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

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# Treatment Study Plan for Nitrate Salt Waste Remediation

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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

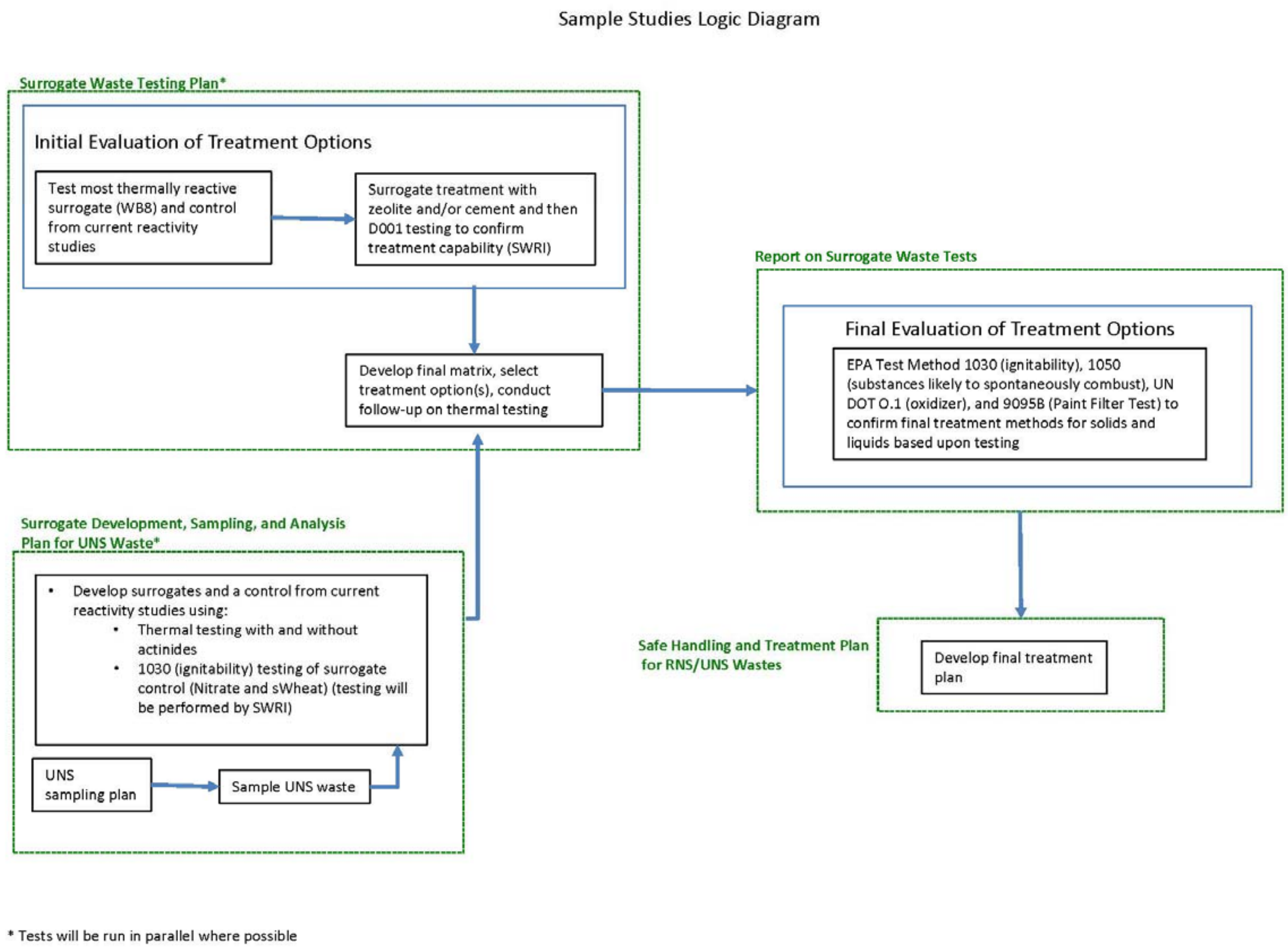
Twenty nine unremediated nitrate salt waste parent containers and 60 remediated nitrate salt waste containers must undergo treatment prior to off-site disposal. The objective of this study is to determine which treatment method, zeolite addition or cementation, will be most effective at safely removing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