LA-UR-16-21746
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| Title: | PLAN-TA9-2443(U), Rev. B Remediated Nitrate Salt (RNS) Surrogate Formulation and Testing Standard Procedure |
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| Intended for: | Needed for the NMED Submittal UNS Nonsample Technical Justification |
| Issued: | 2016-03-16 |

Los Alamos NATIONAL LABORATORY EST. 1943

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

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

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

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

**Document Owner**

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**Revision History**

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

# 1.0 INTRODUCTION

## 1.1 Purpose

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

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

This Test Plan describes the requirements, responsibilities, and process for preparing and testing a range of chemical surrogates intended to mimic the energetic response of waste created during processing of legacy nitrate salts. The surrogates developed are expected to bound1 the thermal and mechanical sensitivity of such waste, allowing for the development of process parameters required to minimize the risk to worker and public when processing this waste.  Such parameters will be based on the worst-case kinetic parameters as derived from APTAC measurements as well as the development of controls to mitigate sensitivities that may exist due to friction, impact, and spark. This Test Plan will define the scope and technical approach for activities that implement Quality Assurance requirements relevant to formulation and testing.  This Test Plan conforms to ASME NQA-1-2009A, Subpart 4.2, “Guidance on Graded Application of the Nuclear Quality Assurance (NQA) Standard for Research and Development”.

## 1.2 Scope

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

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

## 2.0 PRECAUTIONS AND LIMITATIONS

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

## 3.0 PREREQUISITES

### 3.1 Prerequisite Actions

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

### 3.2 Training

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

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

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

## 4.0 PROCEDURE

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

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

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

### 4.1 Surrogate Salt Formulation

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

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

**Nominal Formulation**

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

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

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

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

| | | |
|---|---|---|
| 4% $Pb(NO_3)_3$;<br>15% Swheat | 4% $Pb(NO_3)_3$;<br>25% Swheat | 4% $Pb(NO_3)_3$;<br>35% Swheat |
| 2% $Pb(NO_3)_3$;<br>15% Swheat | 2% $Pb(NO_3)_3$;<br>25% Swheat | 2% $Pb(NO_3)_3$;<br>35% Swheat |
| 1% $Pb(NO_3)_3$;<br>15% Swheat | 1% $Pb(NO_3)_3$;<br>25% Swheat | 1% $Pb(NO_3)_3$;<br>35% Swheat |

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

## 4.2 Formulation

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

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

### 4.3 Sensitivity Testing

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

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

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

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

**4.3.5** The DSC instrument and software operation are verified using an Indium standard supplied by the vendor and traceable to the National Physical Laboratory in the UK. The indium scan verifies the temperature measurement capability of the instrument and the enthalpy measurement capability. For this work we will request that the instrument operation be checked by indium both before and after running the samples. There are no other process aids or equipment that significantly influence the temperatures and enthalpies measured by DSC. The model and serial number of the DSC and balance used for the testing will be recorded in the laboratory report.

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

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

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

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

## 4.4 APTAC Testing

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

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

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

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

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

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

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

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

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

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

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

## 5.0 QUALITY ASSURANCE

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

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

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

## 6.0 NONCONFORMANCES

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

## 7.0 DOCUMENT MANAGEMENT

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

## 8.0 TEST PLAN REVIEW AND APPROVAL

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

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

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

## 9.0 TEST PLAN CHANGES

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

## 10.0 RECORDS AND RECORD REQUIREMENTS

Records compiled or generated by this process include:

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

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

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

## 11.0 SOFTWARE QUALITY MANAGEMENT

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

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

## 12.0 ENVIRONMENT, SAFETY, AND HEALTH

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

## 13.0 RESPONSIBILITIES

### 13.1 Responsible Line Manager

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

### 13.2 Principal Investigator

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

### 13.3 Technical Reviewer

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

### 13.4 Document Control

- Assigns document number and effective date for this Test Plan

### 13.5 Worker

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

## 14.0 ACRONYMS

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

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

## 15.0 ATTACHMENTS

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

## Attachment A: SURROGATE RECIPES

Recipes with 15% SWheat.

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

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

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

## Attachment A: SURROGATE RECIPES (cont'd)

Recipes with 25% SWheat.

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

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

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

## Attachment A: SURROGATE RECIPES (cont'd)

Recipes with 35% SWheat.

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

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

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

## Attachment B: TEST DESCRIPTIONS

### Differential Scanning Calorimetry (DSC)

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

### Drop Weight Impact (DWI)

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

### Friction Sensitivity

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

### Electrostatic Spark Discharge Sensitivity (ESD)

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

## Attachment B: TEST DESCRIPTIONS (cont'd)

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

### Automatic Pressure Tracking Adiabatic Calorimetry (APTAC)

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

## Attachment C - QUALITY IMPLEMENTATION MATRIX

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

## Attachment C: QUALITY IMPLEMENTATION MATRIX

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

## Attachment C: QUALITY IMPLEMENTATION MATRIX

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

## Attachment C: QUALITY IMPLEMENTATION MATRIX

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

## Attachment C: QUALITY IMPLEMENTATION MATRIX

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

## Attachment C: QUALITY IMPLEMENTATION MATRIX

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

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