Los Alamos
NATIONAL LABORATORY
EST 1943

ESHID-601477

Environment Safety & Health
PO Box 1663, MS K491
Los Alamos, New Mexico 87545
(505) 667-4218/Fax (505) 665-3811

National Nuclear Security Administration
Los Alamos Field Office, A316
3747 West Jemez Road
Los Alamos, New Mexico, 87544
(505) 667-5105/Fax (505) 667-5948

Date: MAY 1 3 2016
Symbol: ADESH-16-076
LA-UR: 16-23150, 16-22729 and 16-22658
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

Dear Mr. Kieling:

Subject: Transmittal of Reports Regarding Treatment Effectiveness for Stabilization of Nitrate Salt Waste Streams

The purpose of this letter is to transmit summary reports that the Los Alamos National Security, LLC (LANS) and the U.S. Department of Energy (DOE), the Permittees, committed to providing the New Mexico Environment Department (NMED) in a March 21, 2016 submittal. Enclosure 3 of the Permittees' *Response to Ordered Action 2/3, Attachment A to Settlement Agreement and Stipulated Final Order HWB-14-20, Los Alamos National Laboratory*, included a list of proposed documents and a schedule for submittal of those documents.

Attachment A to the Settlement Agreement and Stipulated Final Order HWB-14-20 required "Final Report on Surrogate Waste Tests (Final Title TBD)". Attachment A also noted that this report will, "include UNS and SWERI analytical results." Since the drafting of Attachment A, the Permittees plan for testing of surrogate materials and the necessity of collecting actual waste samples has changed. Therefore, the documentation that will be provided as evidence of completion has changed. The Permittees have determined that sampling of unremediated nitrate salt (UNS) waste is no longer necessary for characterization activities, therefore, analytical results for UNS waste will not be available prior to the treatment of the waste. Additionally, the Permittees have expanded surrogate testing and some testing is still underway, consequently, not all reports can be provided at this time. Lastly, the Permittees have concluded that a single summary of both onsite and off-site conducted testing would lead to complicating the intent of each of the tests, so summary reports for each of the projects has been provided herein.

These modifications to the Permittees plan for a path forward on surrogate testing add deliverable documents to the schedule included as Enclosure 3 of the Permittees' *Response to Ordered Action 2/3, Attachment A to Settlement Agreement and Stipulated Final Order HWB-14-20, Los Alamos National Laboratory*. The Permittees' request a change to the schedule to allow for multiple reports on surrogate waste testing to be provided to the NMED. The first set of reports are attached to this letter, and a second set of reports on surrogate waste testing will be provided by July 1, 2016.

Enclosures 1, 2, and 3 of this letter are summary reports regarding surrogate waste testing conducted by the Permittees. Additional testing is still underway, and a summary of those tests will be provided to the NMED when available and prior to July 1, 2016. Enclosures 1 and 2 include descriptions of tests, and summaries of results of those tests, conducted by the Permittees onsite. Enclosure 3 summarizes information of surrogate testing conducted at an off-site analytical laboratory. The summary reports have been included within the enclosures, and the appendices to the reports have been included on the compact disc included with this submittal.

If you have comments or questions regarding this submittal, please contact Mark P. Haagenstad (LANS) at (505) 665-2014 or Jordan Arnswald (DOE) at (505) 667-6764.

Sincerely,

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

Sincerely,

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

MTB:KDL:MPH:LRVH/lm

Enclosures: (1) Summary of Remediated Nitrate Salt Surrogate Formulation and Testing
(2) Initial Results from the Third Round of Remediated Nitrate Salt Surrogate Formulation and Testing
(3) Summary Report of Laboratory Testing to Establish the Effectiveness of Proposed Treatment Methods for Unremediated and Remediated Nitrate Salt Waste Streams

Cy: Ryan Flynn, NMED, Santa Fe, NM, (E-File)
Kathryn M. Roberts, NMED, Santa Fe, NM, (E-File)
Siona Briley, NMED/HWB, Santa Fe, NM, (E-File)
Neelam Dhawan, NMED/HWB, Santa Fe, NM, (E-File)
Todd Shrader, Manager, CBFO, (E-File)
J.R. Stroble, National TRU Program, CBFO, (E-File)
Kimberly Davis Lebak, NA-LA, (E-File)
Douglas E. Hintze, EM-LA, (E-File)
Jody M. Pugh, NA-LA, (E-File)

Cy (continued):
Peter Maggiore, NA-LA, (E-File)
Lisa Cummings, NA-LA, (E-File)
David Nickless, EM-LA, (E-File)
Jordan Arnswald, NA-LA, (E-File)
Kirsten M. Laskey, EM-LA, (E-File)
Craig S. Leasure, PADOPS, (E-File)
William R. Mairson, PADOPS, (E-File)
Randall M. Erickson, ADEM, (E-File)
David Funk, ADEM, (E-File)
Enrique Torres, ADEM, (E-File)
Cheryl D. Cabbil, ADNHHO, (E-File)
Michael T. Brandt, ADESH, (E-File)
Raeanna Sharp-Geiger, ADESH, (E-File)
John P. McCann, EPC-DO, (E-File)
David E. Frederici, WMD, (E-File)
Mark P. Haagenstad, EPC-CP, (E-File)
Deborah Woitte, LC-ESH, (E-File)
Susan McMichael, LC-ESH, (E-File)
Deborah L. Guffee, SI-DC, (E-File)
Yvette S. Branch, SI-DC, (E-File)
Luciana Vigil-Holterman, EPC-CP, (E-File)
lasomailbox@nnsa.doe.gov, (E-File)
emla.docs@em.doe.gov, (E-File
locatesteam@lanl.gov, (E-File)
epc-correspondence@lanl.gov, (E-File)
rcra-prr@lanl.gov, (E-File)
epccat@lanl.gov, (E-File)
adesh@lanl.gov, (E-File)

Los Alamos NATIONAL LABORATORY EST 1943

COPY

Environment Safety & Health
PO Box 1663, MS K491
Los Alamos, New Mexico 87545
(505) 667-4218/Fax (505) 665-3811

National Nuclear Security Administration
Los Alamos Field Office, A316
3747 West Jemez Road
Los Alamos, New Mexico, 87544
(505) 667-5105/Fax (505) 667-5948

Date: MAY 1 3 2016
Symbol: ADESH-16-076
LA-UR: 16-23150, 16-22729 and 16-22658
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

Dear Mr. Kieling:

Subject: Transmittal of Reports Regarding Treatment Effectiveness for Stabilization of Nitrate Salt Waste Streams

The purpose of this letter is to transmit summary reports that the Los Alamos National Security, LLC (LANS) and the U.S. Department of Energy (DOE), the Permittees, committed to providing the New Mexico Environment Department (NMED) in a March 21, 2016 submittal. Enclosure 3 of the Permittees' *Response to Ordered Action 2/3, Attachment A to Settlement Agreement and Stipulated Final Order HWB-14-20, Los Alamos National Laboratory*, included a list of proposed documents and a schedule for submittal of those documents.

Attachment A to the Settlement Agreement and Stipulated Final Order HWB-14-20 required "Final Report on Surrogate Waste Tests (Final Title TBD)". Attachment A also noted that this report will, "include UNS and SWERI analytical results." Since the drafting of Attachment A, the Permittees plan for testing of surrogate materials and the necessity of collecting actual waste samples has changed. Therefore, the documentation that will be provided as evidence of completion has changed. The Permittees have determined that sampling of unremediated nitrate salt (UNS) waste is no longer necessary for characterization activities, therefore, analytical results for UNS waste will not be available prior to the treatment of the waste. Additionally, the Permittees have expanded surrogate testing and some testing is still underway, consequently, not all reports can be provided at this time. Lastly, the Permittees have concluded that a single summary of both onsite and off-site conducted testing would lead to complicating the intent of each of the tests, so summary reports for each of the projects has been provided herein.

ENCLOSURE 1

Summary of Remediated Nitrate Salt Surrogate Formulation and Testing

ADESH-16-076

LA-UR-16-23150

Date: MAY 1 3 2016

LA-UR-16-23150
Approved for public release; distribution is unlimited.

Title: Summary of Remediated Nitrate Salt Surrogate Formulation and Testing

Author(s): Brown, Geoffrey Wayne
Leonard, Philip
Hartline, Ernest Leon
Tian, Hongzhao

Intended for: Report

Issued: 2016-05-05

Los Alamos
NATIONAL LABORATORY
memorandum

M-7: High Explosive Science and Technology

To/MS: David J. Funk, ADEP, djf@lanl.gov
From/MS: Geoffrey W. Brown, M-7, geoffb@lanl.gov
Phone/Fax: 7-6718/7-0500
Symbol: M7-16-6063
Date: May 3, 2016

SUBJECT: Summary of Remediated Nitrate Salt Surrogate Formulation and Testing

Contributors: Geoff Brown, Phil Leonard, Ernie Hartline, Hongzhao Tian (M-7)

Introduction

High Explosives Science and Technology (M-7) completed all required formulation and testing of Remediated Nitrate Salt (RNS) surrogates on April 27, 2016 as specified in PLAN-TA9-2443 Rev B, "Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing Standard Procedure", released February 16, 2016. This report summarizes the results of the work and also includes additional documentation required in that test plan. All formulation and testing was carried out according to PLAN-TA9-2443 Rev B. The work was carried out in three rounds, with the full matrix of samples formulated and tested in each round. Results from the first round of formulation and testing were documented in memorandum M7-16-6042, "Results from First Round of Remediated Nitrate Salt Surrogate Formulation and Testing." Results from the second round of formulation and testing were documented in M7-16-6053, "Results from the Second Round of Remediated Nitrate Salt Surrogate Formulation and Testing." Initial results from the third round were documented in M7-16-6057, "Initial Results from the Third Round of Remediated Nitrate Salt Formulation and Testing."

Materials

The materials used to formulate the RNS surrogates are listed in the table below. All chemicals were ordered through IESL-approved vendors using the Oracle iProcurement system. The Certificates of Analysis and packing slips for each item were scanned and documented in memorandum M7-15-6033, "Starting Materials Available for RNS Surrogate Formulation."

Table 1. Materials used for formulating RNS surrogates

Material	Lot Number
Aluminum nitrate nonahydrate	142299
Calcium nitrate tetrahydrate	144946
Chromium nitrate nonahydrate	F04Y024
Iron nitrate nonahydrate	A0355097
Water, LCMS grade	145280
Magnesium nitrate	147856
Sodium nitrate	144821
Lead nitrate	143996
Oxalic acid	143866 and 145400
Potassium carbonate	145088

sWheat pet litter was obtained commercially by LANL Environmental Programs and a 20 lb bag was supplied to M-7 per PLAN-TA9-2443 Rev B.

Formulation

The base RNS surrogate component is designated SFWB-8 and consists of the mixture listed in Table 2.

Table 2. SFWB-8 composition

Material	Weight %
$Al(NO_3)_3 * 9\ H_20$	3.20
$Ca(NO_3)_2 * 4\ H_2O$	12.72
$Cr(NO_3)_3 * 9H_2O$	0.16
$Fe(NO_3)_3 * 9H_2O$	4.86
$Mg(NO_3)_2 * 6H_2O$	35.69
$NaNO_3$	7.91
$(COOH)_2 * 2H_20$	2.89
K_2CO_3	1.51
Water	4.31

The final RNS surrogate formulations consist of SFWB-8 with lead nitrate and sWheat pet litter in the weight ratios shown in Table 3. Table 3 is the matrix tested in each round noted above.

In the discussions below, the samples will be designated with a shorthand notation of the form SFWB8-XX-Y, where XX is the sWheat percentage and Y is the Pb percentage. For example, SFWB8-15-1 is the mixture made using SFWB-8 with 15% sWheat and 1% Pb.

Table 3. RNS surrogate formulation matrix. Weight percent values shown.

	Increasing sWheat ->		
Increasing Lead ->	4% $Pb(NO_3)_2$ 15% sWheat 81% WB-8	4% $Pb(NO_3)_2$ 25% sWheat 71% WB-8	4% $Pb(NO_3)_2$ 35% sWheat 61% WB-8
	2% $Pb(NO_3)_2$ 15% sWheat 83% WB-8	2% $Pb(NO_3)_2$ 25% sWheat 73% WB-8	2% $Pb(NO_3)_2$ 35% sWheat 63% WB-8
	1% $Pb(NO_3)_2$ 15% sWheat 84% WB-8	1% $Pb(NO_3)_2$ 25% sWheat 74% WB-8	1% $Pb(NO_3)_2$ 35% sWheat 64% WB-8

Formulation followed section 4.2 of PLAN-TA9-2443 Rev B with any relevant observations from the third round documented in the formulation notes in Appendix A of this report. Observations from previous rounds are documented in the memoranda noted above.

Testing

Details of all test methods are outlined in PLAN-TA9-2443 Rev B, Attachment B. The results of the tests are documented in M-7 Analytical Laboratory number 52279 (M-7-AC-52279). For tests carried out with the aid of control or analysis software, Software Quality Management documentation is included in Appendix B of this report. Adiabatic calorimetry notebook pages for the third round are in Appendix C.

In the first round of testing, Differential Scanning Calorimetry (DSC) data was acquired for each sample. After the first round was completed, the DSC data were evaluated by subject matter experts and it was determined that this test was not providing useful information about the RNS formulations. The data was not reproducible in replicate runs, making it impossible to say what the characteristic thermal features were for any given RNS formulation or to determine trends as a function of sWheat or Pb content. This was caused by the very small sample sizes required for the DSC used by M-7 (less than 3 mg) and the inhomogeneity of the RNS formulations. The amounts of the materials going into the formulations are not measured out finer than the 10 mg level due to granularity and the grinding step in the formulation procedure is not able to homogenize the samples well enough for the small sample sizes required by DSC. Based on these considerations and the corrosion that the RNS formulations were causing in the DSC cell, it was decided to discontinue DSC data acquisition for the second and third rounds of testing.

Drop Weight Impact (DWI) Testing

No RNS surrogates showed any impact sensitivity in any round of testing. Each one showed 15 consecutive No-Go responses when the test weight was dropped from 320 cm. Note that all internal standards showed expected behavior.

Friction Testing

No RNS surrogates showed any friction sensitivity in any round of testing. Each one showed 15 consecutive No-Go responses with the largest weight at the full extent of the lever arm. Note that all internal standards showed expected behavior.

Electrostatic Discharge (ESD) Testing

The RNS surrogates showed some spark sensitivity in all rounds of testing but there were no trends with sWheat content or Pb content. Since the samples did not show any sensitivity to impact or friction, this spark sensitivity may be due to gases evolved from the sample. In the ESD sample holder, the sample is confined and any reactive gases are trapped until the probe needle discharges through the holder, causing any volatile head space to react. Note that all internal standards showed expected behavior.

Automatic Pressure Tracking Adiabatic Calorimetry (APTAC) Testing

APTAC testing measures the sample self-heating as it is heated adiabatically and step-wise in 2 ˚C increments (Heat-Wait-Search mode). Notebook pages from the third round of testing are documented below in Appendix C. Those pages also include the temperature and pressure verifications and instrument verification runs with di-tert butyl peroxide (DTBP) in toluene. Verifications were carried out before and after running the formulations in the test matrix. The CoA's for the toluene and the DTBP, obtained from IESL vendors, are also included in Appendix A.

Table 4 shows the temperatures of onset of self-heating for all 9 formulations for all three rounds of testing. The onset is defined as the temperature at which the sample self-heating rate exceeded 0.02 ˚C/min. The largest variability across nominally equivalent formulations is 8 ˚C. Averaging the temperature span of each cell produces a value of 5 ˚C. This average temperature span is used for error bars in Figure 1 where the average onset is plotted vs formulation.

Table 4. Temperatures of onset of self-heating for all 9 formulations in Table 2. Each cell contains onset temperatures from all three rounds of testing (rounds 1 to 3 ordered from top to bottom in each cell).

	15 % sWheat	**25 % sWheat**	**35 % sWheat**
4% Pb	42 ˚C 48 ˚C 50 ˚C	56 ˚C 56 ˚C 58 ˚C	64 ˚C 58 ˚C 56 ˚C
2 % Pb	48 ˚C 48 ˚C 44 ˚C	56 ˚C 56 ˚C 60 ˚C	60 ˚C 52 ˚C 58 ˚C
1% Pb	52 ˚C 48 ˚C 50 ˚C	52 ˚C 59 ˚C 54 ˚C	58 ˚C 54 ˚C 56 ˚C

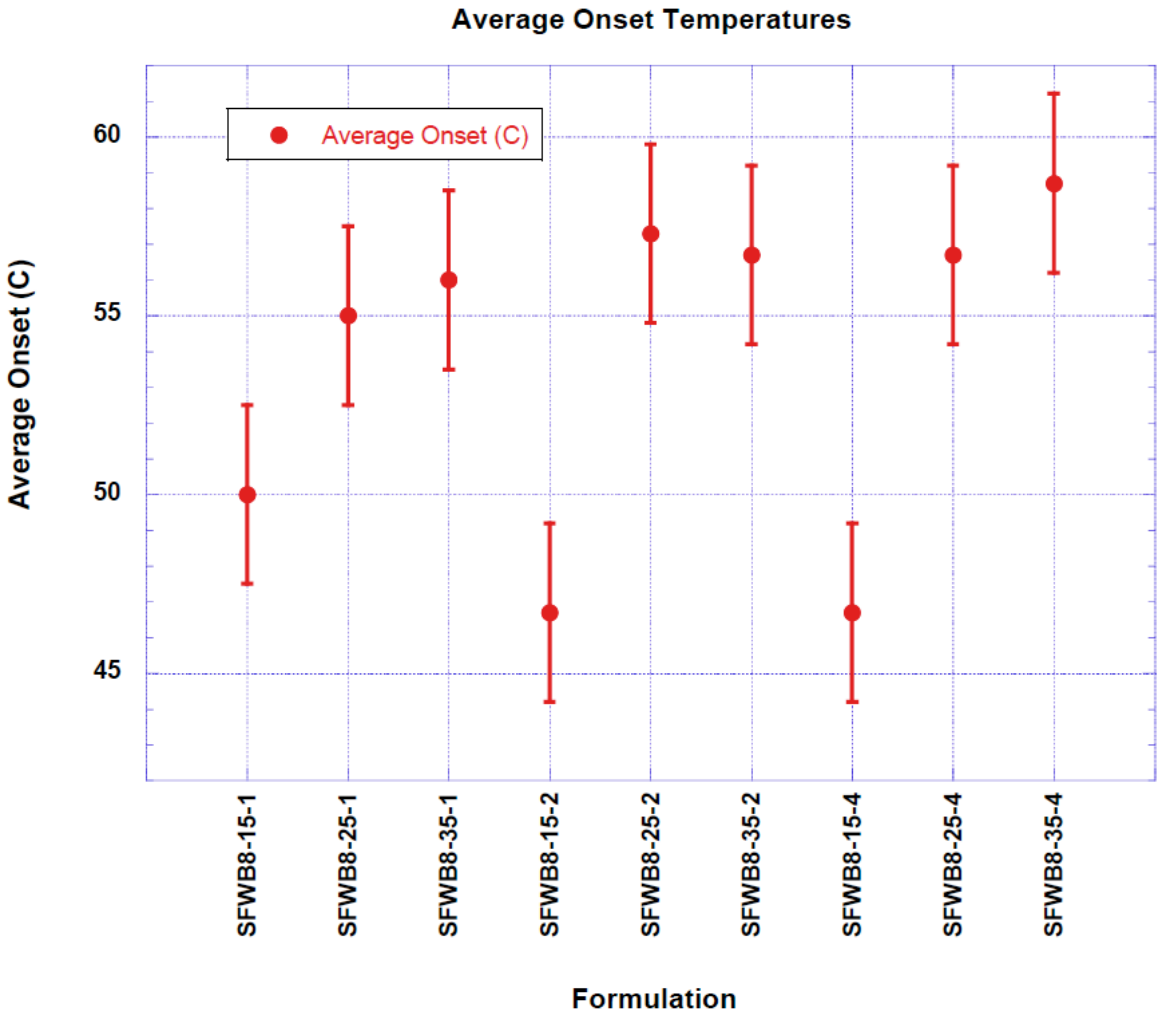


Figure 1. Average onset temperatures for all formulations. Error bars are the average measurement span of the cells in Table 4.

From Figure 1 it appears that the 15% mixtures have lower onsets than the other mixtures by at least 5 ˚C but that there is little variation with Pb content in any mixture and little variation between the 25% and 35% sWheat formulations.

Figures 2 through 4 show the heat flow traces for all formulations grouped by sWheat content. Each graph has results from all three rounds overlaid. The traces have been offset in time so that 0 minutes is the point at which self-heating was first detected. In this way it is possible to see the variability in the onset temperatures and the different rates at which self-heating progressed for each sample.

Examination of Figures 2 through 4 shows some traces that proceed from onset to rapid thermal runaway smoothly and some that go through one or more transitions – observed as a change in curvature and/or slope. There is no correlation between mixture type and the trace profile. The cases where the trace does show plateaus indicates that different reactions are dominating at different times either due to

consumption of some reactants or the effect of different kinetic parameters. This is not surprising given the multicomponent nature of the mixtures. Based on this observation and the lack of correlation with mixture type, it is reasonable to assume that a set of nominally equivalent reactions is occurring in all of the mixtures at various times and with various levels of heat generation. In the mixtures that appear to be closer to a single smooth exothermic event, these reactions would overlap – the heat from one driving the next as the material thermally runs away.

Figures 2 through 4 also show a large amount of variability in the time to the most rapid thermal runaway for several of the mixtures. Examples are the 4% Pb mixtures in Figure 2, the 2% Pb mixtures in Figure 3, and the 2% Pb mixtures in Figure 4. The time to this final runaway does not appear to be correlated with the onset temperature. The variability also illustrates the inherent inhomogeneity in these multicomponent mixtures.

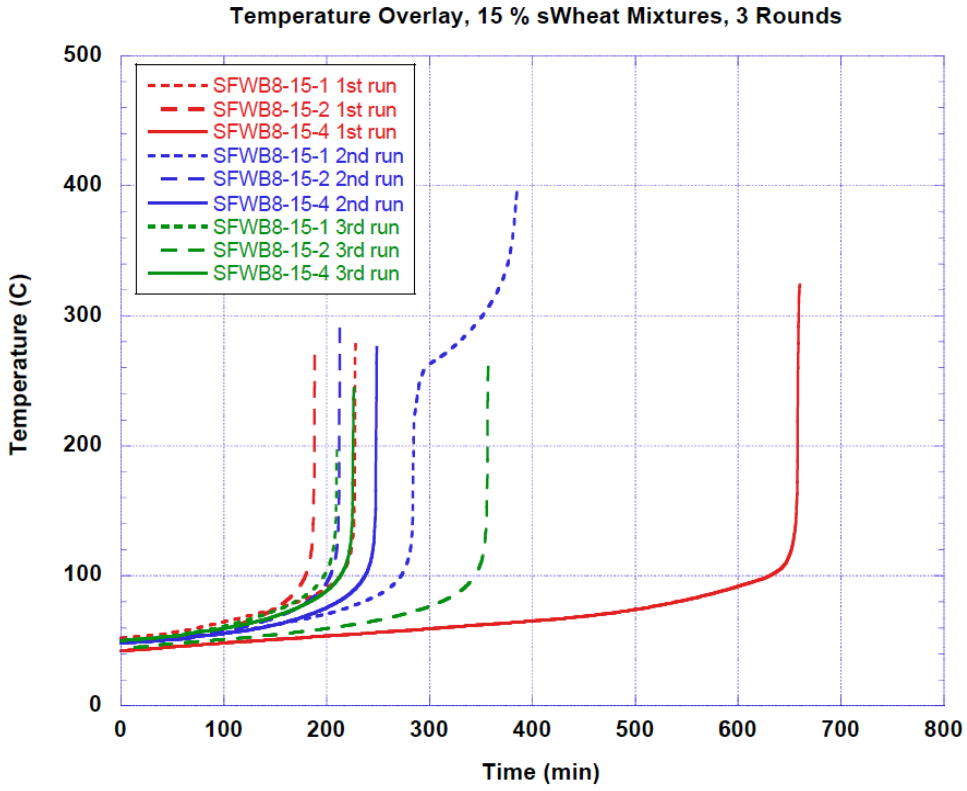


Figure 2. APTAC temperature vs time plots for all 15% sWheat RNS mixtures

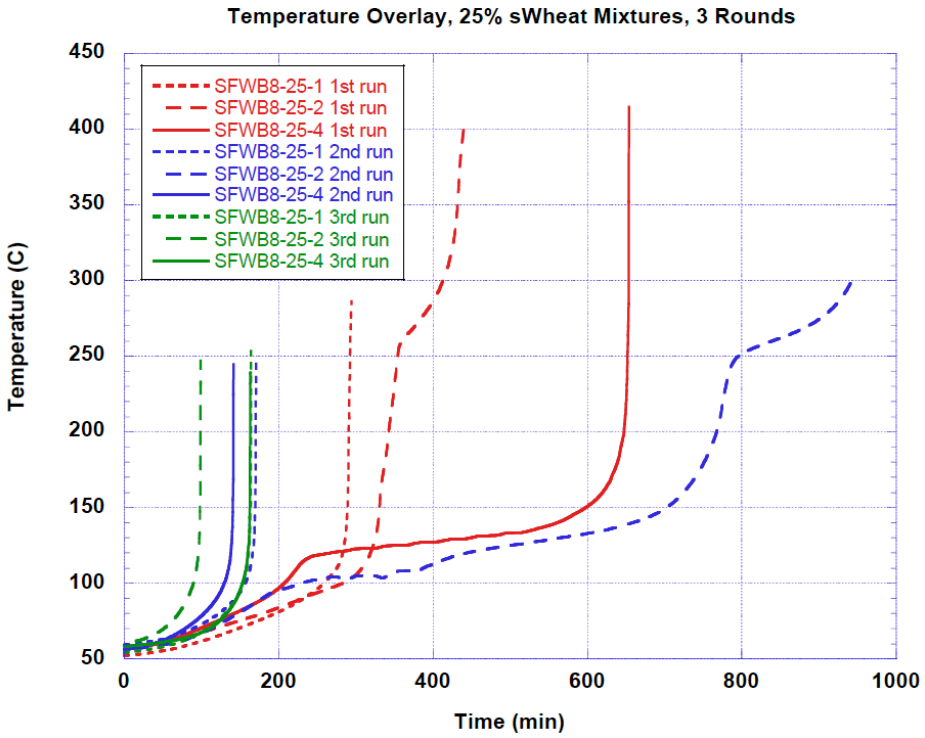


Figure 3. APTAC temperature vs time plots for all 25% sWheat RNS mixtures

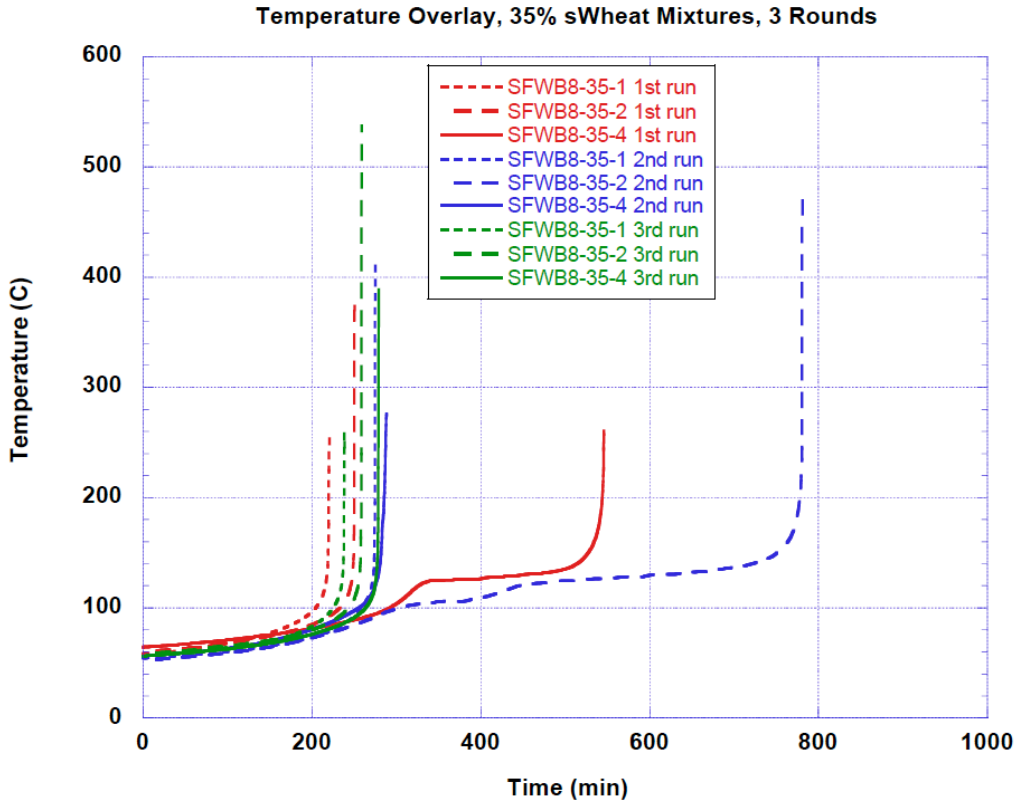


Figure 4. APTAC temperature vs time plots for all 35% sWheat RNS mixtures

Figures 5 through 7 show the pressure traces associated with each heat flow trace in Figures 2 through 4. The curves have been offset in time in the same way that the temperature data were offset. The pressure traces also show contributions from different reactions as multiple dips, peaks, and slopes. In all cases, the most vigorous pressure generation corresponds to the fastest heat generation in the corresponding Figure above. Note that this does not either support or refute that the reactions are pressure-dependent. There is no way (in this test configuration) to determine whether the increase in pressure is driving the increase in temperature. The data would have a similar appearance if the pressure increase was the result of increased generation of gaseous products or simply due to the ideal gas law.

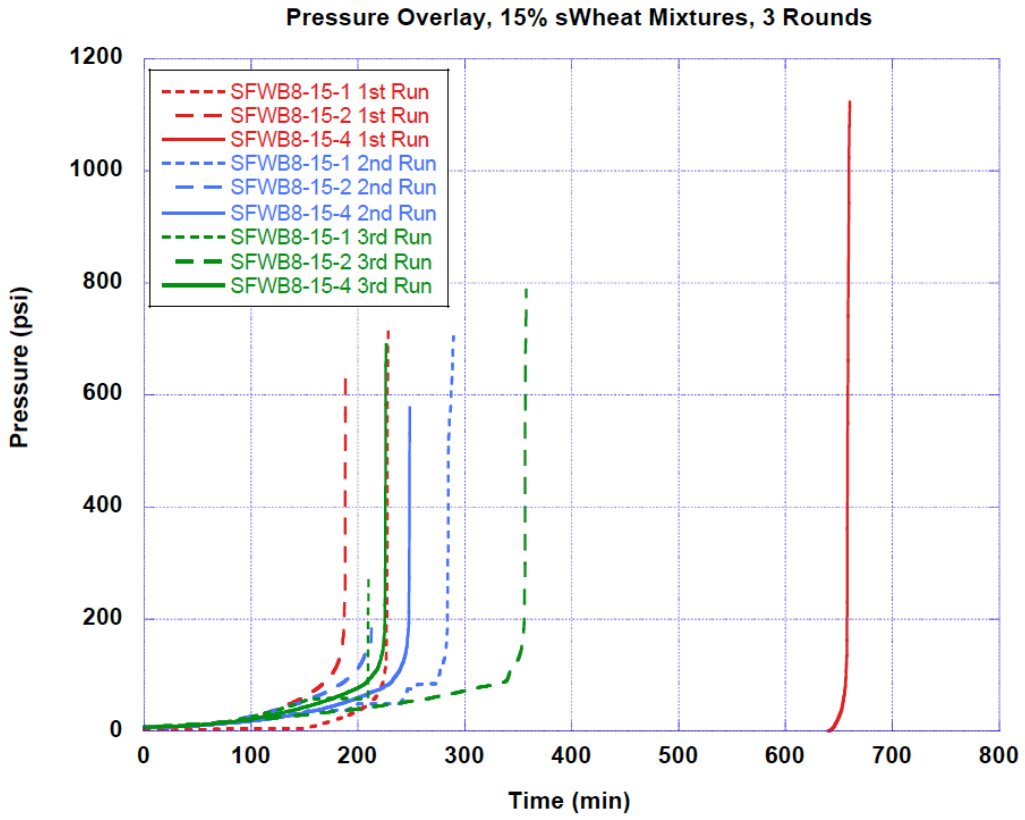


Figure 5. APTAC pressure vs time plots for all 15% sWheat RNS mixtures

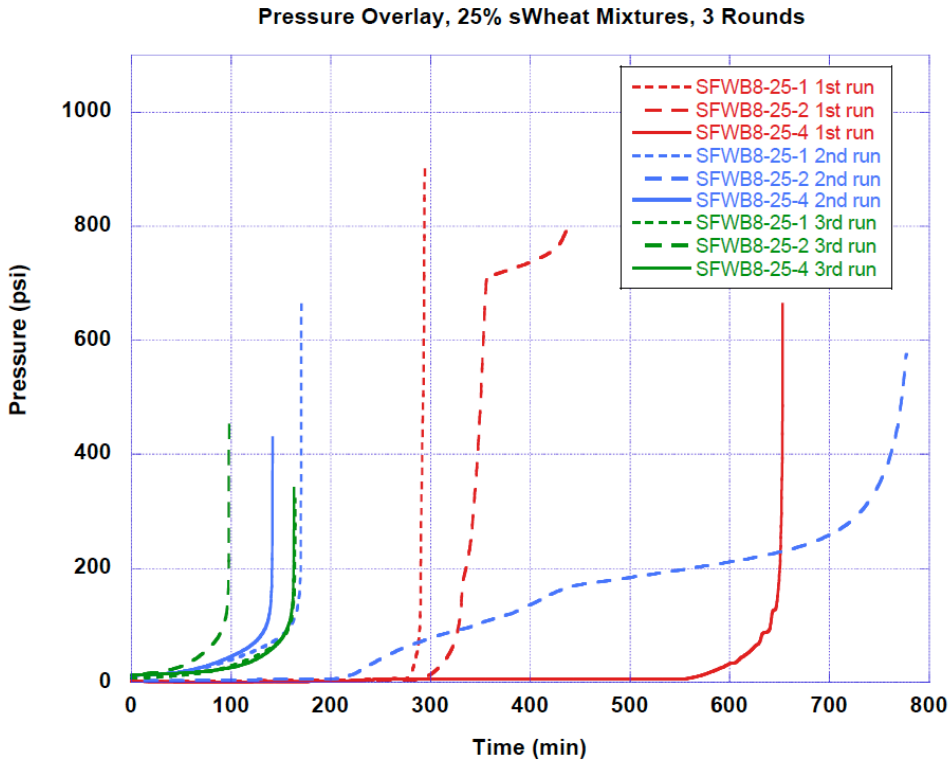


Figure 6. APTAC pressure vs time plots for all 25% sWheat RNS mixtures

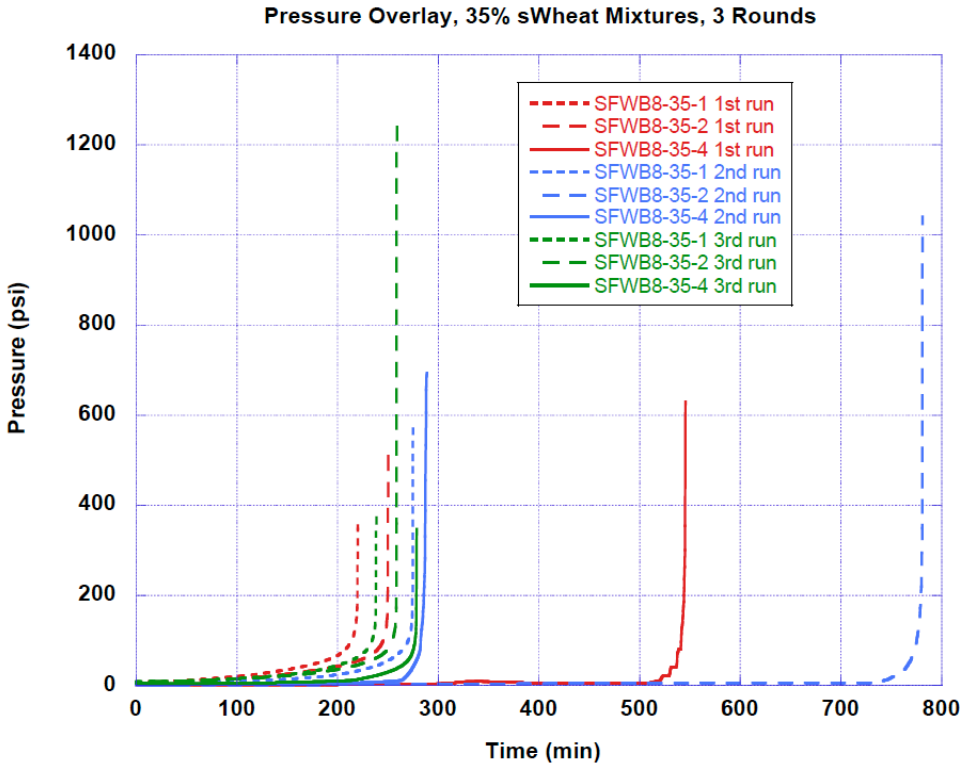


Figure 7. APTAC pressure vs time plots for all 35% sWheat RNS mixtures

The self-heating segments from the APTAC runs were analyzed with vendor-supplied software to determine kinetic parameters. This analysis was less than robust for two reasons. First, all of the available models that can be fit to the data assume single reactions, but many of the data sets show slope changes or other features indicative of multiple reactions. Second, most of the initial self-heating segments drove the APTAC instrument to shut down due to a temperature, temperature rate, or pressurization rate limit being exceeded. As a result, these data sets do not include the heat generated through full completion of the reaction under adiabatic conditions, violating an assumption used for kinetic analysis.

In order to estimate the relative kinetics of the different compositions, all data sets were fit to a first order Arrhenius model with endpoints adjusted so that the resulting parameters best fit a maximal portion of the data set. This approach is somewhat subjective but does provide a relative comparison. The results of this approach are shown in Table 5 with results from all three rounds in each cell, ordered from top to bottom. The scatter in the data is apparent and reflects the experimental issues noted above as well as inhomogeneity of the samples. There are no obvious trends with sWheat or Pb content.

Table 5. Arrhenius kinetic parameters for the initial self-heating segments of the RNS surrogate formulations. "A" is the pre-exponential factor in log(1/s) and "E_a" is the activation energy in kJ/mol. Each cell contains kinetic parameters from all three rounds of testing (rounds 1 to 3 ordered from top to bottom in each cell).

SFWB8-15-4	SFWB8-25-4	SFWB8-35-4
A = 4.5, E_a = 63*	A = 5.4, E_a = 62	A = 8.3, E_a = 85
A = 11.1, E_a = 106	A = 9.7, E_a = 95	A = 6.4, E_a = 73
A = 7.1, E_a = 77	A = 7.3, E_a = 77	A = 8.2, E_a = 86
SFWB8-15-2	SFWB8-25-2	SFWB8-35-2
A = 9.4, E_a = 93	A = 12.1, E_a = 116	A = 8.5, E_a = 88
A = 6.4, E_a = 71	A = 11.8, E_a = 103	A = 8.1, E_a = 80
A = 7.9, E_a = 84	A = 7.3, E_a = 77	A = 7.7, E_a = 83
SFWB8-15-1	SFWB8-25-1	SFWB8-35-1
A = 11.9, E_a = 113	A = 11.2, E_a = 110	A = 11.0, E_a = 107
A = 9.7, E_a = 95	A = 9.9, E_a = 97	A = 12.6, E_a = 118
A = 9.5, E_a = 94	A = 7.7, E_a = 81	A = 7.8, E_a = 82

* SFWB8-15-4 results were reanalyzed for this report.

Summary

Nine RNS surrogate formulations were formulated and analyzed in triplicate per PLAN-TA9-2443, Rev B. Sensitivity testing showed all formulations were insensitive to impact and friction although most had some sensitivity to spark discharge. Comparison of APTAC data from all three rounds of testing showed no obvious trends with sWheat or Pb content with the exception that the 15% Swheat formulations exhibited self-heating onset temperatures that were about 5 ˚C lower than those of the other formulations. Onset temperatures for all mixtures in the second round of testing ranged from 42 ˚C to 64 ˚C, with an average of 54 ˚C.

Contents of Appendices

GB:mq

Cy: MDO DCRM file, P942

ENCLOSURE 2

Initial Results from the Third Round of Remediated Nitrate Salt Surrogate Formulation and Testing

ADESH-16-076

LA-UR-16-22729

Date: MAY 1 3 2016

LA-UR-16-22729
Approved for public release; distribution is unlimited.

Title: Initial Results from the Third Round of Remediated Nitrate Salt Surrogate Formulation and Testing

Author(s): Brown, Geoffrey Wayne
Leonard, Philip
Hartline, Ernest Leon
Tian, Hongzhao

Intended for: Report

Issued: 2016-04-20

Los Alamos
NATIONAL LABORATORY
memorandum

M-7: High Explosive Science and Technology

To/MS: David J. Funk, ADEP, djf@lanl.gov
From/MS: Geoffrey Brown, M-7, geoffb@lanl.gov
Phone/Fax: 7-6718/7-0500
Symbol: M7-16-6057
Date: April 12, 2016

SUBJECT: Initial Results from the Third Round of Remediated Nitrate Salt Surrogate Formulation and Testing

Contributors: Geoff Brown, Phil Leonard, Ernie Hartline, Hongzhao Tian (M-7)

Introduction

High Explosives Science and Technology (M-7) is currently working on the third round of formulation and testing of Remediated Nitrate Salt (RNS) surrogates. This report summarizes the calorimetry results from the 15 % sWheat mixtures. All formulation and testing was carried out according to PLAN-TA9-2443 Rev B, "Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing Standard Procedure", released February 16, 2016. Results from the first and second rounds of formulation and testing were documented in memoranda M7-16-6042 and M7-16-6053.

Materials

The materials used to formulate the RNS surrogates are listed in the table below. All chemicals were ordered through IESL-approved vendors using the Oracle iProcurement system. The Certificates of Analysis and packing slips for each item were scanned and documented in memorandum M7-15-6033, "Starting Materials Available for RNS Surrogate Formulation."

Table 1. Materials used for formulating RNS surrogates

Material	Lot Number
Aluminum nitrate nonahydrate	142299
Calcium nitrate tetrahydrate	144946
Chromium nitrate nonahydrate	F04Y024
Iron nitrate nonahydrate	A0355097
Water, LCMS grade	145280
Magnesium nitrate	147856
Sodium nitrate	144821
Lead nitrate	143996
Oxalic acid	143866 and 145400
Potassium carbonate	145088

sWheat pet litter was obtained commercially by LANL Environmental Programs and a 20 lb bag was supplied to M-7 per PLAN-TA9-2443 Rev B.

Formulation

The base RNS surrogate component is designated SFWB-8 and consists of the mixture listed in Table 2.

Table 2. SFWB-8 composition

Material	Weight %
$Al(NO_3)_3 * 9\ H_20$	3.20
$Ca(NO_3)_2 * 4\ H_2O$	12.72
$Cr(NO_3)_3 * 9H_2O$	0.16
$Fe(NO_3)_3 * 9H_2O$	4.86
$Mg(NO_3)_2 * 6H_2O$	35.69
$NaNO_3$	7.91
$(COOH)_2 * 2H_20$	2.89
K_2CO_3	1.51
Water	4.31

The final RNS surrogate formulations consist of SFWB-8 with lead nitrate and sWheat pet litter in the weight ratios shown in Table 3.

In the discussions below, the samples will be designated with a shorthand notation of the form SFWB8-XX-Y, where XX is the sWheat percentage and Y is the Pb percentage. For example, SFWB8-15-1 is the mixture made using SFWB-8 with 15% sWheat and 1% Pb.

Table 3. RNS surrogate formulation matrix for 15% sWheat mixtures. Weight percent values are shown.

SFWB8-15-1	SFWB8-15-2	SFWB8-15-4
1% $Pb(NO_3)_2$	2% $Pb(NO_3)_2$	4% $Pb(NO_3)_2$
15% Swheat	15% Swheat	15% Swheat
84% WB-8	83% WB-8	81% WB-8

Formulation followed section 4.2 of PLAN-TA9-2443 Rev B with any relevant observations documented in the formulation notes that will be included in a future revision of this memorandum.

Testing

Details of the calorimetry test method, Automatic Pressure Tracking Adiabatic Calorimetry (APTAC), are outlined in PLAN-TA9-2443 Rev B, Attachment B. The results of the tests will be formally documented in M-7 Analytical Laboratory number 52279 (M-7-AC-52279) when all 9 formulations have been tested. The control and analysis software used for this work have Software Quality Management documentation that will be included in a future revision of this report.

Automatic Pressure Tracking Adiabatic Calorimetry (APTAC) Testing

APTAC testing measures the self-heating and associated kinetic parameters as the sample is heated adiabatically and step-wise in 2 ˚C increments (Heat-Wait-Search mode). Approximately 4 grams of sample are heated in a sealed vessel for this test.

Table 4 shows the temperatures of onset of self-heating for the 15% sWheat formulations for all three rounds of testing. The onset is defined as the temperature at which the sample self-heating rate exceeded 0.02 ˚C/min. The largest variability across nominally equivalent formulations is 8 ˚C for the 4% Pb mixtures. If that is taken as an indication of the possible standard deviation then the values in Table 4 are all statistically indistinguishable and suggest an average onset for the 15% mixtures of 48 ˚C +/- 3 ˚C. This observation and examination of the variation in each column suggests that Pb has little effect on the temperature onset for these 15 % sWheat formulations.

Table 4. Temperatures of onset of self-heating for 15% sWheat mixtures in all 3 rounds.

	15 % sWheat 1st Round	**15 % sWheat 2nd Round**	**15 % sWheat 3rd Round**
4% Pb	42 ˚C	48 ˚C	50 ˚C
2 % Pb	48 ˚C	48 ˚C	44 ˚C
1% Pb	52 ˚C	48 ˚C	50 ˚C

Figure 1 shows heat flow traces for the three rounds of the 15% sWheat formulations. The traces have been offset in time so that 0 minutes is the point at which self-heating was first detected. This illustrates the different onset temperatures and the different rates at which self-heating progressed for each sample.

Examination of Figure 1 shows wide variability in the reaction progress for replicate mixtures. The most extreme difference is seen in the SFWB8-15-4 mixtures. For those, the 3rd run begins its fastest thermal runaway over 400 minutes earlier than the 1st run even though the 3rd run had a higher onset temperature. Another variation is seen in the second run of SFWB-15-1 as a plateau around 280 minutes. This was noted in previous reports for other mixtures as well and is attributed to an initial reaction reaching completion while other reactions continue to generate heat. This is not surprising given the multicomponent nature of the mixtures. These data also suggest that Pb has little effect on the reaction since neither the time to fastest runaway or the run up to that point show any obvious trends.

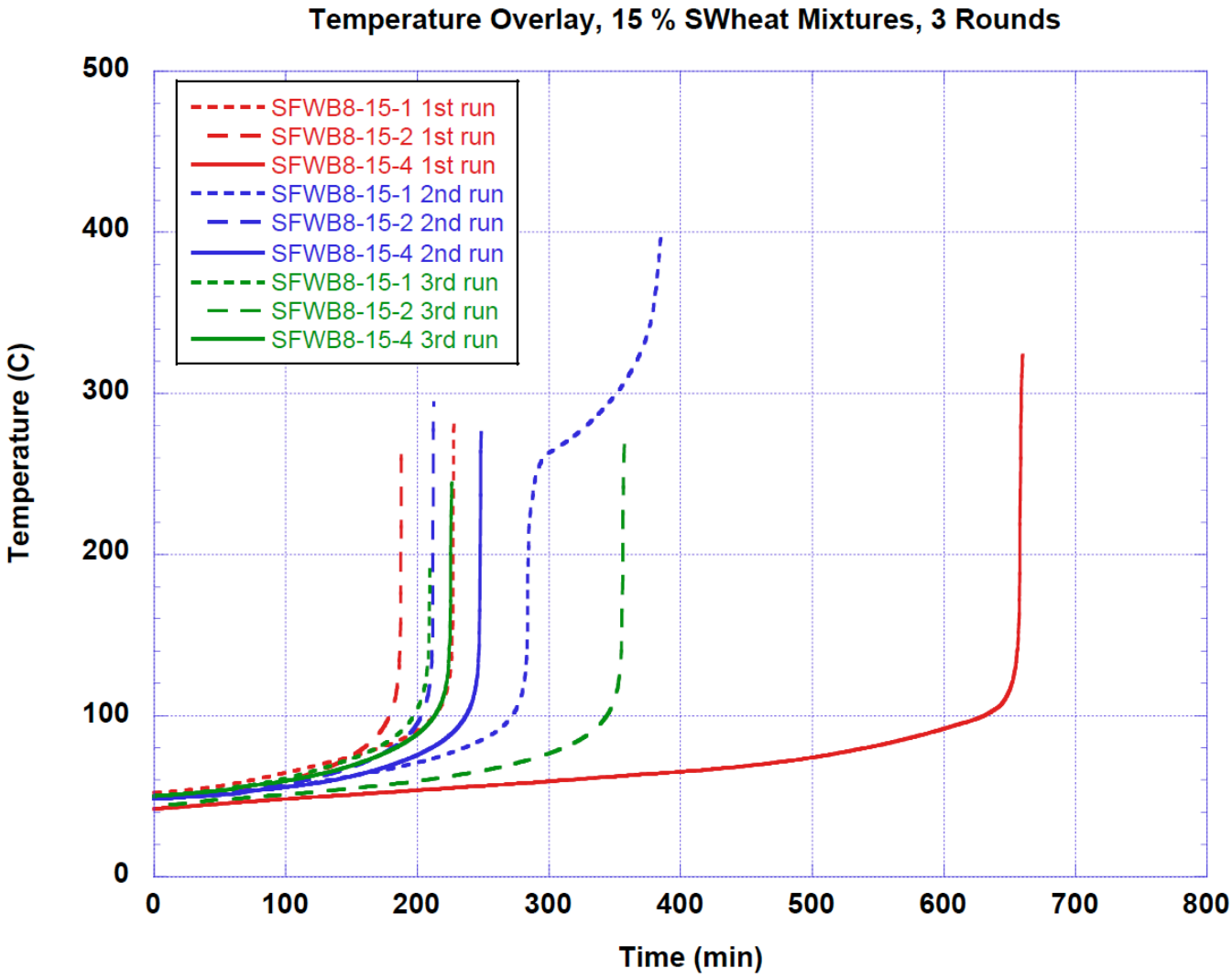


Figure 1. APTAC temperature vs time plots for all 15% sWheat RNS mixtures.

Figure 2 shows the pressure traces associated with each heat flow trace in Figure 1. The curves have been offset in time in the same way as those of Figure 1. In each case, the most rapid pressure generation corresponds to the fastest self-heating. From the data it is not possible to say whether the increase in pressure is driving the increase in temperature or vice versa. There are some step structures in some of the pressure traces that are attributed to temporary partial plugs in the pressure lines leading to the transducers since they do not correspond to any thermal events. Plugging is also often observed after the runs complete and it can be difficult to clear.

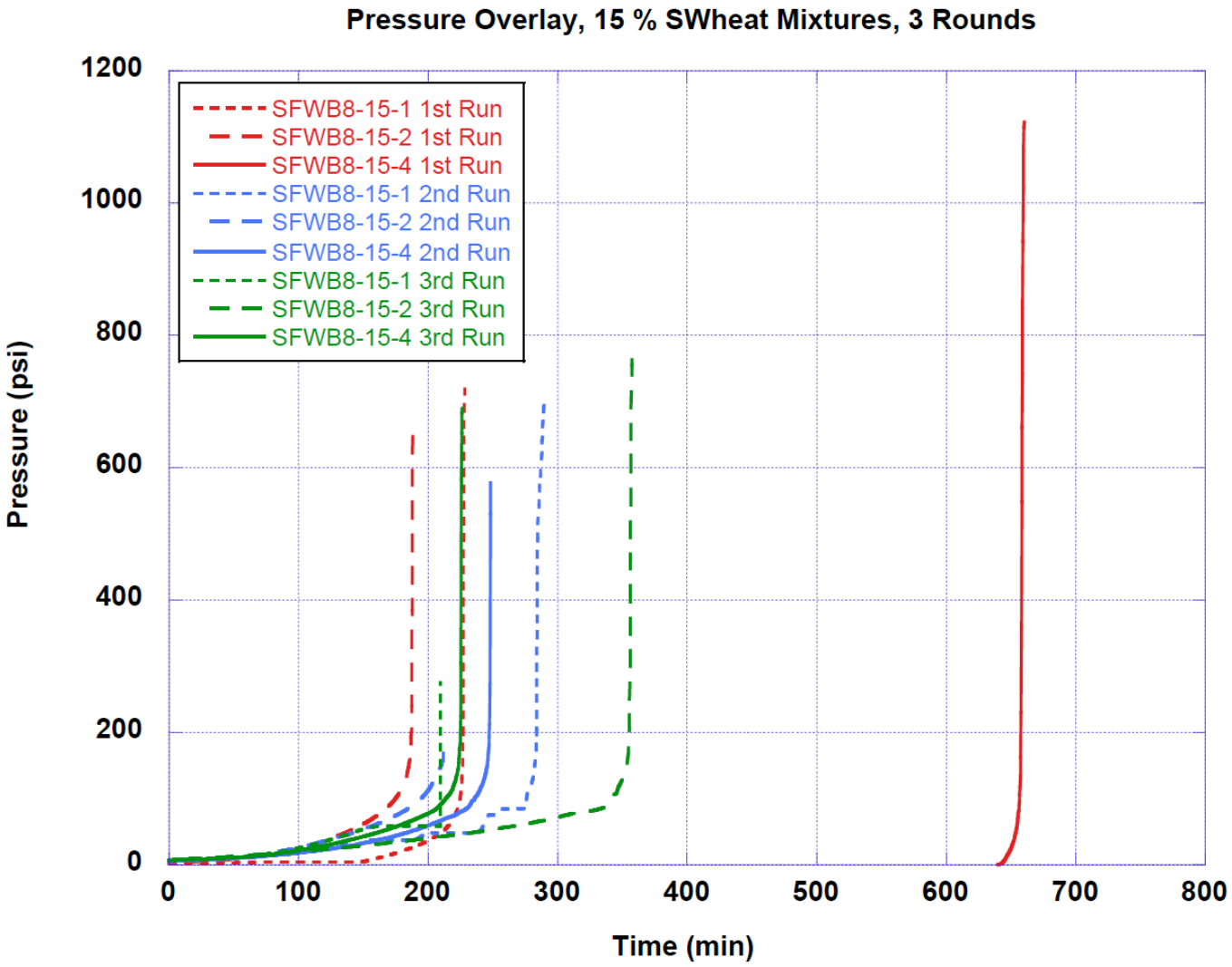


Figure 2. APTAC pressure vs time plots for all 15% sWheat RNS mixtures.

Each initial self-heating segment from the APTAC runs was analyzed with vendor-supplied software to determine kinetic parameters. This was less than a robust analysis, for two reasons. First, all of the available models that can be fit to the data assume single reactions, but many of the data sets show slope changes or other features indicative of multiple reactions. Second, most of the initial self-heating segments drove the APTAC instrument to shut down due to a temperature, temperature rate, or

pressurization rate limit being exceeded. As a result, these data sets do not include the heat generated through full completion of the reaction under adiabatic conditions, violating an assumption used for kinetic analysis.

In order to provide some estimates of the relative kinetics of the different compositions, all data sets were fit to a first order Arrhenius model with endpoints adjusted so that the resulting parameters best fit a maximal portion of the data set. This approach is obviously somewhat subjective but does allow some relative comparison. The results of this approach are shown in Table 5. The scatter in the data is apparent and reflects the experimental issues noted above. There are no obvious trends with Pb content.

Table 5. Arrhenius kinetic parameters for the initial self-heating segments of the 15 % sWheat RNS surrogate formulations. "A" is the pre-exponential factor in log(1/s) and "E_a" is the activation energy in kJ/mol.

	15 % sWheat 1st Round	**15 % sWheat 2nd Round**	**15 % sWheat 3rd Round**
4% Pb	A = 2.3 E_a = 47	A = 11.1 E_a = 106	A = 7.1 E_a = 77
2 % Pb	A = 9.4 E_a = 93	A = 6.4 E_a = 71	A = 8.0 E_a = 84
1% Pb	A = 11.9 E_a = 113	A = 9.7 E_a = 95	A = 9.5 E_a = 94

Summary

Three rounds of the 15% sWheat RNS surrogate formulations have been analyzed per PLAN-TA9-2443, Rev B. There were no general trends observed as a function of Pb content after examining onset temperature, kinetic parameters, and overlays of temperature and pressure traces. The average onset temperature for the 15 % sWheat formulations from these data is 48 ˚C +/- 3 ˚C.

GB:mq

Cy: MDO DCRM file, P942

ENCLOSURE 3

Summary Report of Laboratory Testing to Establish the Effectiveness of Proposed Treatment Methods for Unremediated and Remediated Nitrate Salt Waste Streams

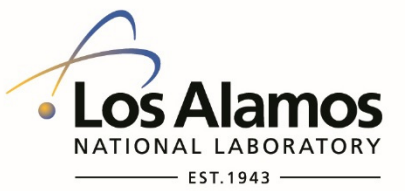
ADESH-16-076

LA-UR-16-22658

Date: MAY 1 3 2016

LA-UR-16-22658
May 2016

Summary Report of Laboratory Testing to Establish the Effectiveness of Proposed Treatment Methods for Unremediated and Remediated Nitrate Salt Waste Streams



Prepared by Kurt Anast and David J. Funk

EXECUTIVE SUMMARY

The inadvertent creation of transuranic waste carrying hazardous waste codes D001 and D002 requires the treatment of the material to eliminate the hazardous characteristics and allow its eventual shipment and disposal at the Waste Isolation Pilot Plant (WIPP). This report documents the effectiveness of two treatment methods proposed to stabilize both the unremediated and remediated nitrate salt waste streams (UNS and RNS, respectively). The two technologies include the addition of zeolite (with and without the addition of water as a processing aid) and cementation. Surrogates were developed to evaluate both the solid and liquid fractions expected from parent waste containers, and both the solid and liquid fractions were tested. Both technologies are shown to be effective at eliminating the characteristic of ignitability (D001), and the addition of zeolite was determined to be effective at eliminating corrosivity (D002), with the preferred option[1] of zeolite addition currently planned for implementation at the Waste Characterization, Reduction, and Repackaging Facility. During the course of this work, we established the need to evaluate and demonstrate the effectiveness of the proposed remedy for debris material, if required. The evaluation determined that Wypalls absorbed with saturated nitrate salt solutions exhibit the ignitability characteristic (all other expected debris is not classified as ignitable). Follow-on studies will be developed to demonstrate the effectiveness of stabilization for ignitable Wypall debris. Finally, liquid surrogates containing saturated nitrate salts did not exhibit the characteristic of ignitability in their pure form (those neutralized with Kolorsafe and mixed with sWheat did exhibit D001). As a result, additional nitrate salt solutions (those exhibiting the oxidizer characteristic) will be tested to demonstrate the effectiveness of the remedy.

.

CONTENTS

Tables

1.0 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 (LANL) was later identified as the source of the release.[2] The waste container in question, drum 68660, was improperly remediated and contained an ignitable mixture of nitrate salt waste and organic absorbent material (sWheat Scoop kitty litter).

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, the remediation path for the nitrate salt waste was identified to be the addition of kitty litter/zeolite clay to the nitrate salts within the containers and absorption of liquids in the containers also by kitty litter/zeolite clay. These included containers that had previously been remediated using an absorbent called Waste Lock 770. Therefore, before they are shipped to WIPP, the containers were to be opened in a glovebox, free liquids were to be absorbed with kitty litter/zeolite clay, and the salt material was to be mixed with the absorbent in a ratio that would stabilize the salts. Resulting material was to be repackaged to meet the WIPP waste acceptance requirements. Waste processing of many of these containers was conducted, but the absorbent utilized was an organic kitty litter absorbent instead of the Difficult Waste Team prescription of kitty litter/zeolite clay, resulting in the generation of an incompatible mixture that led to spontaneous combustion of the waste, as documented through investigation into the WIPP event.[2]

From these waste-processing activities, daughter containers were generated that hold 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 containers, unremediated nitrate salt (UNS) wastes, as well as 60 containers with remediated, absorbed, and repackaged nitrate salt wastes. Containers of remediated and unremediated nitrate salt (RNS) and UNS waste are characterized as exhibiting the U.S. Environmental Protection Agency (EPA) Hazardous Waste Number (HWN) D001 for ignitability (both RNS and UNS waste) and D002 for corrosivity (RNS and UNS 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 RNS and UNS waste must be conducted before certification, shipment, and disposal at that facility.

The stabilization treatment methods that are to be examined for their effectiveness in the treatment of both the UNS and RNS wastes include (1) the addition of zeolite (with and without the addition of water) and (2) cementation. Adding zeolite is proposed (and preferred) based on the results of several previous studies and analyses that specifically examined the effectiveness of this process for deactivating nitrate salts.[3] Cementation is also being assessed because of the prevalence of cementation as an immobilization method for similar wastes at numerous facilities around the U.S. Department of Energy complex, including at LANL. The results of the treatment study will be used with other testing to provide the basis for a request to the New Mexico Environment Department Hazardous Waste Bureau for approval of a Resource Conservation and Recovery Act (RCRA) permit modification for the proposed treatment process and the associated facilities.

The specific purpose of this report is to summarize the initial testing conducted to validate that the proposed treatment methods are effective at safely removing the EPA HWNs D001 and D002 from both the UNS and RNS wastes. These studies also demonstrated that the proposed volumetric quantities for the mixtures are sufficient to ensure removal of EPA HWNs D001 and D002 (ignitability and corrosivity characteristics) as required for disposal at WIPP (at a minimum). Appendixes A through E to this report provide test methods used and Appendixes F through L provide detailed information on test results.

Testing was conducted by an independent contract laboratory, Southwest Research Institute (SwRI), located in San Antonio, Texas, using nonradioactive surrogate samples to avoid worker safety risks associated with testing, packaging, and transporting samples of actual radioactive waste materials. Additional characterization and treatment testing activities were conducted on-site at LANL and are discussed below and in Appendix K.

2.0 RNS DRUM COMPOSITION

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-gal. drum. The salt/sWheat blend was placed directly into the fiberboard liner without any protective plastic around the waste. 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 drums contain 20 volume-percent or more debris waste. Twelve RNS containers consist of 12-in. pipe overpack containers (POCs) inside 55-gal. drums.

Free liquid can be identified using real-time radiography (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 POCs that were taken in 2011 and 2012. Five RNS drums (at that time) were reported to contain liquid: three contain less than 100 mL and two POCs contain about 2 L outside the containment bag in the POC.

The waste in the RNS container largely consists of the following waste matrices:

- Homogeneous solids—nitrate salts absorbed with organic kitty litter (SWheat)
- Scrap lead
- Leaded gloves
- Rubber gloves
- Scrap metal
- Plastics—rigid liner, plastic bags, plastic sheeting
- Cardboard/fiberboard liner
- Cellulose

3.0 SCOPE OF WORK

SwRI was contracted to provide qualified personnel, equipment, materials, and facilities to formulate and analyze waste surrogates and treatment options in support of LANL's effort to evaluate RNS waste and to provide insight into the effectiveness of treatment options. This effort included formulating surrogate nitrate salts, preparing nitrate salt solutions, and blending the nitrated salts or solutions with sWheat kitty litter, zeolite, cement, water, or Waste Lock 770. The effectiveness for cementation as a treatment technology and the addition of zeolite were assessed for surrogate nitrate salt wastes and the blends prepared to simulate treatment to ensure that RCRA characteristics of ignitability (and corrosivity, where applicable) are removed from the waste after treatment. Debris waste found in the drums was also submitted for evaluation and examination of the impact of RNS waste and UNS solution on the behavior of the debris when it undergoes reactivity testing.

The test work includes the following areas of focus:

- Preparation of surrogate salt known as WB8 identified in the analysis by Wesibrod et al.[4] and evaluated through testing by Brown, et al.[5] This test work is presented in Section 4.1, and the memorandum summarizing the first round of small-scale testing at LANL is included as Appendix K of this report.
- Preparation of surrogate UNS solution utilizing saturated nitrate solutions derived from Reference 4 and neutralized with Spilfyter as discussed in Reference 2. This test work is discussed in Section 4.3.
- Preparation and evaluation of surrogate RNS waste prepared from WB8 salt and sWheat kitty litter to simulate the possible RNS waste material found in the 60 RNS drums to be remediated at LANL. This test work is discussed in Section 6.1.
- Preparation and evaluation of blends composed of surrogate RNS waste and zeolite with and without the addition of water to simulate the remediation of RNS waste by blending with zeolite. This test work is discussed in Section 6.2.
- Preparation and evaluation of blends of surrogate RNS waste mixed with water and cement to simulate the remediation of RNS waste by cementation. This test work is discussed in Section 6.3.
- Preparation and evaluation of blends composed of surrogate UNS solution (prepared from the UNS surrogate solution recipe) and sWheat kitty litter and then blended with zeolite. This test work is discussed in Section 6.4.
- Preparation of surrogate debris waste to simulate the debris waste found in the RNS waste drums at LANL and evaluation for D001 characteristic. This test work is found in Section 7.1

4.0 TEST MATERIALS

Test materials used in the test work conducted by SwRI are described in this section.

4.1 Surrogate Nitrated Salt – WB8

Nitrated salts were removed from UNS drums, mixed with sWheat, and placed in daughter drums now referred to as RNS drums. The recipe for the surrogate nitrate salt WB8, used in all SwRI nitrate salt blends, is presented in Table 1. This nitrate salt recipe is based upon previous test work conducted at LANL[4,5,6] and is discussed as a bounding surrogate in "Technical Basis for the Removal of Unremediated Nitrate Salt Sampling (UNS) to Support LANL Treatment Studies."[7] The material selected is supported through studies that evaluated the sensitivity of RNS waste that used sWheat Scoop kitty litter as an absorbent. Previous testing has indicated that at least two factors are critical for ignition of the formulation: the ratio of sWheat scoop kitty 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. Prolonged testing verified that lead nitrate is a catalyst for the ignition process. The amount of lead actually present in the waste is difficult to estimate precisely because of the complexity of its formation. In addition, heating and partial drying of the materials will result in additional worst-case scenarios: previous testing has indicated the dried material is more thermally sensitive.

Table 1
WB8 Surrogate

Material	Milligrams	Grams	Wt%
Al(NO3)3 * 9 H20	2145	2.145	4.21
Ca(NO3)2 * 4 H2O	8530	8.530	16.73
Cr(NO3)3 * 9H2O	105	0.105	0.21
Fe(NO3)3 * 9H2O	3258	3.258	6.39
Mg(NO3)2 * 6H2O	23939	23.939	46.94
NaNO3	5307	5.307	10.41
Pb(NO3)2	1884	1.884	3.69
(COOH)2*2H20	1936	1.936	3.80
K2CO3	1010	1.010	1.98
Water	2886	2.886	5.66

Note: This table presents an example of 51 g of WB8.

The recipe to prepare WB8 surrogate follows these steps:

- The masses of nitrate salt components are measured in a plastic or aluminum weigh-boat or on waxed paper using a balance calibrated to ± 1-mg uncertainty. The quantity of material measured will be within 10 mg of the desired quantity of material.
- The weighed portion of nitrate salts is transferred to a container for mixing.
- Once all of the nitrate salts are measured and placed into the mixing container, they are manually mixed together.
- The mass of oxalic acid dihydrate and potassium carbonate is measured in a plastic or aluminum weigh-boat or on waxed paper using a balance calibrated to ± 1-mg uncertainty. The quantity of material measured is within 10 mg of the desired quantity of material as follows:
    - ❖ Water is measured into a tared glass beaker or Nalgene bottle using a balance calibrated to ± 1-mg uncertainty.
    - ❖ Oxalic acid is added to the water and mixed thoroughly.
    - ❖ Potassium carbonate is added in small increments and stirred between each addition until foaming and bubbling subsides.
    - ❖ Potassium carbonate is added in this fashion until all of it has been added. The solution makes a slurry with a fair amount of undissolved chemical remaining on the bottom.
- The potassium oxalate solution formed above is shaken and added to the mixed nitrate salt and manually mixed.

4.2 Control Salt – Potassium Nitrate (Anhydrous)

Potassium nitrate was used as the control salt to compare the response of WB8 salt and WB8 blends during D001 and D002 testing. Potassium nitrate was selected as a control, primarily because it is a good oxidizer (Packing Group II) and it was previously used as a control in similar studies conducted by Walsh when analyzing stabilization of nitrates by zeolite.[3]

4.3 Surrogate Nitrate Salt Solution – UNS Surrogate Solution

The surrogate nitrate salt solution, "UNS Surrogate Solution," represents the solution expected within UNS drums and is derived from WB8. This surrogate is a simulant of solution collected when UNS drums were opened and absorbed into sWheat kitty litter during the preparation of the RNS drums. The UNS Surrogate Solution consists of a suite of specified salts presented in Table 2 and mixed with water to dissolve most of the solid salts (remains saturated). This surrogate solution is used to prepare UNS Surrogate Solution blends. UNS Surrogate Solution is then used to blend with sWheat and/or zeolite in various ratios and to simulate the RNS material and the zeolite-blended (remediated) product. It is used as prepared but may also be used after being neutralized with Kolorsafe Spilfyter to represent the neutralized liquid, as discussed in Reference 1. Section 4.8 provides additional details.

Table 2
UNS Surrogate Solution Salt Recipe

Reagent Chemical	Weight (g)	Weight (%)
Al(NO3)3 * 9 H20	2774.2	46.238
Ca(NO3)2 * 4 H2O	280.2	4.670
Cr(NO3)3 * 9H2O	20.6	0.342
Fe(NO3)3 * 9H2O	198.4	3.306
H2O-HNO3-Al-Ca-Cr-Fe-Mg-Ni	213.8	3.563
Mg(NO3)2 * 6H2O	1336.2	22.270
HNO3	21.2	0.354
NaNO3	164.6	2.742
Ni(NO3)2 * 6H2O	0.80	0.013
Pb(NO3)2	198.8	3.314
(COOH)2 * 2H20	791.2	13.188
Total	6000	100.000

Note: This table presents an example of 6000 g of UNS solution salt.

The recipe to prepare UNS Surrogate Solution follows these steps:

- The masses of reagent chemicals are measured in a plastic or aluminum weigh-boat or on waxed paper using a balance calibrated to ± 1-mg uncertainty. The quantity of material measured will be within 10 mg of the desired quantity of material.
- The weighed portion of reagent is transferred to a container for mixing.
- The salt formulation is then dissolved into water using 0.30 g of water per gram of salt.
- If the salts do not all dissolve, water is added in 10-mL increments until nearly all the salts are dissolved (about 2% remain).

4.4 sWheat Kitty Litter

The cat litter sWheat Scoop Original (sWheat) is made from wheat and is available at retail stores and was used in the remediation process that created the RNS waste. The amount of sWheat used was not well documented and as a result was included as a variable to establish the effectiveness of the proposed remedy against the range of materials expected in the RNS waste form.

4.5 Zeolite

KMI zeolite from Sandy Valley, Nevada, was used for the SwRI testing and was provided by LANL from the same lot used in blending-process development tests at LANL. It is one of the highest purity zeolites and has high absorptivity.

4.6 Waste Lock 770

Waste Lock 770 is a solid, granular cross-linked polyacrylate material that swells and absorbs many times its weight in aqueous solutions. Waste Lock 770 has been engineered to absorb water under pressure and has properties for the absorption and solidification of low-level radioactive waste and other types of waste sludges.

4.7 Portland Cement

Portland Cement Type I/II is a common or general-purpose cement that also meets the low-heat requirements of Type II cement.

4.8 Spilfyter Kolorsafe

Spilfyter Kolosafe is a liquid product (triethanolamine is the active chemical) that can adjust the pH of a liquid and change color to indicate the pH of the solution. This material was used to adjust solution pH in the remediation process that created the RNS waste.

5.0 SAMPLE TESTING

5.1 SW-846 Test Method 1030

SW-846 Test Method 1030 is used to help identify those solids that are “capable, under standard temperature and pressure, of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited, burn[s] so vigorously and persistently that it creates a hazard” [40 Code of Federal Regulations 261.21(a) (2)]. It is appropriate for pastes, granular material, solids that can be cut into strips, and powdery substances. The test method is described in Appendix A.

5.2 SW-846 Test Method 1050

SW-846 Test Method 1050 provides a test procedure that may be used to evaluate and categorize liquid and solid wastes that are likely to self-heat or spontaneously combust. The test method is described in Appendix B.

5.3 UN O.1 Test: Test for Oxidizing Solids

UN O.1 is designed to measure the potential for a substance to increase the burning rate or burning intensity of a combustible substance when the two are thoroughly mixed. Tests are conducted on the substance to be evaluated mixed with dry fibrous cellulose in mixing ratios of 1:1 and 4:1 by mass of sample to cellulose. The burning characteristics of the mixtures are compared with the standard 3:7 mixture by mass of potassium bromate to cellulose. If the burning time is equal to or less than the standard mixture, the burning times are compared with those from the Packing Group I or II reference standards. The test method is described in Appendix C.

5.4 UN O.2 Test: Test for Oxidizing Liquids

UN O.2 is designed to measure the potential for a liquid substance to increase the burning rate or burning intensity of a combustible substance when the two are thoroughly mixed or to form a mixture that spontaneously ignites. The liquid is mixed in a 1:1 ratio by mass with fibrous cellulose and heated in a pressure vessel while the rate of pressure rise is determined. The test method is discussed in Appendix D.

5.5 Test Method 9095B (Paint Filter)

Test Method 9095B is used to determine the presence of free liquids in a representative sample of waste. The test method is discussed in Appendix E.

6.0 RNS WASTE BLEND TESTS

6.1 Surrogate RNS Waste Blends to Simulate RNS Waste (Blends 1–8, 25)

Nitrate salt and sWheat blends are composed of a WB8 salt or control salt and sWheat. WB8 and the control salt are blended with sWheat in various ratios to simulate the blended nitrate salt material found in RNS drums. The compositions for the surrogate mixtures and the test results from D001 testing are presented in Table 3. The blends were prepared as follows:

- The appropriate volume of sWheat Scoop kitty litter is transferred to the mixing container (4-L Nalgene bottle).
- The appropriate volume of WB8 salt or control salt is measured and added to the mixing container and mixed manually.
- The sWheat scoop kitty litter and salt mixture are heated at an external temperature of 60 °C for 16 h in the Nalgene bottle with the cap attached but not securely closed.

Table 3
RNS Surrogate Waste & Test Results

	Blend Compositions			Test Results		
	KNO3 (g)	WB8 Salt (g)	Salt:sWheat Volume Ratio	SW-846 Test Method 1030	SW-846 Test Method 1050	UN O.1 Testing
Blend 1	50	0	1:0	NA*	NA	Packing Group II
Blend 2	50	0	1:1	Nonflammable	Not self-heating	Packing Group III
Blend 3	50	0	1:3	Nonflammable	Not self-heating	Not Div. 5.1
Blend 4	50	0	1:4	Nonflammable	Not self-heating	Not Div. 5.1
Blend 5	0	50	1:0	NA	NA	Packing Group III
Blend 6	0	50	1:1	Nonflammable	Packing Group III	Not Div. 5.1
Blend 7	0	50	1:3	Nonflammable	Not self-heating	Not Div. 5.1
Blend 8	0	50	1:4	Nonflammable	Not self-heating	Not Div. 5.1
Blend 25	0	50	1:0.5	Nonflammable	Packing Group III	Packing Group II

*NA = Not applicable.

Details of sample preparation and the testing of Blends 1–8 are included in Appendix F (December 22, 2015, SwRI test report). Blend 25 can be found in Appendix G (January 26, 2016, SwRI test report). We note that the WB8 salts are less oxidizing than KNO_3 (Packing Group II versus Packing Group III) yet create self-heating materials when mixed with sWheat, whereas neat KNO_3 does not.

6.1.1 Discussion of RNS Surrogate Waste Test Results

As identified in Table 3, neat nitrate salts are good oxidizers (Blends 1 and 5), and when mixed with sWheat in ratios of 1:1 or greater, exhibit the characteristic of ignitability (WB8 fails the 1050 test and KNO_3 fails the UN O.1 test). It is interesting to note that at higher concentrations of sWheat, the material would not be considered ignitable based on these tests. Because it is not clear what concentration of sWheat was used in the remediation process, the use of sWheat with nitrates supports application of HWN D001 to this waste form. However, estimates of sWheat to salt ratio in drum 68660 were found to be closer to 1:1, consistent with the most reactive materials identified here.[5]

6.2 RNS Surrogate Blends with Zeolite (Blends 9–24)

Blending waste surrogates with zeolite was assessed for effectiveness to remove the D001, ignitable, designation from the RNS waste.

The RNS surrogate wastes prepared for testing and presented in Table 4 were mixed with zeolite and evaluated to understand the effect on ignitability, spontaneous combustion, and oxidizer testing. Recipes tested for zeolite blending with the surrogate RNS waste include volume ratios of 1:0, 1:1, 1:3, and 1:4 RNS waste to salt at constant zeolite addition (3:1 zeolite:RNS). KMI Zeolite, 100% Multipurpose Zeolite (14 × 40 mesh), was used in the testing. Zeolite was manually mixed with the waste surrogate mixtures outlined in Table 4. The blends were then allowed to set for 24 h before evaluation tests were performed.

Table 4
Blending Recipes for RNS Surrogates

	RNS Surrogate Waste Composition			Zeolite Blend Recipe (S/S = salt/sWheat)		Test Results			
Blend #	KNO3 (g)	WB8 Salt (g)	Salt:sWheat Volume Ratio	Water:(S/S) Volume Ratio	Zeolite:(S/S) Volume Ratio	SW-846 Test Method 1030	SW-846 Test Method 1050	UN O.1 Testing	Paint Filter Test Method 9095B
Blend 9	50	0	1:0	NA*	3:1	NA: Salt	NA: Salt	Not Division 5.1	No water added
Blend 10	0	50	1:0	NA	3:1	NA: Salt	NA: Salt	Not Division 5.1	No water added
Blend 11	0	50	1:1	NA	3:1	Nonflammable	Not self-heating	Not Division 5.1	No water added
Blend 12	0	50	1:3	NA	3:1	Nonflammable	Not self-heating	Not Division 5.1	No water added
Blend 13	0	50	1:4	NA	3:1	Nonflammable	Not self-heating	Not Division 5.1	No water added
Blend 14	0	50	1:1	1:1	3:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 15	0	50	1:3	1:1	3:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 16	0	50	1:4	1:1	3:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 17	0	50	1:1	1:1	2:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 18	0	50	1:3	1:1	2:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 19	0	50	1:4	1:1	2:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 20	0	50	1:1	0.5:1	2:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 21	0	50	1:3	0.5:1	2:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 22	0	50	1:4	0.5:1	2:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 23	0	50	1:0.5	0.5:1	3:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids
Blend 24	0	50	1:0.5	0.5:1	2:1	Nonflammable	Not self-heating	Not Division 5.1	No free liquids

*NA = Not applicable.

The addition of water before the addition of zeolite is expected to be beneficial to the blending process as well as to reduce the amount of salt (oxidizer) available for the organic sWheat. The intent is to blend the salt/sWheat mixture with water before it is blended with zeolite. Recipes tested for zeolite blending with the surrogate RNS waste (with water) include volume ratios of 1:0.5, 1:1, 1:3, and 1:4 RNS waste to salt with water added at ratios of 0.5:1 and 1:1, and zeolite to RNS ratios addition of 3:1 and 2:1).

Blends 14 through 24 represent those where water was added to the surrogate waste before it was mixed with zeolite. These blends were prepared as follows:

1. 50 g of the WB8 salt was blended with sWheat kitty litter in the ratios identified in column labeled "Salt:sWheat Volume Ratio" in Table 4 and allowed to rest for 24 h.
2. Once the salt and sWheat blend has rested, it was mixed each with an equal volume of water for 5 min using a mechanical agitator at 500 revolutions per minute (rpm).
3. After the mixture agitated for 5 min, zeolite was added and hand mixed into the solution. The blend was allowed to rest for 24 h before evaluation tests were performed.

Details of sample preparation and the testing of Blends 9 through 24 is presented in Appendix F (December 22, 2015, SwRI test report) and in Appendix G (January 26, 2016, SwRI test report). UN O.1 test for Blend 9 was rerun to verify the initial positive results were incorrect and are reported in Appendix I (March 3, 2016, SwRI test report).

6.2.1 Discussion of Zeolite Blending Test Results

As noted in Table 4, the addition of zeolite in ratios of 2:1 or greater was effective at eliminating the characteristics of ignitability and corrosivity (eliminates liquids). The process was also effective for all blends involving the addition of zeolite using water as a processing aid (our preferred approach as established in Reference 8), demonstrating the robustness of this approach. Note that Blends 9 and 10 represent solid component for UNS, and Blends 11 through 24 represent solid component for RNS blends. This is not surprising given the test results identified in Reference 3, in which zeolite was effective at eliminating the oxidizing potential of nitrates at ratios as low as 1.2:1. Thus, stabilization using zeolite is effective at eliminating D001 and D002 from salt/sWheat mixtures. Its effectiveness for liquids absorbed using sWheat is discussed in Section 6.4.1.

6.3 Cementation of RNS Surrogate Blends (Cmnt 6–15)

Cementation (Cmnt) was the other primary treatment option identified and recommended in the Options Assessment Report[1] and reviewed for implementation at LANL in the Engineering Options Assessment Report.[8] Testing of cementation recipes at the bench scale at LANL and identified in the Engineering Options Assessment Report were examined and evaluated at SwRI.

The first five formulations (Cmnt 1–5) were prepared and had problems with the formulation and setting of the cement. Based upon these observations, the preparation instructions were changed and the tests were modified to ensure that the cement set properly.

A total of 10 cementation tests were conducted. Surrogate blends for cementing the surrogate waste with Type I/II Portland cement are shown in Table 5. All the formulations with sWheat maintain a 4:1 water-to-sWheat mass ratio. Testing at LANL previously used a 3.5:1 water-to-sWheat mass ratio. A higher ratio was used to reduce the amount of RNS waste in the cemented product and to ensure better remediation performance. The water-to-cement mass ratio for the tests ranged from 0.5:1 to 0.75:1. The lower ratio should provide a stronger, drier material.

Salt or the WB8 surrogate and sWheat in the amounts identified in the Table 5 were mixed together and allowed to dry for 16 h at a temperature of 60 to 65 °C. The dried salt/sWheat mixture was then combined with water, the pH adjusted to a solution pH of 9–11 with NaOH, and mixed with Type I/II Portland cement in a high-shear mixer at about 750 rpm until the cement and waste were well blended.

The cement mixtures were placed into a 32-oz container to set and covered with a plastic lid with a small hole for ventilation. The following observations were made every 24 h, starting 24 h after the mixture was placed in the plastic container:

1. Exterior surface temperature taken at the bottom of the container
2. Picture of the top surface of the mortar in the container
3. Penetrometer (Humboldt H-4134) reading of the top surface of mortar sample

The samples were allowed to cure for 10 d at ~22 °C before testing. The set material was crushed to less than 10 mesh and submitted to SW-846 and UN O.1 tests. Reactivity test results for each cement mix are presented in Table 5. Penetrometer and exterior surface temperature readings for each cement mix are included in Appendix G (the SwRI January 26, 2016, results report).

Table 5
Cement Recipes and Reactivity Test Results for Cementation Tests

	RNS Surrogate Waste Composition[a]				Cement Formulation			Test Results		
Cement Mix Number	KNO3 (g)	WB 8 (g)	sWheat (g)	Salt:sWheat Ratio[b]	Water (g)	Cement (g)	Water: Cement Ratio [b]	SW-846 Test Method 1030	SW-846 Test Method 1050	UN O.1 Testing
Cmnt 6	130	0	0	NA[c]	373	495	0.75:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 7	0	130	0	NA	373	495	0.75:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 8	0	225	75	3:1	300	400	0.75:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 9	0	88	88	1:1	352	470	0.75:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 10	0	68	91	0.75:1	364	485	0.75:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 11	0	82	82	1:1	328	504	0.65:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 12	0	72	72	1:1	288	576	0.5:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 13	0	212	70	3:1	282	432	0.65:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 14	0	63	84	0.75:1	336	517	0.65:1	Nonflammable	Not self-heating	Not Div 5.1
Cmnt 15	0	80	80	1:1	320	530	0.60:1	Nonflammable	Not self-heating	Not Div 5.1

[a] Water-to-sWheat mass ratio = 4:1 for all test mixes.

[b] Mass ratio.

[c] NA = Not applicable.

Details of sample preparation and the testing of cement mixes Cmnt 6–15 are presented in Appendix G (January 26, 2016, SwRI test report).

6.3.1 Discussion of Cementation Results

The results of stabilization with cement are found in Table 5. In all cases, stabilization was demonstrated to remove the D001 characteristic for this material. In discussions with transuranic waste experts around the complex, we found that cementation is not a desirable means of treatment because of potential problems associated with dewatering and potential void generation within the material. Given the results we derived with zeolite, this option will not be pursued further.

6.4 UNS Surrogate Solution Blends with sWheat to Simulate RNS Waste (UNS Blends 1–18)

Blending UNS Surrogate Solution with zeolite was assessed for effectiveness. UNS Surrogate Solution represents the solution found in UNS drums. This surrogate solution simulates the free liquid found in the UNS drums and absorbed into sWheat kitty litter during the preparation of the current set of RNS drums located at LANL. The sWheat blends described in this section represent material found in RNS drums.

Because processing at WCRRF often involved neutralizing liquids using Spilfyter Kolorsafe, some of the blend recipes require the addition of Spilfyter Kolorsafe to neutralize the pH of the UNS surrogate solution before it is mixed with sWheat to create a representative surrogate. For these formulations, Spilfyter Kolorsafe liquid acid neutralizer addition is added to achieve an apparent pH ~of 4 to 9 as measured with Hydrion pH paper.

In addition to the RNS waste processed with sWheat, four containers were processed with Waste Lock 770 to absorb the free liquids from UNS parent containers. We have included testing using Waste Lock 770 to establish the effectiveness of adding zeolite for these materials to eliminate the ignitability and corrosivity.

The UNS Surrogate Solution (pH adjusted if called for) and the sWheat or Waste Lock 770 were combined in a beaker and mixed with a spatula for 2 min to absorb the solution. The mixture set for 1 d. After resting for 1 d, the salt/sWheat mix was mixed with water and/or zeolite according to the blend recipe. The blend composition and test results for the UNS surrogate solution blends are presented in Table 6.

Details of preparation of the samples for UNS Blends 1–14, 16, and 18 and the testing for all except UNS Blends 1 and 14 are included in Appendix H (February 3, 2016, SwRI test report.) Details of the testing for UNS Blends 1 and 4 are included in Appendix L (March 29, 2016, SwRI test report).

Table 6
UNS Surrogate Solution Blend Recipes and Test Results

Blend ID	UNS Blend Formulation					Test Results				
	UNS Sol (mL)	sWheat (mL)	Waste Lock 770 (mL)	Water:Waste (Volume Ratio)	Zeolite:Waste (Volume Ratio)	SW-846 Test Method 1030	SW-846 Test Method 1050	UN O.1 Testing	UN O.2 Testing	Paint Filter Test Method 9095B
UNS Blend 1	50	0	0	0	0	NA: Solution	NA:Solution	NA: Solution	Not Div 5.1	NA: Solution
UNS Blend 2	50	50	0	0	0	Nonflammable	DOT Packing Grp III	Not Div 5.1	NA	No free liquids
UNS Blend 3	50	150	0	0	0	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 4	50*	50	0	0	0	Nonflammable	DOT Packing Grp III	Not Div 5.1	NA	No free liquids
UNS Blend 5	50*	150	0	0	0	Nonflammable	DOT Packing Grp III	Not Div 5.1	NA	No free liquids
UNS Blend 6	50	50	0	0.5:1	3:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 7	50	150	0	0.5:1	3:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 8	50*	50	0	0.5:1	3:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 9	50*	150	0	0.5:1	3:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 10	50	50	0	0.5:1	2:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 11	50	150	0	0.5:1	2:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 12	50*	50	0	0.5:1	2:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 13	50*	150	0	0.5:1	2:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 14	50	0	3.12	0	0	NA: Solution	NA: Solution	NA: Solution	Not Div 5.1	NA: Solution
UNS Blend 16	50	0	3.12	0	3:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids
UNS Blend 18	50	0	3.12	0	2:1	Nonflammable	Not self-heating	Not Div 5.1	NA	No free liquids

Notes: DOT = U.S. Department of Transportation. NA = Not applicable.

* pH-adjusted solution (Spilfyter Kolorsafe).

6.4.1 Discussion of UNS Liquid Surrogate Testing

As Table 6 indicates, almost all of the liquid surrogate/sWheat mixtures exhibited the characteristic of ignitability through failure of the 1050 test and are classified as self-heating solids. In reviewing the data, we are concerned that UNS Blend 2 yielded a suspect result and will be re-run at a later date (the material expanded significantly but likely did not exceed 200 °C, a criterion of the 1050 test). However, we note that the addition of the Spilfyter Kolorsafe led to materials that yielded temperatures in excess of 700 °C in the 1050 test, demonstrating the increased chemical reactivity of the salt when neutralized with triethanolamine. The addition of zeolite and use of water as a processing aid demonstrated the elimination of both the ignitability (D001) and corrosivity (D002) characteristics for the surrogate liquid and demonstrated the effectiveness of this stabilization method for these waste forms. Material was not tested without the addition of water because water used as a processing aid was identified before these tests were executed and was determined to be the optimum processing option as discussed in Reference 8. Finally, we note that the surrogate solutions did not exhibit the characteristic of ignitability as oxidizers. While many of the nitrate salts are not oxidizers in solution (e.g., $CaNO_3$ [Reference 9]), the ratio of zeolite to liquid is greater than the original remedy proposed by the Difficult Waste Team[10] and should be effective. However, we will conduct additional tests with oxidizing nitrate liquids to verify the effectiveness of the remedy.

7.0 DEBRIS TESTING

The RNS waste drums contain debris typically composed of plastic, cardboard, rubber gloves, rags, and lead. It is unclear if the debris that has comingled with RNS waste should carry the D001 code for an oxidizer. To examine this aspect of the waste stream, various tests were performed. Samples of the debris types commonly found in RNS waste drums were subjected to environments that simulate the conditions in the RNS drums and tested to see how they respond to SW-846 Test Methods 1030 and 1050. The debris waste evaluated included cardboard liner, plastic bags, rubber gloves, and Wypall rags.

7.1 Effect of UNS Liquid on Organic Debris

UNS Surrogate Solution was prepared and used to soak test samples of the different types of debris waste. Cardboard, plastic, rubber glove, and rag samples were submerged for 2 h in the UNS Surrogate Solution and then submitted for SW-846 Test Methods 1030 and 1050. Samples of the cardboard liner, plastic, and rubber glove were soaked and then allowed to drain for 1 h, after which they were submitted for testing. The Wypall rags were soaked, squeezed to remove excess moisture, and then submitted for testing.

Test results for the debris soaked in UNS Surrogate Solution are presented in Table 7.

7.2 Effect of RNS Waste on Organic Debris

RNS waste surrogate was prepared using WB8 salt surrogate and sWheat to coat the debris samples. A 1:1 by volume blend of WB8 salt-to-sWheat surrogate was prepared and comingled with the debris to simulate conditions found in the RNS drums

The WB8 salt-to-sWheat surrogate was impregnated onto the debris samples using a rolling pin. The surrogate RNS material was rolled onto the organic debris in an attempt to impregnate the debris with the RNS surrogate. After the material was rolled, the excess surrogate was shaken off. The debris samples were then submitted for SW-846 Test Methods 1030 and 1050 along with samples of untreated materials.

Test results for the debris impregnated with surrogate RNS material are presented in Table 7.

Table 7
Debris Reactivity Test Results

Debris Type	Debris Treatment	SW-846 Test Method 1030	SW-846 Test Method 1050
Cardboard	None	Nonflammable	Not self-heating
Cardboard	Soak in UNS Solution	Nonflammable	Not self-heating
Cardboard	Soak in UNS Solution	Nonflammable	Not self-heating
Cardboard	Comingle with RNS	Nonflammable	Not self-heating
Cardboard	Comingle with RNS	Nonflammable	Not self-heating
Plastic	None	Nonflammable	Not self-heating
Plastic	Soak in UNS Solution	Nonflammable	Not self-heating
Plastic	Soak in UNS Solution	Nonflammable	Not self-heating
Plastic	Comingle with RNS	Nonflammable	Not self-heating
Plastic	Comingle with RNS	Nonflammable	Not self-heating
Rubber glove	None	Nonflammable	Not self-heating
Rubber glove	Soak in UNS Solution	Nonflammable	Not self-heating
Rubber glove	Soak in UNS Solution	Nonflammable	Not self-heating
Rubber glove	Comingle with RNS	Nonflammable	Not self-heating
Rubber glove	Comingle with RNS	Nonflammable	Not self-heating
Wypall rag	None	Flammable with rate of 3.52 mm/s	Not self-heating
Wypall rag	Soak in UNS Solution	Nonflammable	DOT Packing Group II
Wypall rag	Soak in UNS Solution	Nonflammable	No duplicate test
Wypall rag	Comingle with RNS	Nonflammable	Not self-heating
Wypall rag	Comingle with RNS	Nonflammable	Not self-heating

Note: DOT = U.S. Department of Transportation.

Details of the preparation of the samples testing of debris surrogates can be found in Appendix J (March 11, 2016, SwRI test report).

7.3 Discussion of Debris Test Results

Debris samples provided by LANL from actual materials used during the preparation of the RNS waste drums were treated with UNS solution and RNS surrogate waste to determine if the materials might be considered ignitable (D001). The conditions the test materials were subjected to were far harsher than what actual materials will endure in the RNS waste drums before they are remediated. The debris was submerged for 2 h during the UNS solution testing and physically impregnated with RNS-blended salt/sWheat material to simulate contamination with RNS waste material. The tested samples were evaluated in duplicate to ensure results could be replicated, unless a positive result was achieved. It is clear from available RTR information that the debris will not encounter free liquids nor will it be forcefully impregnated with RNS waste material before remediation.

All the tested debris material passed the SW-846 Test Methods 1030 and 1050 testing, except the Wypall rags. This is an expected result because the rags are cellulose-based and are absorbent. The other materials are less absorbent and less cellulose-based materials.

Based upon these results, a process will be developed and evaluated to treat the Wypall rags to ensure they are not ignitable. This approach will utilize blending with water and zeolite to take advantage of the equipment and materials already intended to be used for the RNS waste. Additional testing will be planned to verify that this process is effective and the resulting product passes the reactivity tests. The other debris waste tested (plastic, cardboard liner, rubber gloves) will be not be considered D001 and will not be treated when encountered during remediation of the RNS drums.

8.0 CONCLUSIONS

Small scale, EPA SW-846 and UN DOT testing was conducted to (1) establish the characteristics of surrogates of nitrate salt mixtures and to ensure they bound the expected waste form and (2) establish the effectiveness of stabilization technologies against the expected components found in both the UNS and RNS waste forms. The preferred option, stabilization using zeolite that invokes water as a processing aid, was found to be effective at eliminating both characteristics of D001 and D002 for these materials. Cementation was found to be effective at removing D001 for a subset of the waste stream. Further testing was suspended after the effectiveness of adding zeolite was demonstrated and through consultation with experts on transuranic waste who identified potential issues with cementation as a remedy (dewatering, void generation, etc.).

REFERENCES

[1] B. A. Robinson and P. A. Stevens, "Options Assessment Report: Treatment of Nitrate Salt Waste at Los Alamos National Laboratory," LA-UR-15-27180, Los Alamos National Laboratory, Los Alamos, NM, 2015.

[2] D. L. Clark and D. J. Funk, "Chemical Reactivity and Recommended Remediation Strategy for Los Alamos Remediated Nitrate Salt (RNS) Wastes," LA-UR-15-22393, Los Alamos National Laboratory, Los Alamos, NM, February 17, 2015.

[3] G. Walsh, Research Scientist, Energetic Materials Research and Testing Center, New Mexico Institute of Mining and Technology, Socorro, New Mexico (Certified DOT Testing Laboratory), "Results of Oxidizing Solids Testing, EMRTC Report RF 10-13," prepared for Washington TRU Solutions, LLC, March 12, 2010.

[4] K. Weisbrod, K. Veirs, D. J. Funk, and D. L. Clark, "Salt Composition Derived from Veazey Composition by Thermodynamic Modeling and Predicted Composition of Drum Contents," LA-UR-16-21651, Los Alamos National Laboratory, Los Alamos, NM, 2016.

[5] G. Brown, "Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing Standard Procedure," PLAN-TA-9-2443 (U), Rev. B, and the associated memorandum, "M7-16-6042, Rev. 1, "Results from First Round of Remediated Nitrate Salt Surrogate Formulation and Testing," 2016.

[6] G. R. Parker, M. D. Holmes, E. M. Heatwole, P. Leonard, and C. P. Leibman, "Drum-scale Testing of the Thermolytic Response of a Remediated Nitrate Salt (RNS) Surrogate Waste Mixture," LA-CP-16-20038, Los Alamos National Laboratory, Los Alamos, NM, 2016.

[7] D. J. Funk, "Technical Basis for the Removal of Unremediated Nitrate Salt Sampling (UNS) to Support LANL Treatment Studies," LA-UR-16-21640, Los Alamos National Laboratory, Los Alamos, NM, 2016.

[8] K. Anast, "Engineered Option Treatment of Remediated Nitrate Salts: Surrogate Batch-Blending Testing," LA-UR-16-21653, Los Alamos National Laboratory, Los Alamos, NM, 2016.

[9] United Nations, "Recommendations on the Transport of Dangerous Goods Manual of Tests and Criteria," Fifth Revised Edition, United Nations, New York and Geneva, 2009.

[10] LANL-Carlsbad Office Difficult Waste Team, "Amount of Zeolite Required to Meet the Constraints Established by the EMRTC Report RF 10-13: Application to LANL Evaporator Nitrate Salts," May 8, 2012.