Appendix E
FCT Document Cover Sheet

Name/Title of Deliverable/Milestone	Test Plan Revision Based on External Review and Prioritized Test Matrix
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This deliverable was prepared in accordance with Sandia National Laboratories
(Participant/National Laboratory Name)

QA program which meets the requirements of
☒ DOE Order 414.1 ☐ NQA-1-2000

This Deliverable was subjected to:

☒ Technical Review

☐ Peer Review

Technical Review (TR)

Review Documentation Provided

☐ Signed TR Report or,
☒ Signed TR Concurrence Sheet or,
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Peer Review (PR)

Review Documentation Provided

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Name and Signature of Reviewers

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Sandia National Laboratories[1]
Fuel Cycle Technology (FCT) Program

Consolidation of Crushed Salt at Temperatures up to 250°C, under Hydrostatic and Shear Stresses, Rev. 1

Effective Date: 06/06/2012

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1 *Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.*

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1 ABBREVIATIONS, ACRONYMS, AND INITIALISMS

Cal Lab	SNL Primary Standards Calibration Laboratory
DAS	Data Acquisition System
DOE	Department of Energy
ES&H	Environmental Safety and Health
HA	Hazard Analysis
NQ	non-QA
PA	Performance Assessment
PHS	Primary Hazard Screening
PI	SNL Principal Investigator, or Designee
QA	Quality Assurance
QAPD	QA Program Document
SNL	Sandia National Laboratories
SP	Activity/Project Specific Procedure
US	United States
TRU	Transuranic
WIPP	Waste Isolation Pilot Plant

2 REVISION HISTORY

This is the first revision (Revision 1) of this Test Plan. Future revisions will be documented and appear in this section, as applicable. Changes to this Test Plan, other than those defined as editorial changes per Sandia National Laboratories, shall be reviewed and approved by the same organization that performed the original review and approval.

3 PURPOSE AND SCOPE

Reconsolidation of crushed salt is a very important physical phenomenon when backfilling or sealing nuclear waste repositories in salt is considered. There is a long history of testing crushed salt backfill for salt repository applications. Callahan (1999) summarized the constitutive model for room temperature consolidation application to the shaft seal system at the Waste Isolation Pilot Plant (WIPP). Over the years, salt reconsolidation has been a topic of great interest to international salt repository studies as exemplified at symposia (for example: Aubertin and Hardy, 1996 and Wallner et al., 2007). A preponderance of these studies has been at room temperature, with a few tests at elevated temperatures up to 100°C. Today there is a renewed national and international interest in salt reconsolidation at elevated temperature, particularly as applied to disposal of heat-generating nuclear waste. This Test Plan puts forward the experimental procedure for a laboratory study of reconsolidation of crushed salt, emphasizing testing at elevated temperature.

The primary purpose of these experiments is to quantitatively evaluate consolidation as a function of stress and temperature. A secondary purpose is to establish the deformational processes by which the salt reconsolidates. To successfully complete these experiments, challenging geomechanics techniques will have to be developed. The salt used in these experiments is "mine-run," which means the aggregate was produced during normal mining operations. The planned laboratory studies are intended to provide data representing consolidation behavior as a function of stress state and temperature up to 250°C. The deformational processes will be determined by microscopic examination of the salt substructures after the tests. Other related components of crushed salt testing, such as thermal conductivity as a function of porosity, will be pursued under separate Test Plans. Thus, some of the consolidated samples created as a result of these tests may be used for thermal conductivity testing at a later date.

The reconsolidation model developed by Callahan (1999) is quite sophisticated. It accounts for the effects of moisture through pressure solution and dislocation creep, with both terms dependent on effective stress to account for the effects of porosity. At the limit of zero moisture content, the pressure solution term contributes nothing to the strain rate and at the limit of zero porosity the dislocation creep term becomes equal to the WIPP model for intact salt. Little work has been done on crushed salt at temperatures as high as 250°C, which are expected to dry the salt. One hypothesis is that temperatures significantly above 100°C will drive off all surface-bound water and thereby limit pressure solution effects. There may be some water trapped in the fluid inclusions found within the crystal structure, but may remain trapped. As part of these studies, we will examine the effect of temperatures up to 250°C on the weight loss of a subsample to verify the assumptions regarding water loss as a function of temperature.

The consolidation of granular salt will be examined using both hydrostatic and shear stresses to compact the crushed salt. Hydrostatic refers to the application of an isotropic stress state, usually by means of immersing the jacketed sample in a pressurized fluid. Shear stress control is provided by two independent systems: an axial loading ram to apply stress along one axis of the sample and fluid pressure applied uniformly to the specimen as in the hydrostatic tests. The loading ram is a standard hydraulic actuator driven by a servo valve, and either the ram position or the load on the ram can be used as the feedback control variable. Fluid pressure in the vessel is controlled by a constant-pressure intensifier, which also functions as a dilatometer, making it possible to measure volume changes of samples. Consolidation under shear stress is a more realistic representation of the consolidation expected in alcove disposal where the roof-to-floor closure is expected to be faster than the rib-to-rib closure.

The following objectives are of interest:

- Acquire consolidation data for mine-run salt for temperatures up to 250°C
- Assess the impact of dryness on the consolidation process
- Evaluate the micromechanical deformation processes through optical microscopy and other imaging techniques, e.g., scanning electron microscopy (SEM)

- Develop test techniques for geomechanics experiments above 100°C.

The activities described in this Test Plan will help assess processes of salt consolidation. In turn, the data will be used to evaluate a model for reconsolidation of crushed salt subject to elevated temperature and repository-relevant pressures. Tests will be carried out to investigate the effects of hydrostatic stress on mine-run salt at temperatures up to 250^{o}C, pressures up to 20 MPa, and stress differences up to 15 MPa. The stresses are chosen to correspond to in situ stresses encountered at commercial salt mining operations, but may be adjusted by the Principal Investigator (PI) as more information becomes available. Our primary goal is to investigate the effects of temperatures above 100^{o}C. Because of the high temperatures and venting of the samples, we expect reconsolidation processes may minimize the effective pressure-solution redeposition process because available and accessible water is removed from the grain boundaries. The ideas of drying and potential change of consolidation mechanisms are hypotheses pertaining to crushed salt in a heat-generating salt repository. Such experimental work was identified to be of great interest to national and international salt repository programs (Hansen and Leigh, 2011).

The work will be done to QA standards as delineated in Chapter 9 of this Test Plan. In all cases, this work will follow good scientific practice including: maintaining current instrument calibrations, following a documented procedure for sample preparation, and preservation of samples in their lead jackets for use by later investigators (such as microscopy or thermal conductivity). The post-test microscopy methods are identified in Section 4.2.

The planned test matrix is in Table. The test matrix was derived from extreme near-field repository conditions discussed in Carter et al (2011). Repetition of tests is planned but the number of repeated tests will be determined by the PI based on data and observational work. Three types of tests are listed; hydrostatic, shear, and creep.

In isostatic tests, the jacketed specimen is placed in the pressure vessel, heated to the test temperature (and held there overnight to ensure that the entire sample has reached the test temperature), and then hydrostatic pressure is increased at a predetermined rate. At predetermined pressure levels the confining pressure is cycled down and then back up. This allows assessment of the evolution of elastic properties as compaction occurs (increasing density/decreasing porosity).

In shear tests, the jacketed specimen is placed in the pressure vessel, heated to the test temperature (and held there overnight to ensure that the entire sample has reached the test temperature), and then isostatic pressure is increased at a predetermined rate. Once test pressure is reached, the ram is advanced into the pressure vessel until it contacts the specimen, increasing the axial force/stress on the specimen. At predetermined displacement levels the axial force is cycled down and then back up. This allows assessment of the evolution of elastic properties as compaction and shear stress occur (increasing density/decreasing porosity).

In creep tests, the jacketed specimen is placed in the pressure vessel, heated to the test temperature (and held there overnight to ensure that the entire sample has reached the test temperature), and then isostatic pressure is increased at a predetermined rate. Once test pressure is reached, the ram is advanced relatively quickly into the pressure vessel until it contacts the specimen, increasing the axial force/stress on the specimen; the axial stress is maintained constant for the duration of the test.

The test matrix reflects an attempt to obtain more than one type of test result from single samples. For example, tests are described in the comment section as phases because a single test may contain isostatic, shear, and creep phases to maximize mechanical information from individual samples.

The values in Table 1 are selected to emphasize portions of the parameter space well outside current experience. Alternatives such as stepped stress tests or stepped temperature tests may be considered, at the discretion of the PI, as a way of extracting more information from each test.

At the end of a test, the high-temperature, high-pressure test conditions are diminished as quickly as possible in an attempt to freeze the microstructure developed during the deformation. In practice, once the desired load or displacement is reached, the sample is unloaded to an isostatic stress state, the heaters are turned off and then the isostatic pressure is reduced to zero within a few minutes. It then takes a few hours for the vessel and sample to cool to room temperature.

Table 1. Recommended Test Matrix.

Test Number	Test Type	T[(a)] (°C)	Maximum Confining Pressure[(b)] (MPa)	Axial Stress[(c)] (MPa)	Mean Stress (MPa)	Stress Difference (MPa)	Description[(d)]	Comment
1	Isostatic	100	2.5	2.5	2.5	0.0	Quasistatic	Phase 1 of Test 16
2	Isostatic	100	5.0	5.0	5.0	0.0	Quasistatic	Phase 1 of Test 17
3	Isostatic	175	2.5	2.5	2.5	0.0	Quasistatic	Phase 1 of Test 18
4	Isostatic	175	5.0	5.0	5.0	0.0	Quasistatic	Phase 1 of Test 19
5	Isostatic	250	2.5	2.5	2.5	0.0	Quasistatic	Phase 1 of Test 20
6	Isostatic	250	5.0	5.0	5.0	0.0	Quasistatic	Phase 1 of Test 21
7	Isostatic	250	20.0	20.0	20.0	0.0	Quasistatic	
8	Isostatic	250	10.0	10.0	10.0	0.0	Quasistatic	Phase 1 of Test 9
9	Shear	250	10.0	20.0	13.33	10.0	Quasistatic	Phase 2 of Test 8
10	Shear	100	2.5	5.0/7.5	3.33/4.17	2.50/5.0	Quasistatic	Phase 2 of Test 16
11	Shear	100	5.0	7.5/10.0	5.83/6.67	2.50/5.0	Quasistatic	Phase 2 of Test 17
12	Shear	175	2.5	5.0/7.5	3.33/4.17	2.50/5.0	Quasistatic	Phase 2 of Test 18
13	Shear	175	5.0	7.5/10.0	5.83/6.67	2.50/5.0	Quasistatic	Phase 2 of Test 19
14	Shear	250	2.5	5.0/7.5	3.33/4.17	2.50/5.0	Quasistatic	Phase 2 of Test 20
15	Shear	250	5.0	7.5/10.0	5.83/6.67	2.50/5.0	Quasistatic	Phase 2 of Test 21
16	Shear	100	2.5	5.0/7.5	3.33/4.17	2.50/5.0	Creep	Phase 3 of Tests 1 & 10
17	Shear	100	5.0	7.5/10.0	5.83/6.67	2.50/5.0	Creep	Phase 3 of Tests 2 & 11
18	Shear	175	2.5	5.0/7.5	3.33/4.17	2.50/5.0	Creep	Phase 3 of Tests 3 & 12
19	Shear	175	5.0	7.5/10.0	5.83/6.67	2.50/5.0	Creep	Phase 3 of Tests 4 & 13
20	Shear	250	2.5	5.0/7.5	3.33/4.17	2.50/5.0	Creep	Phase 3 of Tests 5 & 14
21	Shear	250	5.0	7.5/10.0	5.83/6.67	2.50/5.0	Creep	Phase 3 of Tests 6 & 15

[a] T = Temperature
[b] For quasistatic tests, stresses are increased from zero to the prescribed value with periodic unload/reload cycles for elastic property determination. The axial stress value is the same as the confining pressure value in the isostatic tests and greater than the confining pressure in the shear tests.
[c] For the shear loaded creep tests, both axial stress differences ($\Delta\sigma$ = 2.5 and 5.0 MPa) will be run at the lower temperature values, but only the lower stress difference may be run at the higher temperatures depending on prior results.
[d]The term "quasistatic" is used to describe tests wherein stresses are applied rapidly to minimize time-dependent effects. The term "creep" is used to describe tests wherein stresses are held constant at the prescribed test conditions for the duration of the test.

4 EXPERIMENTAL PROCESS DESCRIPTION

This section describes the experimental processes required to study consolidation of crushed salt when subjected to stress and temperature that may be applicable to salt repositories. Standard geomechanics techniques will be used for the most part, with some specialized techniques to acquire volume strain data and jacketing rugged enough to withstand the test temperatures and high deformations imposed by the large particle size of the crushed salt.

4.1 Description of the Proposed Experiments

4.1.1 Relevance of earlier work

Previous work aimed at understanding consolidation of crushed salt was focused on a lower temperature regime than is of interest in this work. It is planned that sample preparation, testing techniques, data acquisition, and some aspects of jacketing will be very similar to that earlier work. Material for preparation of samples was obtained using mine-run salt from WIPP. The following description of procedure is extracted from Holcomb and Zeuch (1990), updated to reflect changes in apparatus.

4.1.2 Preparation of salt

From previous work, the mass of salt in each sample is estimated to be ~1600 g, assuming a cylindrical sample ~10 cm in diameter and ~15 cm in length, and assuming no salt tamping is done to increase starting density. To ensure consistency of the tested material, sufficient material will be pre-sieved to make the planned specimens. Using a customary ratio of particle size to specimen size of at least 10 requires that the mine-run salt be sieved through a 10-mm screen, or the closest standard sieve, which is 9.5 mm. After all material is passed through a 9.5-mm sieve to remove large grains, it will be divided into 1700-g lots, each of which will constitute the material for one sample (although not all salt from each lot will be used). Each lot will be passed through an additional 10 sieves of mesh size 6.35 mm, 4.75 mm, 4.00 mm, 3.35 mm, 2.80 mm, 2.36 mm, 2.00 mm, 1.40 mm, 1.00 mm, and 0.60 mm. For every sample, the material collected in each sieve will be weighed in order to accurately characterize the particle size distribution and for comparison with results from previous work by Holcomb and Hannum (1982)

(Table 2). Sieving is done to establish the grain size of starting material, not to construct samples with pre-determined grain size distributions.

After sieving, each lot will be dried at 105°C for several days, or until weight loss stabilizes, as determined by repeated weighing. Previous experience showed a mass loss of about 0.25% after the first day of heating at 105°C, with minimal further loss after three more days of heating. In previous work, after the mass stabilized at 105°C, a subsample was heated to 175°C and 250°C to determine if further moisture would be driven off. A small amount of additional moisture evaporated at 175°C and at 250°C, resulting in a final moisture loss of 0.3%, as indicated by change in sample mass.

As all testing will be done at 100°C or higher, moisture content should remain low in tested salt, but adsorbed moisture will affect sample preparation and sample mass. The test series will be lengthy, so the planned procedure, in order to restrict re-adsorption of moisture onto the salt, is to store samples in the oven at 105°C until they can be tested. Immediately before testing begins, the salt will be removed from the oven, loaded into a lead jacket, and sealed as described in section 4.1.3. If sample material needs to be removed from the oven for extended periods of time, an airtight storage container with desiccant packs should be used for storing the salt. Plastic bags are insufficient to keep the salt dry, as most plastic bagging material is relatively permeable to atmospheric moisture.

4.1.3 Jacket Construction

In prior work, samples were prepared using the procedure described by Holcomb and Hannum (1982), but it was soon found that the Viton jacketing material was not suitable for temperatures in excess of 100°C. Figure 1 shows details of the current design of an assembled sample. As discussed later, a major technical obstacle to obtaining high quality results from these tests is the difficulty of making precise determinations of the sample porosity during the course of the test using indirect methods. Applying the various required corrections and calibrations is significantly easier if the samples are prepared to be as nearly identical as possible.

Samples will be constructed as right circular cylinders, using 10.16-cm-diameter aluminum end caps. A beveled face plate is placed between each end cap and the salt, and the top end is vented via a nipple to the vessel exterior. The bevel is designed to accommodate the large decreases in jacket diameter that occur as the salt compacts, leading to high shear between the undeformed jacket overlapping the end cap and the deformed jacket contacting the salt.

A pressed steel mesh (“felt metal”) is placed on top of the crushed salt, beneath the upper beveled plate. This mesh enables rising air or fluid to move laterally towards the vent in the center of the upper end cap, instead of being trapped or obstructed at the perimeter of the cylinder where the salt meets the end cap.

Two lead jackets are used to contain and protect the sample, each consisting of a 1.6-millimeter-thick lead sheet. The inner jacket is 16.5 cm tall and forms the cylinder that directly holds the salt. The cylinder has a slightly smaller diameter than the beveled plates, such that it rests on the beveled surface and the plates protrude a short way into the cylinder. The outer jacket is about 24.5 cm tall and is used as an exterior layer to exclude the silicone confining pressure medium (e.g. Dow Chemical Co. Dowtherm A) from the sample cell. Double-wall lead jacketing is necessary to cope with jacket intrusions into the large void spaces that unavoidably exist between the coarse salt grains.

Table 2 Sieve analysis for results from Holcomb and Hannum, (1982).

Sieve (mm)	% retained	% passed
10.00	0.00	100.00
6.35	20.50	79.50
4.75	9.50	70.00
3.99	5.90	64.10
3.55	6.90	57.20
2.82	7.20	50.00
2.38	6.20	43.80
2.00	3.80	40.00
1.41	10.1	29.90
1.00	8.10	21.80
0.59	8.10	13.70
Pan	13.70	0.00

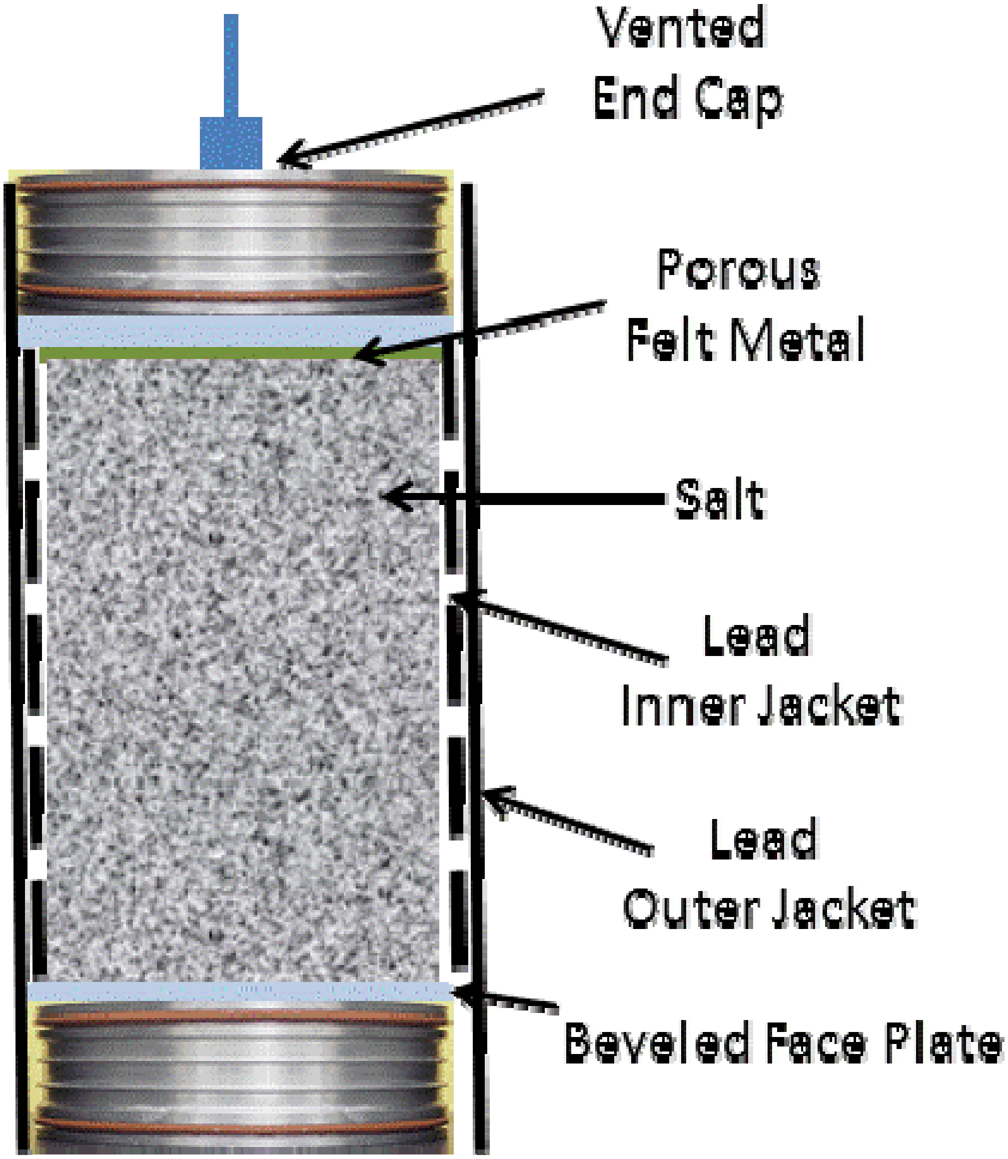

Figure 1 Cross section schematic of an assembled sample.

Preformed lead tubing will be used for the outer jackets, with the length adjusted as needed to approximately 24.5 cm. Lead has been shown to be a suitable jacket material, although it has an upper temperature limit set by its melting point of 325°C. Although lead is capable of forming a pressure-tight jacket, it does not form a pressure-tight seal at the end caps without additional work. Typically O-rings are used to create a seal, but at the required temperatures for these tests, O-rings have proven to be unreliable, due in large part to the long thermal soak time required to bring the sample up to temperature. Either the O-rings will not form a seal with the lead, or the combination of high temperature and silicone fluid turn the O-rings to jelly. An effective solution requires eliminating the reliance on elastomers to form a seal.

A metal-to-metal sealing system using only the lead jacket and aluminum end caps has been tested and shown reliable under the conditions anticipated for these tests. New end caps were machined with a pair of grooves for O-rings and a pair of sharp “knife-edge” ridges between the O-rings. The lower end cap is inserted into the outer jacket and the assembly is placed in a vessel that is pressurized with nitrogen to about 1000 psi. The O-rings create a sealed volume around the ridges, preventing pressure in that volume from equilibrating with pressure in the vessel. As a result, the gas pressure on the exterior of the sealed volume deforms the lead until it conforms to the ridges. This lead-aluminum contact forms a high-pressure, high-temperature seal: It cannot be bypassed by silicone oil and is unaffected by temperatures up to 250°C. Once the metal-to-metal seal has formed, the O-rings remain in place but are not an important component of the seal. **Error! Reference source not found.** shows the elements of this sealing system. The diagram shows only the edge of a circular end cap.

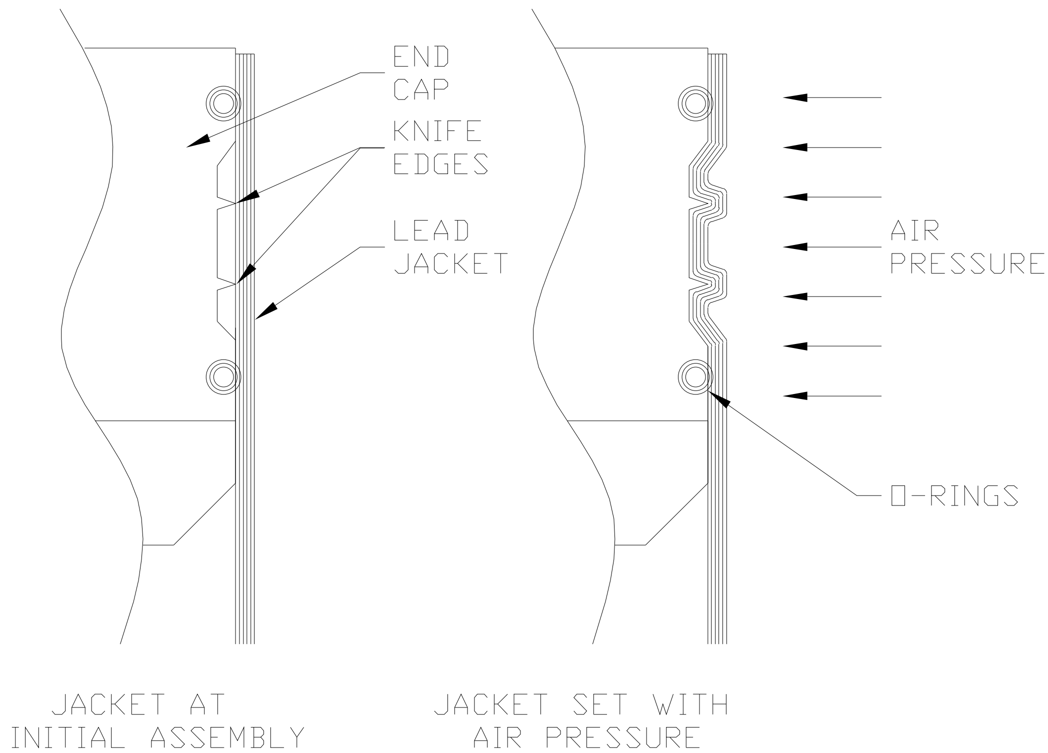

Figure 2 Schematic of the sealing region.

After the lower end cap is sealed, the salt and all other components of the sample can be assembled, and the upper end cap inserted into the jacket. The entire sample is placed inside the pressure vessel, where gas pressure is used to seal the upper end cap in the same manner as the lower end cap. As the gas has access to the interior of the sample through the vent in the upper end cap, pressure has no effect on the salt or lead jacket in the region outside the O-rings.

An example of this sealing technique is shown in Figure 3, which shows a jacketed sample after being subjected to about 15 MPa gas pressure. Note that the salt-filled portion of the lead jacket is undeformed because the gas had access to the exterior and interior of this portion of the sample. The salt, of course, is also unaffected and ready to be tested.

In contrast, the portion of the jacket between the two O-rings is highly deformed, having been pressed into the grooves and around the sharp ridges to form a seal. The seal has been found to be impervious to the effects of the hot silicone fluid for test durations thus far on the order of 36 hours.

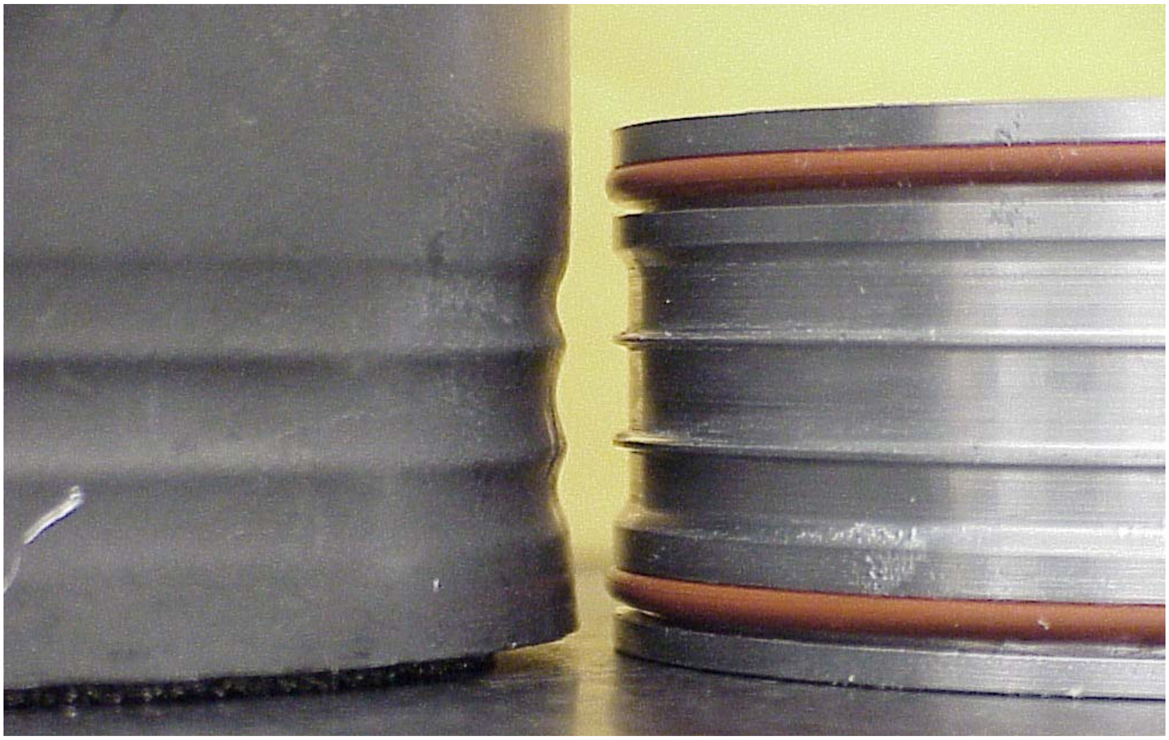

Figure 3 Metal-to-metal seal formed with gas pressure.

Although samples sealed this way have proven to be pressure-tight at the end caps, the outer jackets alone are still prone to rupture at about 6 MPa due to the large voids created by centimeter-sized grains in the crushed salt (**Error! Reference source not found.**). This is the reason for the inner lead sleeve, which serves to fill voids in front of the pressure-tight outer jacket. (Figure 5). No attempt is made to seal the inner sleeve against pressure.

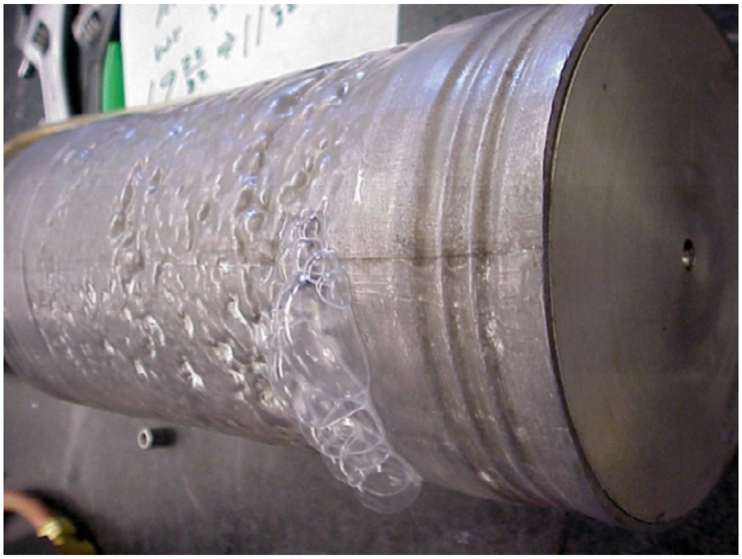

Figure 4 Post-test leak check.

Figure 5 Inner liner showing overlapping joint.

4.1.4 Sample Preparation

Sample preparation is somewhat more elaborate than the usual jacketing process used in the SNL Geomechanics laboratory due to the need to determine starting density and to prepare as homogenous a sample as possible. Direct determination of the starting density will be done by measuring sample dimensions and mass, relying on the circularity of the lead tubes to fix the sample cross section. Prior to assembly, the combined mass of all jacket components will be measured. After partially assembling the jacket by attaching the outer lead tube and liner to the beveled face plate and unvented lower end cap, the cylinder will be filled with dried, sieved salt to a depth of ~15 centimeters. The fill depth matches that used in previous work and will be adjusted as needed to accommodate jacket changes, etc. Because the salt is relatively compactable compared to the oil and aluminum space-fillers in the test chamber, a longer sample decreases the piston stroke available for pressurizing the vessel by pushing in the main ram.

Pouring salt directly from the storage container in which it is dried into the cylindrical jacket is not a good practice as particle sizes tends to segregate during the pouring. To increase the uniformity of the particle distribution in the sample, a larger quantity of crushed salt than needed will be poured into a conical heap and wedges then taken from the heap, with each wedge containing a sample of all particle sizes. Each wedge will be placed on a separate plastic sheet and carefully lowered (not poured) into the jacket. Samples will be constructed by placing in as many of these wedges as necessary to obtain the desired salt column length. As in previous work no attempt will be made to compact the sample before sealing.

4.1.5 Test Procedure

All tests will be conducted under isostatic and triaxial stress conditions using a triaxial test system designed by W.R. Wawersik [Wawersik and Preece, 1984]. Stress can be controlled by a combination of independent systems: a ram and a dilatometer, either or both of which will be used depending on the test type and load path. A separate auxiliary (ISCO) pump will be used in some tests to deliver low fluid pressures (approximately 500 psi), as the dilatometer system has limited volume/stroke. The loading ram is a standard hydraulic cylinder controlled by a servo valve that uses either the ram position or the pressure on the ram as the feedback control variable.

Samples are suspended from the upper vessel closure and vented through a nipple in the upper end cap that connects the interior of the sample to the exterior of the vessel. This differs from the procedure in earlier work where the sample was mounted to the main ram and vented through the lower end cap. Because of the importance of accurately measuring porosity changes, methods are used which require a pressure-tight connection between the vent nipple on the sample and the vessel wall. This is not possible in the present vessel configuration if venting occurs through the lower end cap and ram.

All heated tests will begin by loading the specimen into the vessel, filling the vessel with silicone oil and flushing oil through the vessel to remove trapped air. The vessel will then be pre-heated to the planned test temperature. Any moisture re-adsorbed onto the previously dried salt is assumed to be driven off by the pre-heating step, which typically takes 12–18 hours to ensure thermal equilibrium.

Quasistatic tests, designed to look at the short-term response of the salt to increasing pressure, will be done by advancing the ram into the vessel at a rate of ~$1.27x10^{-3}$ centimeters per second while the confining fluid pressure, ram displacement and fluid temperature are recorded. As the ram enters the sealed pressure vessel, the confining pressure fluid is additionally pressurized. Because the sample is not in contact with the ram during this stage, the sample will be subjected to pure hydrostatic stress.

Changes in sample volume due to compaction are made in two ways. In the first method, used for hydrostatic tests, sample volume can be calculated using the ram displacement and the corrections discussed in a later section. In this mode the entire vessel serves as a dilatometer, thus avoiding the problems associated with inferring volume change on the basis of point measurements of sample dimensions (described later). Assuming no fluid leaks, the decrease in sample volume due to increasing pressure is essentially equal to the product of the area of the ram and the distance it advances into the vessel. Maximum ram displacement is 10 cm and the ram area is 62.06 cm^2, allowing a system volume change of about 621 cm^3. When higher pressures are desired, the ram position may need to be reset by backing out of the vessel while the pressure is maintained by a separate system. To minimize corrections due to fluid compression, a set of relatively incompressible aluminum shapes are used as “stuffers” to fill as much of the vessel space as possible and minimize the volume of confining pressure fluid used for this test series.

The second method of determining changes in sample volume relies on displacement measurements across the sample diameter. Because the sample surface deforms in a very irregular fashion (Figure 6), displacement measurements across the sample diameter are perhaps less accurate but are necessary in shear and creep tests. When these measurements are done, clip-on “Schuler type” gauges are used; these gauges reflect diametrical displacement of the sample made on a small area of the side of the sample (Figure 6).

As a check on the accuracy of real-time volume measurements, fluid displacement may be occasionally used to determine the volume of the fully assembled sample pre- and post-test, with the post-test decrease in volume attributed to compaction of the salt.

(a) undeformed sample (b) deformed sample

Figure 6 “Schuler-type” gages placed on samples.

Creep tests may require maintaining a constant pressure for several days and measuring small volume changes. Initial pressurization will be accomplished using the methods employed for the quasistatic tests; a combination of the ISCO syringe pump and dilatometer can be used to achieve the desired confining pressure; the main ram can then be used to apply a differential stress by switching its control mode from constant rate (stroke control) to constant pressure, while the dilatometer maintains the constant confining pressure.

Any changes in room temperature or oil temperature have a deleterious effect on creep tests, both by direct effect on the salt behavior and by changing the volume of the oil. Errors due to fluctuations in room temperature are typically negligible as room temperature is generally controlled to +/- 0.5^{o}C. There is a transient rise in oil temperature due to adiabatic heating during pressurization, which calibrations show to be negligible for these tests.

4.2 Observational Microscopy

Sandia has existing activity/project specific procedures (SPs) which are available online documents (http://www.nwmp.sandia.gov/onlinedocuments). For the experiments in this Test Plan we intend to use the Scanning Electron Microscope (SEM) and the optical microscope, which involves the following SPs:

- SP 12-11 Fischer Scientific Stereomaster Microscope
- SP 12-13 Olympus BX-60 Microscope
- SP12-17 Scanning Electron Microscope Imaging and Energy Dispersive Spectroscopy

For the analyses of crushed salt samples, we will follow the procedures within the SPs and document the results and process in a logbook (not an official scientific notebook). This will provide results as Non-Q. This strategy will be reviewed as work progresses.

4.3 Modifications to Experimental Process

Modifications to test procedures outlined in Section 4.1 may be required during test deployment. These modifications will be conducted at the direction of the PI, and will be documented in the scientific notebook(s) as part of the QA records. Such modifications are not deviations and will not be reported as non-conformances that require corrective action.

If test conditions deviate appreciably from the anticipated execution of the current version of this Test Plan, the Test Plan will be revised.

5 TEST EQUIPMENT

This Test Plan will require equipment to measure loads, pressures and deformations on the sample. Additional equipment will be required for determining the sample volume changes. The equipment may consist of “off the shelf” items ordered directly from suppliers, standard equipment provided by service companies, and/or custom-built equipment designed and built for a specific task(s) governed by the Test Plan. All equipment used will follow the supplier’s/designer’s operation and calibration recommendations (as required). All equipment with calibration requirements and quality-affecting operations will be documented as part of the QA records and controlled by FCT QAP 12-1 (see Subsection 9.4).

6 DATA-ACQUISITION PLAN

Both manually- and electronically-collected data will be acquired during the test activities. The following types of data may be recorded:

- data from the DAS
- manually collected test data.

6.1 Scientific Notebook(s)

A scientific notebook(s) will be used in accordance with FCT QAP 20-2 (see Subsection 9.4) to document all activities and decisions during the Test Plan (except as noted in Section 4.2). Specific information that may be entered in the scientific notebook(s) consists of:

- a statement of the objectives and description of work to be performed, as well as a reference to this Test Plan;
- a written account of all activities associated with the development and implementation of the mine-by test;
- documentation of safety meetings;

- a list of equipment used during each activity, including make, model, and operating system (if applicable);
- traceable references to calibration information for instruments and/or gauges calibrated elsewhere; and
- discussions of the information and/or observations leading to decisions to initiate, terminate, or modify test activities.

All entries in the scientific notebook(s) will be signed and dated by the person making the entry. The scientific notebook(s) for this Test Plan will be reviewed by an independent, technically-qualified individual at a minimum of every six months to verify that sufficient detail has been recorded to retrace the activities and confirm the results.

Manually collected data may also be recorded on specially prepared forms rather than in the scientific notebook(s) when that process will provide a more efficient means of data collection and tracking. In particular, a standard geomechanics test data form should be prepared and completed as each test is carried out. These will be included in a supplementary binder to the scientific notebook.

6.2 Electronic Data Acquisition

The DAS will be used to record instrumentation data during the test. Electronic data file-management information will be documented in the scientific notebook(s) for these activities.

6.3 Manual Data Acquisition

Manual data collection will be carried out during the test using a scientific notebook(s) or forms designed specifically for each activity or data type. Information will be documented such that duplication of information will be minimized. The PI will determine the means of documenting manually-acquired data and will ensure that all quality-affecting information is documented.

6.4 On-Site Validation

During the test activities, the PI will evaluate the data, as they are acquired. The data will be diagnosed for any equipment failure and/or procedure-induced effect that may degrade the data quality. The PI will take immediate action (if required) to make any necessary changes to the equipment configuration or the procedures to assure the data quality is consistent with the objectives of these activities.

The PI will use real-time evaluation of the acquired data during test activity to assure that the data are usable in a detailed interpretation, the conditions can be maintained over the planned duration of the activity, and an activity will not be terminated before the minimum objectives can be achieved under the given time restraints. The PI may utilize some or all of the following procedures and analytical tools:

- real-time inspection of signal quality to assure useable data
- real-time analysis of the acquired data to assess transducer functioning and proper operation of the DAS; and

If at any time the PI determines that a test activity objective cannot be accomplished due to time constraints, problems concerning the performance of the equipment, or unsuitability of initial conditions, the PI will consult with cognizant personnel to terminate the activity, or develop a recovery plan. The PI will document all real-time evaluation of data and conditions in the scientific notebook(s).

7 SAMPLING AND SAMPLE CONTROL

Crushed salt samples will be prepared under this Test Plan using mine-run salt. Following preparation and testing, the samples with lead jackets still attached will be marked with unique identification numbers and sealed in plastic bags. Subsequently, these sealed samples may be placed in on-site inventory or transported to other locations for further characterization, e.g., optical microscopy. Sample handling and transport will be controlled following requirements in FCT QAP 13-1 *Control of Samples.*

8 TRAINING

All personnel who will perform quality-affecting activities under this Test Plan will have training in the SNL QA program and relevant procedures according to FCT QAP 2-1 *Qualification and Training.*

9 QUALITY ASSURANCE

9.1 Quality-Affecting Activities

Activities performed under this Test Plan are quality affecting, except for those specifically noted in section 4.2. The intent of the data and observations made in these studies are expected to be used in design considerations and repository performance expectations.

9.2 Quality Assurance Program Description

Activities are conducted in accordance with the requirements specified in the FCT Quality Assurance Program Document. A complete discussion of this integration is given in Appendix A.

9.3 QA Procedures

The QAPs and SPs that may apply to work performed under this Test Plan include:
FCT **QAP 2-1** Qualification and Training
FCT **QAP 5-1** Implementing Procedures
FCT **QAP 6-1** Document Review Process
FCT **QAP 6-2** Document Control
FCT **QAP 9-1** Analysis
FCT **QAP 13-1** Control of Samples and Standards
FCT **SP 13-1** Chain of Custody
FCT **QAP 20-1** Test Plans
FCT **QAP 20-2** Scientific Notebooks

Modification to these procedures may be required during testing activities. Such modifications are not deviations and will not be reported as non-conformances that require corrective action. However, the PI will document modifications in the scientific notebook(s) as they occur as part of the QA records.

9.4 Manufacturers QA Procedures

Manufacturers' QA procedures that may apply to work performed under this Test Plan:

None.

9.5 Data Integrity

Care will be taken throughout the performance of the operations for this Test Plan to ensure the integrity of all data collected including documentation on hard copy and data collected on storage media. Duplicate copies of all data will be produced as quickly as possible and the duplicate copies will be maintained at a location separate from the test site to ensure that data are not lost.

9.6 Records

Records will be maintained as described in this Test Plan and applicable FCT QA implementing procedures. These records may consist of bound scientific notebook(s), loose-leaf pages, forms, printouts, or information stored on storage media. The PI or designee will ensure that the required records are maintained and submitted to the designated storage location.

9.6.1 Required QA Records

As a minimum, QA records will include:

- Scientific notebook(s);
- Calibration records for all controlled equipment;
- Equipment-specification sheets or information (if available);
- All forms containing manually-collected data;
- Standard geomechanics test forms, fully completed
- Chain of Custody Forms

9.6.2 Miscellaneous Non-QA Records

Additional records that are useful in documenting the history of the activities, but are considered non-QA records may be maintained and submitted to the SNL FCT Quality Assurance SharePoint Site or EIMS FileNet. . These records include:

- safety briefings;

- ES&H documentation;
- as-built diagrams of equipment
- equipment manuals and specifications;
- equipment manifests; and

These records do not support regulatory compliance and, therefore, are not quality-affecting information.

9.6.3 Submittal of Records

QA records generated through the implementation of this Test Plan shall be prepared and submitted to the SNL FCT Quality Assurance SharePoint site and submitted to EIMS Filenet in accordance with the SNL Records Management Manual and IM 100.2.2 Control of Records (manage and Protect Information). EIMS Filenet in accordance with the SNL Records Management Manual and IM 100.2.2 Control of Records.

10 HEALTH AND SAFETY

The safety practices and policies will meet the requirements of the SNL ES&H Manual. Operational safety will be addressed through an ES&H Primary Hazard Screening (PHS), a Hazard Analysis (HA), and a Pressure Safety Data Package (if required) developed by SNL. Work planning and controls will be implemented and records will be maintained by and in accordance with Division 6000 corporate policy. For example a specific JSA was developed for the lead jacket handling.

11 PERMITTING/LICENSING

There are no special licenses or permitting requirements for the work described in this Test Plan.

12 REFERENCES

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Appendix A - SDI Test Activities Quality Assurance

Organization

Sandia National Laboratories' organization is fully described on the Sandia Internal Website (Techweb). The activities described in this Test Plan are the responsibility of Organization 06914 (Geomechanics), within SNL's Geoscience, Climate, and Consequence Effects Center (06900), as shown on the abbreviated organization chart illustrated in Figure A-1. Figure A-1 also shows the QA organizational interfaces for the activity.

This R&D activity, managed as Work Package FTSN11UF0353, is conducted by Sandia National Laboratories under contract to U.S. DOE as part of the DOE-NE Fuel Cycle Technologies (FCT) program Used Fuel Disposition (UFD) Campaign. As an FCT UFD R&D activity, it is conducted in accordance with the FCT QAPD[1], SNL's DOE approved QA Program Description (SNL-QAPD)[2] and SNL's UFDC Preliminary Quality Assurance Implementation Plan[3] (SNL-UFCD-QAIP). Management decided to augment basic requirements for this QRL3 activity, based on the potential utility of the results. Hence, this Appendix to the activity Test Plan is provided in compliance with the provisions of SNL-UFCD-QAIP Section 4.

Quality Assurance Program

To summarize the minimum requirements for this QRL3 activity, as described in SNL-UFCD-QAIP, the activity is to be conducted in compliance with the SNL-QAPD, with the additional requirement that deliverables receive a technical review in accordance with the FCT QAPD Appendix B.

Management decided that certain quality assurance improvements would be beneficial to the conduct of this activity. A meeting was held on January 6, 2011, involving Organization 06914 management and technical staff, and the FCT QA POC to grade the quality assurance requirements to apply this activity. Table A-1 reflects the outcome of the grading considerations.

Quality Assurance requirements flow down from the FCT QAPD, the SNL-QAPD

1 U.S Department of Energy, Office of Nuclear Energy, Fuel Cycle Technologies Quality Assurance Program Document, Washington, D.C., October 13, 2010.

2 Sandia National Laboratories Quality Assurance Program Description, June 25, 2010, WebFileShare ID: WFS1043674

3 Sandia National Laboratories Used Fuel Disposition Campaign Preliminary Quality Assurance Implementation Plan; Fuel Cycle Research & Development; Prepared for U.S. Department of Energy Used Fuel Disposition Campaign December 2010 FCR&D-USED-2011-000019; January 13, 2011.

and SNL-UFCD-QAIP, as illustrated in Figure A-1. Predominantly, procedures from the Sandia Corporate Policy System (CPS) apply to this activity's quality elements, consistent with the approved SNL-QAPD. In selected instances, specific procedures were developed to improve quality to approximate NQA-1 levels for certain quality elements. Table A-1 identifies the CPS procedures that are generally applicable as well as the specific procedural augmentations that apply.

Figure A-1 – General QA Requirements Flow Down for SDI Activities

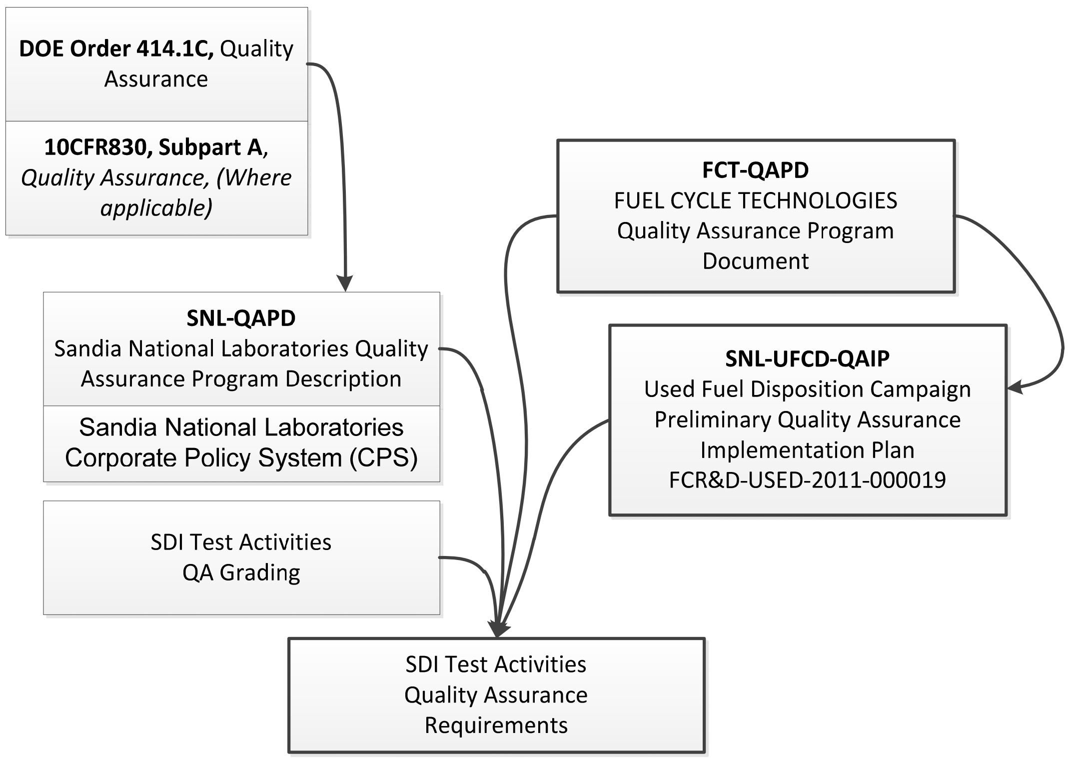

Table A-1 Graded Quality Assurance Requirements for SDI Activity

NQA-1 (2008) Requirement Excerpt4	Summary of Grading	Procedures as Appropriate
Organization - Responsibilities for the establishment and implementation of the quality assurance program shall be defined. The organizational structure, functional responsibilities, levels of authority, and lines of communications for activities affecting quality hall be documented.	A description of performing organizations placement within laboratory organization, including interfaces, is provided above. Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**CG 100.1** - Establish the Decision-Making Framework **CG 100.1.1** - Create and Maintain the Mgmt. Structure **CG 100.1.2** - Create or Change a Policy - Process - or Procedure **CG 100.1.3** - Define Roles - Responsibilities - Accountabilities - and Authorities **CG100.6.8** - Identify and Manage Corporate Issues **CG100.6.16** - Conduct Management Reviews **CG100.6.17** - Conduct EO/LLT Management Reviews

4 Refer to ASME NQA-1-2008 (Revision of ASME NQA-1-2004) Quality Assurance Requirements for Nuclear Facility Applications for complete description of requirement.

Quality Assurance Program - The program shall identify the activities and items to which it applies. The program shall provide control over activities affecting quality to an extent consistent with their importance.	A description of the Quality Assurance Program, requirements flow down, relationships between FCT QA, NNSA/SSO QA and laboratory QA organizations, is provided above. Rely on CPS procedures, including those listed in adjacent column, as appropriate. Specific qualifications and training required of MOW involved in the activity are addressed by the specific FCT QAP identified.	Multiple CPS procedures in **HR100.2**. **FCT QAP 2-1** Qualification and Training
Design Control - The design shall be defined, controlled, and verified. Design inputs shall be specified on a timely basis and translated into design documents. Design interfaces shall be identified and controlled. **(Note: Includes provisions applicable to use of computer programs.)**	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Related controls are addressed by the specific FCT QAP identified. Note: Requirements specifically identified for software determined to be N/A, because no software is designed as part of this activity.	**ME100.3.1 -** Perform Work **ME100.3.2 –** Manage Projects Throughout Their Lifecycle **ME100.3.3 -** Apply Configuration Management Principles to Documents and Physical Items **FCT QAP 20-1** Test Plans

Procurement Document Control - Applicable design bases and other requirements necessary to assure adequate quality shall be included or referenced in documents for procurement of items and services. To the extent necessary, procurement documents shall require Suppliers to have a quality assurance program consistent with the applicable requirements of NQA-1.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	Multiple procedures in **SCM100** – Manage Property, Material and Services through the Supply Chain
Instructions, Procedures and Drawings - Activities affecting quality and services shall be prescribed by and performed in accordance with documented instructions, procedures, or drawings that include or reference appropriate quantitative or qualitative acceptance criteria for determining that prescribed activities have been satisfactorily accomplished.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Specific controls are addressed by the FCT QAP identified.	**CG 100.1.2** - Create or Change a Policy - Process - or Procedure **FCT QAP 5-1** Implementing Procedures

Document Control - The preparation, issue, and change of documents that specify quality requirements or prescribe activities affecting quality such as instructions, procedures, and drawings shall be controlled to ensure that correct documents are being employed.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. FCT QAPs listed will be modified to rely mostly on CPS, to provide explicit information for the task.	**IM100.2.1** - Control of Documents **IM100.2.2** - Control of Records **HR100.2.15** - Maintain Training Records in TEDS LMS **HR100.5.7** - Manage Corporate Human Resources Records **FCT QAP 6-1** Document Review Process **FCT QAP 6-2** Document Control
Control of Purchased Items and Services - The procurement of items and services shall be controlled to ensure conformance with specified requirements.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1** - Perform Work **SCM100.2.2** - Acquire Property (Requirements and Instructions - Inspect and Return Property section) **SCM100.3.10** - Do's and Don'ts for Requesters and SDRs During Contract Management Activities (Requirements and Instructions - step 3)

Identification and Control of Items - Controls shall be established to assure that only correct and accepted items are used or installed.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1 -** Perform Work **SCM100.2.2 -** Acquire Property (Requirements and Instructions - Inspect and Return Property section) **SCM100.3.3** – Manage Property **SCM100.3.10 -** Do's and Don'ts for Requesters and SDRs During Contract Management Activities (Requirements and Instructions - step 3) **SCM100.3.13** – Manage Suspect or Counterfeit Items **SCM100.3.14 -** Store General Materials at Sandia National Laboratories
Control of Special Processes - Special processes that control or verify quality, such as those used in welding, heat treating, and nondestructive examination, shall be performed by qualified personnel using qualified procedures in accordance with specified requirements.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Additional controls are addressed by the FCT QAP identified.	**ME100.3.1 -** Perform Work **FCT QAP 9-1** Analysis

Inspection - Inspections required to verify conformance of an item or activity to specified requirements or continued acceptability of items in service shall be planned and executed.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1 -** Perform Work **SCM100.2.2** - Acquire Property (Requirements and Instructions - Inspect and Return Property section) **SCM100.3.10** - Do's and Don'ts for Requesters and SDRs During Contract Management Activities (Requirements and Instructions - step 3) **SCM100.3.13** – Manage Suspect or Counterfeit Items
Test Control - Tests required to collect data such as for siting or design input, to verify conformance of an item or computer program to specified requirements, or to demonstrate satisfactory performance for service shall be planned and executed. **(Note: Applicable to testing of computer programs, hardware and operating systems.)**.	Activity specific controls are addressed by the FCT QAPs identified. Note: Requirements specifically identified for software determined to be N/A, because of the nature and use of data recording and later processing.	**FCT QAP 20-1** Test Plans **FCT QAP 20-2** Scientific Notebooks
Control of Measuring and Test Equipment - Tools, gages, instruments, and other measuring and test equipment used for activities affecting quality shall be controlled, calibrated at specific periods, adjusted, and maintained to required accuracy limits.	Rely on CPS procedures, including those listed in adjacent column, which requires a measurement assurance plan consistent with Primary Standards Lab (PSL) practices.	**ME100.3.1 -** Perform Work

Handling, Storage and Shipping - Handling, storage, cleaning, packaging, shipping, and preservation of items shall be controlled to prevent damage or loss and to minimize deterioration.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Additional controls are addressed by the FCT QAP / FCT SPs identified.	**SCM100.3.3** – Manage Property **SCM100.3.14 -** Store General Materials at Sandia National Laboratories **FCT QAP-13-1** Control of Samples **FCT SP-13-1** Chain of Custody
Inspection, Test, and Operating Status - The status of inspection and test activities shall be identified either on the items or in documents traceable to the items where it is necessary to ensure that required inspections and tests are performed and to ensure that items that have not passed the required inspections and tests are not inadvertently installed, used, or operated.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**ME100.3.1 -** Perform Work **CG100.5.5 -** Control Item and Process Nonconformances **SCM100.3.13 -** Manage Suspect or Counterfeit Items

Control of Nonconforming Items – Items that do not conform to specified requirements shall be controlled to prevent inadvertent installation or use.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**SCM100.3.13** - Manage Suspect or Counterfeit Items **SCM100.3.13** - Manage Suspect or Counterfeit Items **CG100.6.6** - Perform Corrective Action **CG100.6.9** - Conduct Root Cause Analysis and Extent of Condition Reviews **CG100.5.5** - Control Item and Process Nonconformances
Corrective Action - Conditions adverse to quality shall be identified promptly and corrected as soon as practicable. In the case of a significant condition adverse to quality, the cause of the condition shall be determined and corrective action taken to preclude recurrence.	Rely on CPS procedures, including those listed in adjacent column, as appropriate.	**CG100.6.6** - Perform Corrective Action **CG100.6.9** - Conduct Root Cause Analysis and Extent of Condition Reviews

Quality Assurance Records - The control of quality assurance records shall be established consistently with the schedule for accomplishing work activities. Quality assurance records shall furnish documentary evidence that items or activities meet specified quality requirements. Quality assurance records shall be identified, generated, authenticated, and maintained, and their final disposition specified.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Transitory working information and non-record materials will be managed in the ANEP SharePoint site. The ANEP SharePoint site will be used for interim storage of records for convenience FCT deliverables and associated records will be managed in accordance with the FCT Records Management Plan upon finalization of the deliverable. (Submittal of records to the Fuel Cycle Research and Development Document Management System will maintain the associations between deliverables and supporting documentation, to the extent practicable.)	**IM100.2.1** - Control of Documents **IM100.2.2**- Control of Records **IM100.2.3** - Prepare and Release Information **IM100.2.4** - Cancelled - Manage Records Throughout their Lifecycle **IM100.2.5**- Identify and Protect Unclassified Information

Audits - Audits shall be performed to verify compliance to quality assurance program requirements, to verify that performance criteria are met, and to determine the effectiveness of the program. Audit results shall be documented and reported to and reviewed by responsible management. Follow-up action shall be taken where indicated.	Rely on CPS procedures, including those listed in adjacent column, as appropriate. Note: It was determined that auditing the activity was not considered as value added, surveillance/assessment was considered adequate.	**CG100.6.3** - Perform Assessments **CG100.6.7** - Conduct Independent Internal Audits