the surrogate samples should provide an indication of the required mix time for RNS waste material. Tests at SWRI will provide guidance on the effectiveness of dissolving nitrate salts during this step.

Table 2
Solubility of Various Salts in Water and Volume of Water Required to Dissolve Salt in Various RNS Salt/Swheat Blends

	Pure Salt	**Water Required to Dissolve Salt in RNS**		
Salt	Solubility@ 25 °C (g/L)	1:1 Swheat:salt (L H_2O/L Waste)	2:1 Swheat:salt (L H_2O/L Waste)	3:1 Swheat:salt (L H_2O/L Waste)
MMLM	370 *(2.16)**	2.91	1.94	1.46
SWRI Surrogate Salt Blend	684 *(1.76)**	1.05	0.79	0.64
$Mg(NO_3)_2 * 6H_2O$ (22%)	625	1.07	0.71	0.53
$Ca(NO_3)_2 * 4H_2O$ (5%)	1290	0.78	0.52	0.39
$NaNO_3$ (3%)	912	1.25	0.83	0.62
$Fe(NO_3)_3 * 9H_2O$ (3%)	1500	0.56	0.37	0.28
$Al(NO_3)_3 * 9H_2O$ (46%)	673	1.28	0.85	0.64
$Pb(NO_3)_2$ (3%)	565	4.00	2.67	2.00

* = Density of salt

Six tests were completed using the dry/high salt surrogate to determine the impact of water volume, mixing speed, mixing duration, and water temperature on salt dissolution. The test conditions and resulting time required to dissolve the contained salt are presented in Table 3. A surrogate-to-water volume ratio of 1 resulted in a very thin, runny consistency that allowed the salt to dissolve fairly quickly but resulted in excessive liquid for the zeolite to absorb, an effect that becomes worse with a wetter surrogate. Surrogate-to-water ratios of 0.5 and 0.75 provided adequate liquid for most of the salt to dissolve, but as the volume approached the 0.5 ratio, the dissolution time became longer and the consistency of the mix was thicker. The mixing speed seemed to have some impact on the dissolution

[5] Source: Table 1: WB8 surrogate from SWRI Exhibit D for the SWRI contract.

rate, but using a faster speed resulted in splashing of the contents. Water temperature had an expected impact of reducing the dissolution time. The final test examined the impact of the wet/high salt surrogate on salt dissolution. The dissolution time was similar to the dry/high salt surrogate; however, the resulting mix was of a thinner consistency.

The following conditions are recommended for the RNS waste dissolution step:

- RNS waste-to-water volume ratio: 1:0.65
- Mixer speed: 4 (with the addition of a lid during mixing)
- Blend duration: 3 min

Table 3
RNS Waste Dissolution Step Tests

Surrogate Type	Surrogate:Water Volume Ratio	Blend speed	Water Temp ^{0}C	Time (min) Salt Dissolution
Dry/High Salt	1:1	3	10	**2.5**
Dry/high Salt	1:0.75	3	10	**3.0**
Dry High Salt	1:0.5	3	10	**3.5**
Dry high Salt	1:0.66	4	10	**3.0**
Dry High Salt	1:0.66	4	37	**2.0**
Wet/High Salt	1: 066	4	37	**2.0**

2.3.2 Steps 2 and 3 Tests: Addition of Zeolite

Zeolite is added to absorb water, provide an inorganic matrix to hold the nitrate salts, and insulate the Swheat from interacting with available oxidizing salts. The zeolite used for these tests is from KMI Zeolite[6], located in Sandy Valley, Nevada, and is 14 × 40-mesh size. KMI zeolite is the same zeolite used in the blending tests at SWRI. Zeolite must be well mixed with the Swheat to effectively stabilize the waste and ensure it is no longer ignitable. The red-dyed Swheat provides a visual means to evaluate the effectiveness of producing a well-blended, homogeneous product.

The amount of zeolite required on a batch basis has not been determined yet. Previous test work at Energetic Materials Research and Testing Center in Socorro, New Mexico, indicates that nitrate salts can effectively be treated using a 1.2:1 volume ratio of zeolite-to-nitrate salts[7] and zeolite is effective at mitigating the oxidizer properties of nitrates in waste surrogates. SWRI is currently engaged in test work that will provide additional guidance on the amount of zeolite required to ensure the RNS waste is no longer ignitable. SWRI blends samples by hand, mixing the salt/Swheat surrogate into the zeolite using a spatula. Use of the Kitchen Aid mixer provides a more consistent blending approach and will be used in the final SWRI process verification tests.

Table 4 presents the details of the 12 tests conducted, including the impact blend duration, blend speed, RNS surrogate characteristics, and zeolite volume on blending performance. The recommended

[6] KMI Zeolite is typically 97%+ pure clinoptilolite zeolite by weight.

[7] Walsh, G., March 2010. “Results of Oxidizing Solids Testing,” Energetic Materials Research and Training Center Report FR 10-13, New Mexico Bureau of Mines & Mineral Resources, New Mexico Institute of Mining and Technology, Socorro, New Mexico.

conditions identified from Step 1 testing were used for all of the zeolite addition tests. The zeolite addition test conditions and resulting blend performance notes are shown in Table 4.

Step 1 conditions for all zeolite addition tests:

- RNS waste-to-water volume ratio: 1:0.65
  - 1 qt of surrogate waste
  - 0.65 qt of water
- Mixer speed setting: 4
- Blend duration: 3 min

Table 4
Zeolite Addition Tests (Steps 2 & 3) Tests

Blend #	Surrogate Waste Type	Zeolite (qt)	Blend Duration (min)	Comments/Blend Speed
Moist 1	Moist/Low Salt	3	1.0	Added zeolite all at once and maintained speed @ 3 Material was well blended and had a layer of buildup on bottom/sides of bowl
Moist 2	Moist/Low Salt	2	1.0	Added zeolite all at once and maintained speed @ 3 Material was well blended and had a layer of buildup on bottom/sides of bowl
Moist 3	Moist/Low Salt	4	1.0	Added zeolite all at once and maintained speed @ 3 Material was well blended and had a layer of buildup on bottom/sides of bowl
Moist 4	Moist/Low Salt	3	1.0	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 1 min Material was well blended and no Swheat build up on bottom
Dry 5	Dry/High Salt	3	1.0	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 1 min Material was well blended and no Swheat buildup on bottom
Wet 6	Wet/High Salt	3	1.0	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 1 min Material was well blended with no Swheat buildup on bottom
Moist 7	Moist/High Salt	2	1.0	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 1 min Material was well blended with no Swheat buildup on bottom

Table 4 (continued)

Blend #	Surrogate Waste Type	Zeolite (Qt)	Blend Duration (min)	Comments/Blend Speed
Dry 8	Dry/High Salt	3	0.5	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 30 s Material was well blended with no Swheat buildup on bottom
Wet 9	Wet/Low Salt	4	0.5	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 30 sec Material was well blended with no Swheat buildup on bottom
Vol 10	Dry/High	4.4	0.5[a]	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 30 s Material was well blended with no Swheat buildup on bottom
Vol 11	Wet/Low	4.4	0.5[a]	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 30 s Material was well blended with no Swheat buildup on bottom
Vol 12	Moist/High	4.4	0.5[a]	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 30 s Material was well blended with no Swheat build up on bottom
WL[b] 13	WL	4.4	0.5[a]	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 30 s Material was well blended with layer of surrogate buildup on bottom
WL[b] 14	WL	4.4	0.5[a]	Added ½ zeolite and ran speed at 8 for 10 s, then added remaining zeolite and blended at 3 for 30 s Material was well blended with layer of surrogate buildup on bottom

[a] Maximized volume in mixing bowl

[b] WL = Waste Lock 770

Initial tests (Moist 1–3) with moist/low salt surrogate waste produced a well-blended product after the zeolite was added all at once. Zeolite addition ratios of 2:1 to 4:1 (zeolite:RNS waste surrogate) by volume were evaluated. Blending for a minute on speed setting 3 effectively blended the Swheat into the zeolite matrix for the first three tests. The amount of zeolite added did not have a noticeable impact on the effectiveness of mixing the Swheat and salt into the zeolite matrix. When the bowl was emptied, an approximately 0.25-in. layer was stuck on the bottom third of the bowl, did not appear to be blended, and was primarily red Swheat.

Two changes were made for the fourth test (Moist 4) zeolite was added in two steps: half was added and the mix speed was raised to speed setting 8 for 10 seconds, then reset to speed setting 3 and the remaining zeolite was added. The change was effective at eliminating the bottom layer. When material adhered to the side, it was well blended when it was dumped, not a layer of pure Swheat.

Conditions for Moist 4 were then used on dry and wet surrogates. Tests Dry 5 and Wet 6 produced a well-blended product and were blended under the same conditions as Moist 4. A Swheat layer was not found on the bottom of the bowl for either test.

Test Dry 8 and Wet 9 evaluated a shorter blend time for the zeolite addition. These tests had mix times of 30 s for zeolite, once all of the zeolite was added.

Tests Vol 10 and Vol 12 focused on maximizing the available volume in the bowl. The bowl has maximum capacity of 8-qt, and a 6-qt batch size was used for these tests. A range of RNS surrogates were used to verify performance with the larger volume. The surrogate used in the tests did impact the overall moistness of the final product, but the resulting material was well blended and the zeolite addition method eliminated layering of the Swheat on the bottom of the bowl.

Step 1 conditions for zeolite addition tests: (Vol 10 through Vol 12)

- RNS waste-to-water volume ratio: 1:0.65
    - 1.75 qt of surrogate waste
    - 1.15 qt of water
- Mixer speed setting: 4
- Blend duration: 3 min

Figure 6 shows product from Vol 10 test. The red surrogate Swheat material appears well blended into the zeolite matrix and no salt crystals are visible, although upon very close examination small, fine particles can be identified.

Figure 6. Final blended product from Test Vol 10

Two tests were performed to examine blending of salt coated with Waste Lock 770. Waste Lock 770 is found in three drums. Surrogate material was prepared by mixing 1 volume of salt with 0.1 volume of water and 3 volumes of Waste Lock 770.

The same Step 1 recipe and conditions used for tests Vol 10 and Vol 12 were followed for the Waste Lock 770 tests, WL 13 and WL 14. The surrogate Waste Lock 770 material produced after Step 1 is shown in Figure 7. The surrogate waste was gelatinous, and it was difficult to determine salt dissolution. The gelatinous material provided a wet coating around the salt crystals.

Figure 7 Surrogate Waste Lock 770 and salt (3:1 volume ratio)

The zeolite mixed well with the gelatinous Waste Lock 770 surrogate material produced in Step1. A total of 4.4 qt of zeolite was added and blended in a similar manner as previous tests. The product shown in Figure 8 is well blended, fluffy, and drier in appearance than comparable samples of product that contains Swheat. The mixer bowl was nearly full, but the contents did not spill out during the 30-s blend. When the bowl was emptied, a layer of material was observed at the bottom of the bowl that was primarily the white surrogate waste material. Further testing with Waste Lock 770 continued to result in a thin layer of unmixed material at the bottom of the bowl.

Figure 8 Waste Lock 770 surrogate waste blended with zeolite (Test WL 13)

3.0 CONCLUSIONS/RECOMMENDATIONS

- The Kitchen Aid 8-qt model blender effectively blends a range of surrogate Swheat/salt and Waste Lock 770/salt material into the zeolite.
- The effectiveness of the blending is not related to the wetness of the materials or the salt content of the surrogates at the blend ratios tested.
- Dissolution of salt into water requires about 3 min, and the amount of water is related to the amount and type of salt found in the waste. The amount of water and required mix time will be determined after SWRI tests results are reviewed.
- A three-step approach was effective at dissolving most of the surrogate salt material and blending the resulting Swheat or Waste Lock 770 into the zeolite.
- Optimal blend ratios pending results from SWRI are
    1. RNS waste-to-water volume minimum ratio: 1:0.65
    2. RNS waste-to-zeolite volume minimum ratio: 1:2.5
- The recommended RNS waste process steps, pending SWRI tests results, are as follows:
    1. Add 1.15 qt warm (~100 ^{o}F) water to bowl
    2. Add 1.75 qt RNS waste
    3. Blend on speed setting 4 for 3 min
    4. Add 2.20 qt of zeolite
    5. Blend on speed setting 8 for 10 s
    6. Reduce speed to setting 3
    7. Add 2.20 qt of zeolite
    8. Blend for 30 s
- Filling the bowl with 6 qt of material results in material spilling over the sides of the bowl. Three bowl modifications are recommended:
    1. Modify the bowl to include a wider diameter rim around the top
    2. Fabricate a cover that can be easily attached and removed to prevent material from spilling over the side of the bowl during blending
    3. Lead-line the exterior of the bowl and the lid to limit radiation exposure to workers

- Volumetric containers for water and RNS waste should be designed and fabricated once the final optimum recipe has been tested at SWRI. The containers should

  1. be fabricated from a denser material or have lead lining for RNS material and have lids that will help reduce the radiation exposure to workers, and

  2. be of a specific volume to eliminate the potential to incorrectly measure ingredients for blending

- Zeolite should be premeasured according to the volume required for each batch and placed in bags that could be loaded into the daughter drum for feeding the operation in the glovebox. As the RNS waste is blended, the bags can be used for adding zeolite to the blending operation.

  Full-scale testing of the process should be done in a mock-up glovebox that simulates the WCRRF glovebox and use waste drums that simulate the RNS drum configuration.

ENCLOSURE 3

Los Alamos National Laboratory Nitrate Salt-Bearing Waste
Treatment Planning Schedule

ADESH-16-043

LA-UR-16-21587

Date: MAR 2 1 2016

Los Alamos National Laboratory Nitrate Salt-Bearing Waste Treatment Planning Schedule

1. Introduction and Background

This document responds to Ordered Action 2/3, Item C in Attachment A of the Settlement Agreement and Stipulated Final Order HWB-14-20 entered into by the New Mexico Environment Department (NMED) and the U.S. Department of Energy (DOE) and Los Alamos National Security, LLC (LANS) (collectively the Respondents) on January 22, 2016. This plan outlines the steps that need to be completed by the Respondents prior to requesting a permit modification to treat nitrate salt-bearing waste that is currently stored at the Los Alamos National Laboratory (LANL).

A transuranic mixed waste container generated and processed at LANL was determined to be the container from which the February 14, 2014 incident in the underground repository at the Waste Isolation Pilot Plant (WIPP) originated. The Respondents have identified waste containers located at LANL that are similar to the waste type that was the cause of the incident at the WIPP. These nitrate salt-bearing wastes are addressed under the Settlement Agreement and Stipulated Final Order HWB-14-20, and the Respondents are required to determine a safe handling and treatment plan for nitrate salt wastes.

These wastes can be generally described in two categories: 1) remediated nitrate salt-bearing wastes; and 2) unremediated nitrate salt wastes. “Remediated” containers are defined as LANL unconsolidated nitrate salts that were remediated with an organic absorbent and were repackaged into new waste containers. “Unremediated” containers are defined as LANL unconsolidated nitrate salts drums to which absorbent material has not been added. Other waste types that are determined to contain nitrate salts will be treated utilizing similar treatment processes as those determined to be effective for remediated and unremediated nitrate salt-bearing waste containers.

In order to determine treatment methods for the nitrate salt-bearing waste streams and develop a plan for Items C.i through C.iv of Ordered Action 2/3, the Respondents must accomplish the following:

1. Determine the properties of nitrate salt-bearing waste containers and ensure that waste characterization for the waste is complete. This includes the Resppondent’s currentintention to not sample unremediated nitrate salt waste containers as proposed in the sampling plan listed as Item C.i of Ordered Action 2/3 and linked within Enclosure 2 of this response submittal.
2. Test the proposed treatment technology for effectiveness at removing applicable Resource Conservation and Recovery Act (RCRA) hazardous waste characteristics. This determination requires a plan for surrogate testing and a report on surrogate testing results (Items C.ii and C.iii of Ordered Action 2/3).
3. Determine the location and the methods that will be used to physically conduct the treatment process(es).
4. Develop a plan to safely manage and treat nitrate salt-bearing waste as listed as Item C.iv of Ordered Action 2/3.

This plan outlines the Respondents' schedule for conducting, and the plan for documenting, the aforementioned tasks as part of the response actions listed in Attachment A of the Settlement Agreement and Stipulated Final Order HWB-14-20, and as part of the important process to develop methods for treatment of nitrate salt-bearing waste. The schedule for submittal of document deliverables is included in Table 1.

2. Waste Characterization Determination

The initial step for the Respondents to determine treatment methods for the nitrate salt waste streams was to ensure that waste characterization for the waste was complete. Waste re-characterization for the nitrate salt-bearing waste streams began immediately after the determination that the waste container at the WIPP was generated at LANL. These efforts have been documented in several previous submittals to the NMED including:

1. *Addendum to the Los Alamos National Laboratory Hazardous Waste Facility Permit Reporting on Instances of Noncompliance and Releases for Fiscal Years 2012 And 2013* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ERID-257845),
2. *Transmittal of Waste Characterization Documentation for Nitrate Salt-Bearing Waste Containers* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ERID-260195),
3. *Response to Information Request Regarding the Los Alamos National Laboratory Nitrate Salt Bearing Waste Container Isolation Plan* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ERID-260905),
4. *Response to LANL Nitrate Salt-Bearing Waste Container Isolation Plan* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ERID-261850),
5. *Second Addendum, Reporting Additional Instances of Noncompliance with Hazardous Waste Facility Permit and Generator Requirements, Los Alamos National Laboratory* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ERID-262519),
6. *Chemical Reactivity and Recommended Remediation Strategy For Los Alamos Remediated Nitrate Salt (RNS) Wastes* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600350),
7. *Self-Disclosure of Non-Compliances Resulting From the Extent of Condition Review, Los Alamos National Laboratory Hazardous Waste Facility Permit No. NM0890010515* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600898), and
8. *Sampling and Analysis Information for LA-CIN01 Waste Containers Los Alamos National Laboratory* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-601010).

Based on studies and re-characterization efforts, surrogates were developed for nitrate salt-bearing wastes. The recipes for these surrogates and description for the development of the surrogate wastes is discussed in *Remediated Nitrate Salt Surrogate Formulation, Aging, and Testing Procedure*, Appendix 1 in Enclosure 2. These developed surrogates are the media being utilized for testing treatment effectiveness as outlined in Section 3.

Additionally, the Respondents previously determined that sampling and LANL-internal analysis of unremediated nitrate salt waste was necessary to fully determine that the surrogates developed were bounding of the waste streams. Since the development and submittal of the *Sampling and Analysis Plan, Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory* (http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600920), it has been determined

that sampling and analysis of unremediated nitrate salt waste is not necessary for characterization efforts because the surrogates developed are bounding in that:

1. The material has sufficient chemical potential (energy content) that would allow for the resulting condition observed within the mine at the WIPP. This criterion can be simplified to: contains sufficient concentration of oxidizers as mixed with the fuel to create an ignitable hazard.
2. The material has a rate of reactivity (rate of energy release) that would enable thermal runway as quickly as or sooner than the most unstable remediated nitrate salt-bearing waste container (drum 68660). That is, the heat generation rate exceeds the heat dissipation rate, accelerating the reactivity, ultimately enabling the pressurization of a container and causing it to breach.
3. The chemical constituents are consistent with the major components expected for the waste form.

At this time, unremediated nitrate salt waste sampling and analysis is not scheduled to be performed as part of the development of a treatment method. If it is determined that sampling of these waste containers must be conducted, the schedule will have to be revised to accommodate this activity. Additionally, as part of evaluating surrogate materials, evaluation and extensive modeling of surrogate kinetics has been conducted to demonstrate the likelihood of runaway after 650 days in storage has become exceedingly small. Results of these evaluations will be provided to the NMED upon completion as shown within the schedule in Table 1.

Overall, nitrate salt-bearing wastes have been determined to contain RCRA hazardous wastes characteristic for ignitability and corrosivity (where liquids are present) and retain the U.S. Environmental Protection Agency (EPA) Hazardous Waste Numbers for these characteristics—D001 and D002, respectively. While other EPA Hazardous Waste Numbers have been assigned to these waste streams, only D001 and D002 cannot be accepted at the WIPP. Therefore, these characteristics must be treated and removed prior to shipment to the WIPP for disposal.

3. Treatment Effectiveness Testing

The *Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory*, (included as Appendix 3 of Enclosure 2), is an analysis that documents the original methodology used to select a treatment method for remediated and unremediated nitrate salt-bearing waste. The document concludes that cooling of remediated nitrate salt-bearing waste containers and stabilization using zeolite, for both remediated and unremediated nitrate salt waste would be the most effective treatment method for implementation at LANL. Cementation of the waste would be a second option.

The Respondents have developed surrogates, and testing of those surrogate wastes was determined to be the most effective method for proving treatment effectiveness for nitrate salt-bearing waste streams. Surrogates without radioactive properties are being utilized to eliminate the risk to personnel due to the radiological hazards presented by the nitrate salt-bearing wastes. Additionally, a method to ship samples of the actual waste material for testing off-site has not been developed at this time. Various treatment effectiveness tests that include surrogate materials mixed with zeolite and cementation are being conducted by the Respondents internally at LANL, as well as at an off-site analytical laboratory using EPA-approved test methods to ensure that the characteristics of ignitability and corrosivity are removed from the waste prior to repackaging.

The Respondents developed a plan for testing of surrogates and have augmented the testing as additional questions/concerns were identified. The *Treatment Study Plan for Nitrate Salt Waste Remediation*, (included as Appendix 5 of Enclosure 2), is a plan that outlines the use of sensitivity testing being conducted by the Respondents, and off-site testing of treatment processes that are designed to ensure the removal of D001 and D002 from the nitrate salt-bearing waste. The testing has been augmented as the Respondents have identified new information about nitrate salt waste during testing conducted at LANL. Testing on surrogate waste materials is still underway, both onsite and off-site.

A summary of results for all surrogate testing will be drafted upon completion of all of the required testing and is on the schedule in Table 1. The Respondents anticipate proving that the addition of zeolite to nitrate salt-bearing waste to be an effective treatment method for the removal of the EPA Hazardous Waste Numbers for ignitability and corrosivity (D001 and D002).

4. Location and Treatment Planning

Treatment processes have been evaluated to assess available systems and facilities considered for carrying out these activities on remediated nitrate salt-bearing and unremediated nitrate salt wastes at LANL. *Engineering Options Assessment Report: Nitrate Salt Waste Stream Processing*, included as Appendix 8 of Enclosure 2, documents the Respondents' assessment of potential locations for implementation of the preferred treatment options. This report includes recommendations for additional studies being planned regarding specific equipment to be utilized during the treatment, and recommends additional studies regarding debris waste that is located within remediated nitrate salt containers. The report also recommends that the glovebox within the storage unit at TA-50-69 (known as the Waste Characterization Reduction and Repackaging Facility [WCRRF]) be utilized for zeolite blending treatment.

Additional evaluation and testing has been completed to determine the blending techniques for batch blending of nitrate salt-bearing waste with water and zeolite utilizing blenders and mixers within the glovebox at TA-50-69 (WCRRF). The report regarding the completed evaluation is included within Enclosure 2 as Appendix 8, *Engineered Option Treatment of Remediated Nitrate Salts: Surrogate Batch-Blending Testing*. The Respondents anticipate that cooling of remediated nitrate salt-bearing waste containers will occur at the permitted storage unit at TA-54, Area G, Dome 375 and that both remediated and unremediated nitrate salt-bearing wastes are expected to be treated by blending with water and zeolite within the glovebox at the permitted storage unit at TA-50-69 (WCRRF).

5. Safe Handling and Treatment Plan

The permit modification request for the LANL Hazardous Waste Facility Permit will include a plan for safely moving the remediated nitrate salt-bearing waste containers out of isolated storage in accordance with the most current *LANL Nitrate Salt-Bearing Waste Container Isolation Plan*, as well as the request to add treatment process(es) at the permitted unit at TA-50-69 (WCRRF). The treatment process(es) will allow both remediated and unremediated nitrate salt-bearing waste to be treated by blending with water and zeolite. The permit modification request will be submitted to the NMED for review and approval upon completion, and is included on the schedule in Table 1.

Table 1. List of Proposed Deliverable Documents and Schedule

Description of Document	Date Due/ Date Delivered	Location
Sampling and Analysis Plan for Unremediated Nitrate Salt Waste Containers at Los Alamos National Laboratory	September 17, 2015	http://permalink.lanl.gov/object/tr?what=info:lanl-repo/eprr/ESHID-600920
Surrogate waste testing plan	Included with this submittal	*Treatment Study Plan for Nitrate Salt Waste Remediation Revision 1.0*, Enclosure 2, Appendix 5
Evaluation of the potential for thermal runaway of nitrate salt containers in storage	April 30, 2016	
Report on surrogate waste tests	May 16, 2016	
Safe handling and treatment plan for both remediated and unremediated nitrate salt wastes (i.e. draft permit modification request)	May 16, 2016 (draft) July 1, 2016 (final)	