APPENDIX E

FCT DOCUMENT COVER SHEET [1]

Name/Title of Deliverable/Milestone/Revision No. M3FT-14SN0818015: Modified Test Plan For Salt Reconsolidation Experiments at Sandia National Laboratories

Work Package Title and Number DR Salt R&D - SNL

Work Package WBS Number 1.02.08.18

Responsible Work Package Manager Christie Leigh
(Name/Signature)

Date Submitted 6/30/2014

Quality Rigor Level for Deliverable/Milestone[2]	☒ QRL-3	☐ QRL-2	☐ QRL-1 Nuclear Data	☐ Lab/Participant QA Program (no additional FCT QA requirements)

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 ☐ Other

This Deliverable was subjected to:

☒ Technical Review ☐ Peer Review

Technical Review (TR)
Review Documentation Provided

☐ Signed TR Report or,
☒ Signed TR Concurrence Sheet or,
☐ Signature of TR Reviewer(s) below

Peer Review (PR)
Review Documentation Provided

☐ Signed PR Report or,
☐ Signed PR Concurrence Sheet or,
☐ Signature of PR Reviewer(s) below

Name and Signature of Reviewers

Kris Kuhlman (Review authenticated and documented on SNL FCT QAP 6-1 Document Review and Comment Form)

Michael Schuhen (Review authenticated and documented on SNL FCT QAP 6-1 Document Review and Comment Form)

NOTE 1: *Appendix E should be filled out and submitted with the deliverable. Or, if the PICS:NE system permits, completely enter all applicable information in the PICS:NE Deliverable Form. The requirement is to ensure that all applicable information is entered either in the PICS:NE system or by using the FCT Document Cover Sheet.*

NOTE 2: *In some cases there may be a milestone where an item is being fabricated, maintenance is being performed on a facility, or a document is being issued through a formal document control process where it specifically calls out a formal review of the document. In these cases, documentation (e.g., inspection report, maintenance request, work planning package documentation or the documented review of the issued document through the document control process) of the completion of the activity, along with the Document Cover Sheet, is sufficient to demonstrate achieving the milestone. If QRL 1, 2, or 3 is not assigned, then the Lab / Participant QA Program (no additional FCT QA requirements) box must be checked, and the work is understood to be performed and any deliverable developed in conformance with the respective National Laboratory / Participant, DOE or NNSA-approved QA Program.*

Sandia National Laboratories*
Fuel Cycle Technology (FCT) Program

Test Plan SNL-FCT-TP-14-002

Experimental Investigation of Two-Phase Flow in Rock Salt

Revision 0

Effective Date: 6/30/2014

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* *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
FCT	Fuel Cycle Technologies
JHA	Job Hazard Analysis
PHS	Primary Hazard Screening
PI	SNL Principal Investigator, or Designee
QA	Quality Assurance
QAP	Quality Activity Plan
QAPD	QA Program Document
SNL	Sandia National Laboratories
SP	Activity/Project Specific Procedure
WIPP	Waste Isolation Pilot Plant

2 REVISION HISTORY

This is the original version (Revision 0) 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 organizations that performed the original review and approval.

3 PURPOSE AND SCOPE

Geologic salt deposits are being evaluated around the world as candidate locations for nuclear waste disposal. Fluid transport of radionuclides is the most credible mechanism by which repository releases could be realized. Repository sealing systems need to account for fluid flow properties of intact and crushed salt because both are essential elements comprising repository sealing systems. Waste disposal and mine operations disturb the intact salt, altering its flow properties, so understanding the disturbed rock salt is also necessary for realistic repository performance predictions.

Waste release scenarios involve gas and brine flow. The repository is sealed under atmospheric pressure and waste degradation is expected to generate gas, which could challenge seal systems. Release scenarios also address formation brines where geologic media are saturated, as at the Waste Isolation Pilot Plant (WIPP). Finally, salt continues to be studied by the Department of Energy (DOE) for possible disposal of heat-generating waste, so understanding its fluid flow properties under heated conditions is essential to providing a technical basis for realistic repository performance predictions.

This Test Plan describes procedures for conducting laboratory scale flow tests on intact, damaged, crushed, and consolidated crushed salt to measure the capillary pressure and relative permeability functions. No data exists at present to estimate or measure the parameters used in capillary pressure and relative permeability models (Davis and LaVenue, 1990; Davis, 1991). The primary focus of the tests will be on samples of bedded geologic salt from the WIPP underground. However, the tests described herein are directly applicable to domal salt. Samples being tested will be confined by a range of triaxial stress states ranging from atmospheric pressure up to those approximating lithostatic. Initially these tests will be conducted at room temperature, but testing procedures and equipment will be evaluated to determine adaptability to conducting similar tests under elevated temperatures.

In general, laboratory studies permit measurement of generic salt properties (mechanical, thermal, hydrologic, and chemical) under controlled conditions. Significant research has been conducted on understanding the phenomenology of salt over a broad range of temperatures expected in geologic disposal systems, including those that contain heat-generating waste. This has yielded a large body of knowledge on salt behavior and suitability. The laboratory studies described herein are designed to contribute substantively to the body of knowledge accumulated hitherto. The results will be used generically to develop input parameters for process models that predict repository system behavior and to assess the adequacy of existing empirical models (and to propose new models when existing models are shown experimentally to be inadequate). Specifically, the tests will reduce uncertainties of two-phase flow constitutive models and the associated parameters, as well as in the predictions of two-phase flow process models. Additionally, the objectives of these studies are consistent with those of international salt repository research programs.

Two commonly used closed-form constitutive models for capillary pressure are due to Brooks and Corey (1964) and van Genuchten (1980). These models are used to derive closed-form relative permeability models using the theories of Burdine (1953) and Mualem (1976). They are characterized by a threshold pressure, P_t, residual brine and gas saturations, S_{wr} and S_{gr}, respectively, and pore-size distribution parameters, λ and $m = 1 - 1/n$. These parameters are material-specific and have never been determined for geologic bedded salt. Hence, the purpose of this Test Plan is to

1) Obtain empirical capillary pressure functions (or brine release curves), which will be used to:
   a) Test the validity of the Brooks-Corey and van Genuchten models for capillary pressure behavior of rock salt;
   b) Determine the Brooks-Corey parameters λ, P_t, S_{wr}, and S_{gr} commonly referred to as the pore-size distribution factor, air-entry (or threshold) pressure, residual brine saturation, and residual gas saturation, respectively;
   c) Determine the van Genuchten parameters $m = 1 - 1/n$, $\alpha = 1/P_t$, S_{wr}, and S_{gr}; and
   d) Characterize hysteretic behavior of salt capillary pressure functions during drainage and imbibition cycles.
2) Measure relative brine and gas permeabilities as a function of brine saturation, which will be used to:
   a) Test the validity of the Burdine (1953) and Mualem (1976) models for gas and brine relative permeabilities (as applied to rock salt); and
   b) Characterize hysteretic behavior of relative permeability functions of salt during drainage and imbibition cycles.
3) Evaluate the adaptability of testing equipment and procedures to conducting similar tests at elevated temperatures and confining stresses.

Test activities associated with this Test Plan will be conducted in accordance with Quality Assurance (QA) document FCT QAP-20-1. This Test Plan describes laboratory methods for measuring the capillary pressure function and relative permeabilities of salt in its various forms.

4 EXPERIMENTAL PROCESS DESCRIPTION

Pressure cells and a centrifuge will be used to measure capillary pressure curves. Relative permeability tests will be conducted on core samples of salt by injecting gas and liquid phases into the core to achieve predetermined pressure drops across the length of the sample. The resulting flow rates of the two fluids will be used to infer the respective phase relative permeabilities by invoking Darcy’s law.

Table 1. Sample Test Matrix

Number of samples to test		Test Type	
Sample Type		Capillary pressure	Relative permeability
WIPP salt	crushed/consolidated	6	6
	damaged core	3	3
	pristine core	6	6
Domal salt	crushed/consolidated	5	5
	damaged core	3	3
	pristine core	5	5

Table 1 presents the matrix of conditions and samples to be tested under this test plan. The tests described here in will be performed at Sandia National Laboratories' Carlsbad facilities. Scoping activities, particularly near and at intact core permeabilities will be performed by a qualified supplier (contractor laboratory).

4.1 Overall Strategy and Process

The general strategy for obtaining the empirical capillary (suction) pressure function involves measuring the mechanical forces needed to overcome capillary forces at known brine saturation (degree of wetness). Data will be collected both during drainage (brine release) and imbibition (brine uptake by capillarity) to characterize expected hysteresis effects. The procedure involves adding or removing a known quantity of brine to or from a core of salt using capillary action, gas pressure or centrifugation. At mechanical equilibrium, the capillary pressure equals the gas pressure or the centrifugal stress needed to expel excess fluid or to imbibe a known fluid volume.

The general strategy for measuring relative permeabilities involves application of Darcy's law under two-phase conditions in test cells where flow and pressure boundaries are carefully controlled. This requires measurements of differential pressures and the resultant flow rates (or fluxes). The measured relative permeabilities will be compared to those predicted by the Burdine (1953) and Mualem (1976) integrals and the capillary pressure functions. These integrals provide a means to compute the relative liquid and gas permeabilities from the capillary pressure function. Comparing relative permeability values predicted by these integrals to those measured directly in the tests will provide a way to evaluate the applicability of the Burdine (1953) and Mualem (1976) integrals to rock salt relative permeabilities.

4.2 Description of the Proposed Experiments

4.2.1 Relevance of earlier work

Previous work aimed at experimentally determining two-phase constitutive model parameters focused on capillary pressure and single-phase permeability measurements on anhydrite core from the Salado formation (Howarth and Christian-Frear, 1997). Some of the testing methods and sample preparation techniques used in this test effort will be similar to those used in earlier work. This Test Plan extends earlier work to halite core samples obtained from consolidation of run-of-mine salt, and by the inclusion of relative permeability tests.

Methods for determining the capillary pressure and relative permeability functions of soils and oil reservoir rocks will be modified for intact, crushed, and consolidated salt. The modification will involve use of corrosion resistant materials for use with corrosive brine. Methods for soils (Stephens, 1995) will be used for unconsolidated crushed salt and consolidated samples with low fractional densities to yield parameters that a useful for early-time after repository closure. Methods developed for low permeability oil reservoir rocks (e.g., Osoba et al. (1951) will be used for high consolidation states, particularly at permeabilities approaching those of intact salt.

Single-phase permeabilities from in situ measurements in bedded salt in the WIPP underground are in the range of 10^{-22} to 10^{-20} m^2 (Beauheim et al., 1990; McTigue, 1992). The relative permeabilities to be measured in tests discussed herein are expected to have these as the bounded values.

4.2.2 Sample Preparation

Tests will be conducted on samples of screened granular run-of-mine halite with different degrees (stress levels) of consolidation, and intact salt core (undamaged and damaged). Undamaged cores will be obtained by applying a confining stress (at the lithostatic level) to intact core for some period prior to testing to reverse damage caused by the core extraction process. This damage reversal has been observed experimentally in tests on intact core that showed increases in salt compressive strength with increasing confining stresses due to salt healing (Boresi and Deere, 1963; Wawersik and Hannum, 1980). Intact core damage will be characterized using epoxy dye-penetrant impregnation (Howarth, 1993). Damage in the form of surface microfractures induced by drill coring, laboratory sub-coring, finishing, and pressure relaxation after core extraction from the in-situ lithostatic level, has a strong effect on the capillary pressure and relative permeability functions.

The samples will be cylindrical with diameter and height to fit in a standard Tempe cell and centrifuge core holder when measuring capillary properties and Hassler Cell (explained below) when testing relative permeability. Standard Tempe cells have a diameter of 7.62 cm (3 inches), and a height of 3 or 6 cm.

Compacted salt samples will be prepared as described in Test Plan SNL-FCT-TP-12-0001 (Bauer, 2012). The preparation process can be summarized as follows:

1. Sieving run-of-mine salt with a standard 9.5 mm sieve, and determining the particle size distribution of this sub-9.5 mm salt from five (5) representative sub-samples.
2. Oven-drying the sieved salt at temperatures of 175° to 250°C to drive off free moisture. At these temperatures, run-of-mine salt typically shows a total mass loss of about 0.3%.
3. Compaction of samples weighing 1600 g into cylindrical samples of 10 cm diameter and 15 cm length. Mean compaction stresses will range from 0 to 10 MPa.
4. Cutting and sub-coring these samples to sizes suitable for conducting flow tests. The dry, cut, and sub-cored samples will be weighed to determine the bulk densities. The porosities of these samples will also be measured using standard methods.

Sample storage as well as steps 3 and 4 will occur in such a manner that the samples are maintained dry. Samples will be appropriately labeled and the label will include the stress level used to compact the sample. Sample preparation, utilization, and final disposition will be documented in scientific notebooks. When samples are not in the possession of the individual designated as responsible for their custody, they will be stored in a secure area with associated documentation (Chain of Custody).

Intact core samples will be sub-cored from larger pieces of intact salt using core barrels and compressed air as the circulation fluid. Core barrels and core diameters will match standard core sizes for Hassler cells, which is either 1½ inch or 2 inch. These cores can be cut to length to fit into the Hassler cell, and finished with lapping tools to achieve flat ends perpendicular to the core axis. The sub-cores from intact samples will be obtained both parallel and perpendicular to bedding in order to account for core-scale anisotropy of the relative permeabilities. Similarly, sub-cores of consolidated samples will be obtained in both the radial and axial directions to account for possible “bedding” induced by consolidation loads.

4.2.3 Test Procedure

Saturated brines with known specific gravity will serve as the primary working wetting phase. Brines prepared by dissolution of rock salt in distilled water until saturation will be used for all the flow tests. The primary working gas (non-wetting) phase will be air. Nitrogen or helium may also be used as per the Principal Investigator's (PI’s) discretion.

Task 1: Capillary pressure function measurements

Brine drainage (release) curve will be obtained by draining samples initially at full brine saturation. Brine saturation is defined here as the volume of brine in a rock salt sample per unit total connected pore (void) volume of the sample. Imbibition tests will start with samples initially at residual brine saturation, which is the unchanging minimum saturation attained by the sample at high capillary pressures. To obtain the empirical capillary pressure functions, drainage and imbibition will be achieved by two methods:

1) Displacement of the wetting phase (brine) with a pressurized non-wetting phase (air, Nitrogen, or Helium), and
2) Centrifugation.

The first approach will involve use of pressure chambers where brine will be forced out of salt core samples using step increases in gas phase pressure. A porous ceramic disk will be used in these pressure cells to maintain uniform wetting-phase saturation in the sample. This approach will yield capillary pressure data up to 1.5 MPa (15 bar) and will be used for consolidated salt and low fractional density consolidated salt.

With the second approach, salt core is spun at different rotational speeds to generate different pressures and drive brine into or out of the salt core. This approach, with rotational speeds of up to 20,000 rpm, is expected to generate data for capillary pressure of up to 10 MPa.

Drainage tests will be used to measure the residual brine saturation and threshold (entry) pressure, while imbibition tests will be used to measure the residual gas saturation. The threshold pressure is the value at which the non-wetting phase fully penetrates the sample. The weight of each sample at full saturation will be used to determine the volumetric brine content at saturation, which will be compared to the measured porosity of the respective sample. The unchanging weight of the samples at high pressures will be used to estimate the residual brine content. During the tests, the cumulative brine volume/weight drained from or imbibed by the sample at each pressure level will be monitored. It will be used to compute the brine content of the sample at the respective gas pressures. Volumetric brine content of the samples will also be measured gravimetrically.

Secondary brine release and imbibition curves will be obtained by starting drainage or imbibition at intermediate brine saturations and capillary pressure values. These curves will be used to study hysteresis of the capillary pressure function in compacted halite. Hysteresis is partly explained by trapped air during imbibition and trapped brine during drainage. Trapped brine arises from viscous fingering when the non-wetting phase is used to displace the wetting phase. Phase entrapment may be minimized by using small step changes in non-wetting phase pressures.

Task 2: Relative permeability measurements

Relative brine and gas permeabilities will be measured by flowing brine and gas through salt core samples at predetermined pressure drops across the samples. The resulting phase flow rates and Darcy's law will be used to determine the respective permeabilities. Gravimetric methods will be used to determine the equilibrium brine saturation associated with each pressure-drop and flow rate data pair. The data will be collected using:

1. A Hassler core holder (triaxial flow cell), and
2. Centrifugation.

The Hassler core holder will be used to obtain both brine and gas relative permeabilities, whereas the centrifuge will yield brine relative permeabilities. Saturation distribution through the core samples will be maintained using porous disks with gas threshold pressures above gas injection pressure at the ends of the sample to minimize boundary (end) effects. Osoba et al. (1951) demonstrated that saturation gradients can also be reduced by increasing the flow rates.

A complicating factor for relative permeability measurements is the sensitivity of the single-phase permeability to confining stress (e.g., Stormont and Daemen, 1992; Stormont et al., 1992) due to creep. This will be a source of measurement error in the determination of relative permeability. The magnitude of this error will be evaluated to determine its significance over the testing period.

Another source of error in the measurements would be the dissolution and precipitation of salt in the brine, which can significantly alter the single-phase permeability during the course of the flow tests. The effect of these processes will be minimized by use of brine saturated (or in thermodynamic equilibrium) with dissolved rock salt.

4.3 Data to be collected

The data to be collected or monitored in the tests include

1. Fluid (gas and liquid) inlet, outlet, and confining pressures,
2. Fluid flow rates and cumulative outflow volumes,
3. Sample masses, and
4. Temperature.

4.4 Test Equipment

This Test Plan will require equipment to measure masses, pressures and flow rates. Additional equipment will be required for determining the sample and brine volumes. 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.

4.4.1 Capillary Pressure

Tempe cells, a ceramic plate extractor, and a centrifuge will be used to obtain data for the brine release curves. Tempe cells will be used in the low pressure range of 0 to -0.1 MPa (0 to 1 bar, suction), which would include most data from saturation to the beginning of the dry range. The ceramic plate extractor will be used in the pressure range of -0.1 to -1.5 MPa (1 to 15 bar). Figure 1 shows a schematic that illustrates the operating principle of Tempe cells and the porous ceramic plate extractor.

A centrifuge will be used for capillary pressures higher than 1.5 MPa. A centrifuge that may be used for these measurements is shown in Figure 2. Rotational speeds of up to 20,000 rpm are achievable with an appropriate rotor and sample holder. In these tests the equilibrium fluid mass and volume ejected from or imbibed by the core sample at different rotational speeds will be determined gravimetrically. Other methods, including resistivity and nuclear magnetic resonance (NMR), may also be used for this purpose. This will be used to compute the brine saturation at a given capillary pressure fixed by the rotational speed. The capillary pressure P_c at a given rotational speed ω is estimated by

$$P_c = \frac{1}{2}\left(\rho_b - \rho_g\right)\omega^2(r_2^2 - r_1^2)$$

where ρ_b and ρ_g are brine and gas densities, and r_1 and r_2 are radial distances from axis of rotation to inlet and outlet faces of the salt core. It should be noted the capillary pressure and brine saturation are distributed through the core length $L = r_2 - r_1$. Hence, the measurement

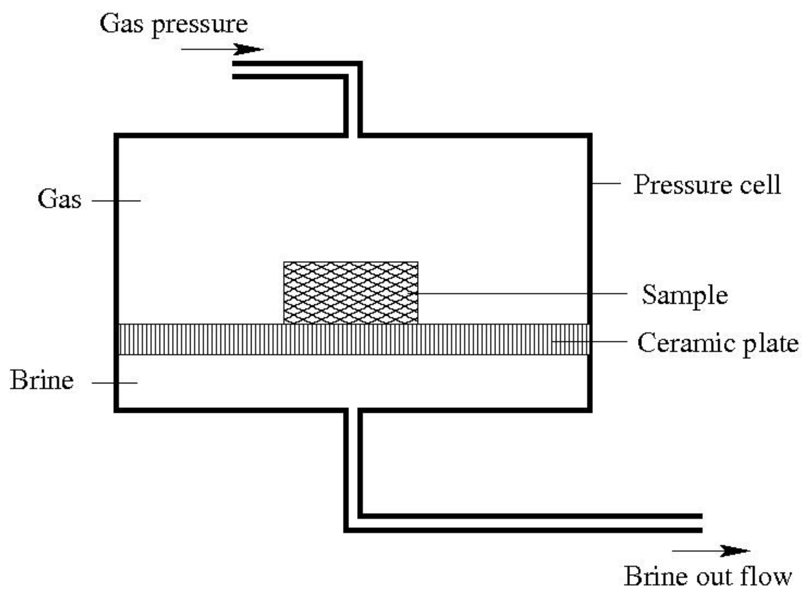

Figure 1. Schematic of the operating principle of Tempe cells and the ceramic plate extractor for measuring capillary pressure as a function of brine saturation.

described here yields the average brine saturation and capillary pressure of the core at a given rotational speed. To minimize the errors associated with this averaging it is desirable to use cores of small length. Methods such as magnetic-resonance imaging (Chen and Balcom, 2005) exist for measuring the brine saturation over the core length. They may be used in this work.

4.4.2 Relative Permeability

4.4.2.1 Steady-state method using the Hassler flow cell

The two-phase permeabilities will be measured with the Hassler method (Osoba et al., 1951; McCaffery and Bennion, 1974). The method is used in the oil and gas industry to measure both gas and liquid permeabilities, and operates on the so-called Hassler's principle. This involves injection of gas and/or liquid phases into a core at flow rates that yield a predetermined pressure drop (gradient) across the core sample. The phase permeabilities are then determined by applying Darcy's law. A schematic of the Hassler core holder is shown in Figure 3.

The Hassler method utilizes porous discs permeable only to the wetting phase at the sample ends to ensure a uniform saturation throughout the core. The pressure drop in the two fluid phases can be measured independently. The sample will initially be at full brine saturation. Brine and gas will be made to flow through the sample at such rates that the same pressure drop in the two phases is realized. This ensures the same capillary pressure at the core ends.

Figure 2. The centrifuge to be used in capillary pressure and relative permeability tests.

The effective flow pressures in the range of 2 to 10 MPa will be used in the flow tests, with the effective pressure, P_m, defined as (Howarth and Christian-Frear, 1997) $P_m = (P_{in} + P_{out})/2$, where P_{in} and P_{out} are the inlet and outlet pressures, respectively, for a given test. In previous such tests to measure the single-phase permeability of Anhydrite core, outlet pressure was fixed at 0.1 MPa, while the inlet pressure was varied to achieve inlet-outlet pressure data pair from which the permeability was determined (Howarth and Christian-Frear, 1997). The differential pressures expected in the tests will generate flow rates of the order 10^{-14} m^3. These are very small rates and may require development of specialized flow meters or the use of gravimetric methods or load cells.

4.4.2.2 Transient method using a Centrifuge

The centrifuge method will also be used to measure relative permeabilities of brine and air. The centrifuge method of Hagoort (1980) will be applied to the measurement of these relative permeabilities. It involves applying Darcy's law in a centrifugal force field to measure relative permeabilities, $k_{r,b}$, which is related to the normalized cumulative brine produced Q_D from the tested sample according to (Hagoort, 1980)

$$k_{r,b}(S_b^*) = \frac{dQ_D}{dt_D},$$

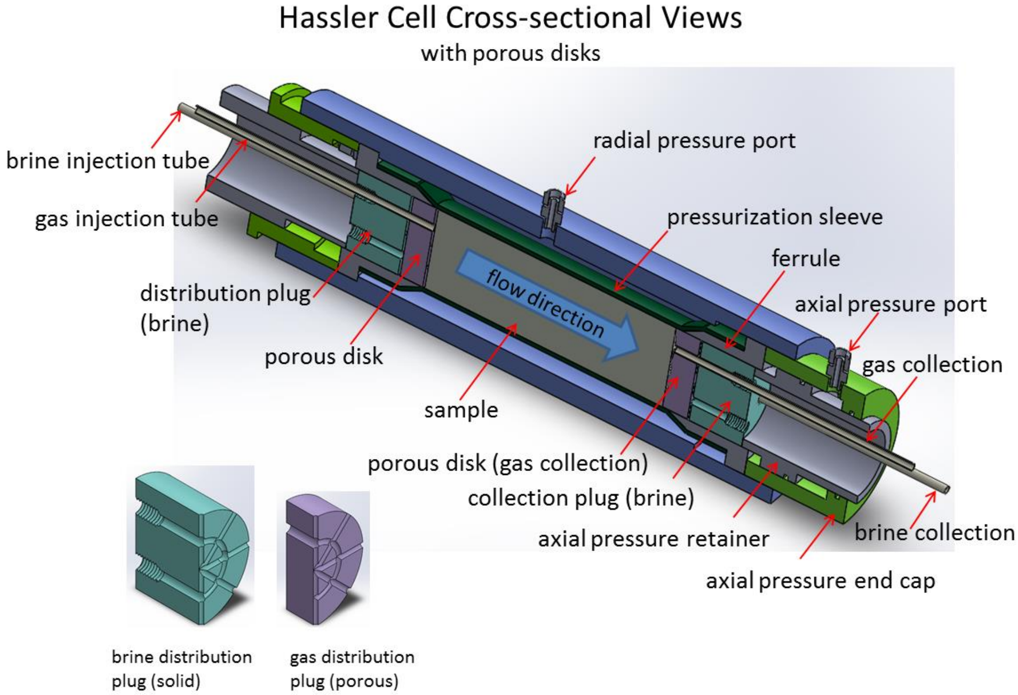

Figure 3. A schematic of the Hassler core holder for measuring brine and gas relative permeabilities.

where S_b^* is the reduced (or effective) brine saturation, $Q_D = Q/V_{b,m}$, Q is the brine volume produced, $V_{b,m}$ is the mobile brine volume, $t_D = t/T_c$ is dimensionless time, and T_c is the characteristic time defined by

$$T_c = \frac{\mu_b \phi^* L}{(2\pi f)^2 r_m \Delta\rho k},$$

where μ_b is brine dynamic viscosity, ϕ^* is reduced core porosity, L is core length, f is rotational frequency (Hz), r_m is radial distance from rotational axis to center of core, $\Delta\rho$ is the differential brine-gas density, and k is the single-phase permeability of the sample. The reduced brine saturation at time t_D is given by (Hagoort, 1980)

$$S_b^*(t_D) = 1 - Q_D(t_D) + t_D \frac{dQ_D}{dt_D}.$$

Hence, the relative permeability and saturation are determined from plots of the normalized cumulative brine produced (Q_D) and dimensionless time (t_D). The brine produced is typically measured by use of a stroboscope, camera, and sample (core) holder with a graduated tube attached to it. The centrifuge method has been used by others including Spronsen (1982), O'Meara and Lease (1983), Ward and Morrow (1987) and Nimmo et al. (1987) to measure relative permeabilities.

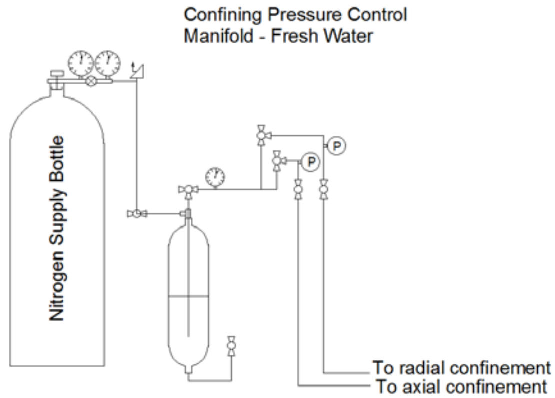

Figure 4. Hassler Cell Confining Pressure Control Manifold

4.4.3 Flow Measurement and Control Systems

Samples tested in Hassler cells will be placed under confining pressures to represent confining pressures associated with underground openings or sealing systems. A radial pressure will be applied through the pressurization sleeve (see Figure 3), while an axial pressure can be applied through the distribution plugs on the effluent end of the core. These pressures can be used to induce desired triaxial stress states. Fresh water will be used to pressurize the radial and axial core surfaces in a manifold configured similar to that shown in Figure 4. Confining pressures of up to 8 MPa will be used in the tests.

Flow rates through the sample will be controlled and measured in the Hassler cell tests under constant inlet and outlet pressure conditions. Gas flow rates will be measured by monitoring pressure drop in a supply bottle in a flow control manifold configured as shown in Figure 5. Constant pressure will be maintained on the porous disk at the high-pressure end of the sample using a gas regulator or some other such device. Gas will be introduced to the sample through an injection tube on the low pressure side of a porous disk configured with a network of distribution channels. Gas will be collected and vented at the sample outflow end in a network of connected channels on the high-pressure side of the porous disk as shown in Figure 3.

Pressurized brine will be introduced to the core through flow distribution channels in a steel distribution plug that is located on the high pressure side of a porous disk used to introduce the gas phase. Brine will be collected and vented from the sample through a brine collection plug on the low pressure side of the core after passing the porous disk gas collection disk. Brine flow rate will be monitored using a flow control manifold as shown in Figure 6. The sensitivity of this manifold can be changed by changing the diameter of the injection leg of the brine reservoirs. Pressure on the low pressure end of the sample can be throttled with a needle valve connected to the brine effluent line. Flow rate on the effluent line could be measured under pressure using a system similar to that on the injection side if desired.

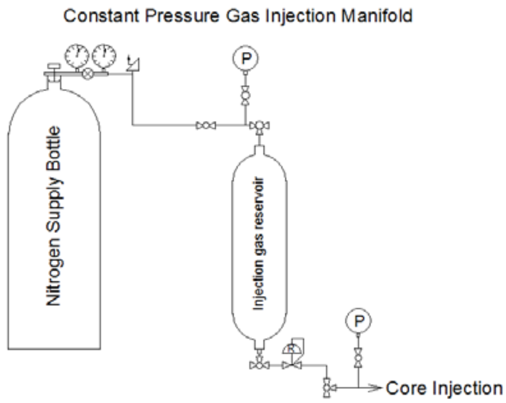

Figure 5. Constant Pressure Gas Injection Control Manifold

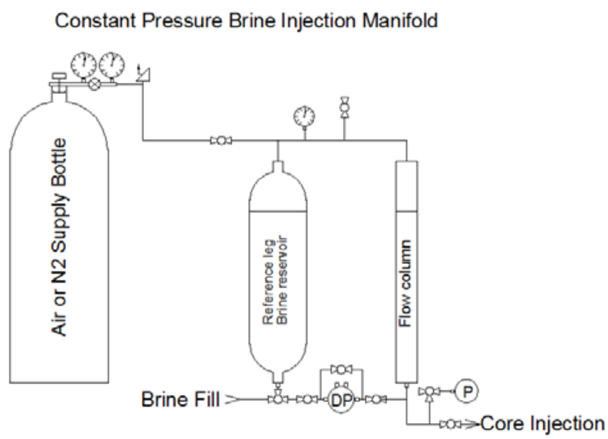

Figure 6. Constant Pressure Brine Flow Control Manifold

4.4.4 Test Equipment Calibration

Testing equipment will include essential instrumentation that needs calibration to comply with data qualification requirements. Gages and equipment needing calibration are listed below as a function of measurement type (discussed above):

- Capillary Pressure Tests
  - Pressure transducers – these will be calibrated by the SNL Primary Standards Calibration Laboratory (Cal Lab)
  - Graduated cylinders – this will be procured with certificates of conformance from Fisher Scientific
  - Centrifuge tachometer – calibration or certification of this will likely need to be worked through the manufacturer.
  - Centrifuge graduated cylinders – accuracy of these graduated cylinders will be confirmed using mass balances in the Carlsbad laboratories.
  - Mass balances – calibrations will be confirmed in accordance with scale operating procedures using masses that have been certified by the Cal Lab

- Relative permeability tests (using flow measurement and control systems)
  - Pressure transducers – used these will be calibrated by the Cal Lab
  - Graduated cylinders – this will be procured with certificates of conformance from Fisher Scientific
  - Mass balances – calibrations will be confirmed in accordance with scale operating procedures using masses that have been certified by the Cal Lab

5 DATA-ACQUISITION PLAN

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

- Electronic data from a data acquisition system (DAS)
- Manually collected test data.

5.1 Scientific Notebook(s)

A scientific notebook(s) will be used in accordance with FCT QAP 20-2 (see Subsection 9.4) to document all Sandia National Laboratory (SNL) activities and decisions during the Test Plan. 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;
- 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 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.

5.2 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.

5.3 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.

5.4 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; and
- Real-time analysis of the acquired data to assess transducer functioning and proper operation of the DAS.

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 such deviations from planned operations in the scientific notebook(s).

6 SAMPLING AND SAMPLE CONTROL

Bedded salt crushed salt samples will be prepared under this Test Plan using WIPP mine-run salt. Following preparation and testing, the samples 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 control following requirements in FCT QAP 13-1 *Control of Samples.*

7 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.* Specific areas of training include:

- Operations of the various pressurized flow control manifolds,
- Operation of the centrifuge,
- Procedures for core handling to maintain saturation as needed,
- Procedures for operating the DAS, and
- Procedures for configuring Hassler cell set-ups with samples and gas and brine distribution plugs.

8 QUALITY ASSURANCE

8.1 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 repository design considerations and repository performance predictions.

8.2 Quality Assurance Program Description

Activities are conducted in accordance with the requirements specified in the Fuel Cycle Technologies (FCT) Quality Assurance Program Document (QAPD).

8.3 QA Procedures

The Quality Activity Plans (QAPs) and activity Specific Procedures (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, and
- FCT **QAP 20-2** Scientific Notebooks.

A complete discussion of this integration is given in Appendix A. Modification to these procedures may be required during testing activities. Such modifications 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.

8.4 Manufacturers QA Procedures

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

None.

8.5 Data Integrity

Care will be taken while conducting these test activities to ensure the integrity of all data collected including documentation on hard copy and electronic 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.

8.6 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. Published technical documents and FCT deliverables constitute transparent and open records of this work.

8.6.1 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 sample description and handling forms, fully completed
- Chain-of-Custody Forms.

8.6.2 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 Records Center. These records include:

- safety briefings;
- Environmental Safety and Health (ES&H) documentation;
- as-built diagrams of equipment;
- equipment manuals and specifications; and
- equipment manifests.

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

8.6.3 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 in accordance with the SNL Records Management Manual and IM 100.2.2 Control of Records (Manage and Protect Information).

9 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 Job Hazard Analysis (JHA), Engineered Safety Package, and a Pressure Safety Data Packages for pressure and flow control systems used by SNL.

10 PERMITTING/LICENSING

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

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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 06212 (Repository Performance), within SNL's Nuclear Energy and Fuel Cycle Programs (06200. Figure A-1 shows the QA requirements interfaces for the activity.

This R&D activity, managed as Work Package FT-14SN081801 and FT-14SN081805 (the former to develop the test plan, and the latter to conduct the testing), 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. Table A-1 reflects the outcome of QA grading considerations.

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1 U.S Department of Energy, Office of Nuclear Energy, Fuel Cycle Technologies Quality Assurance Program Document, Washington, D.C., December 20, 2012.

2 Sandia's Quality Assurance Program Description SNL-QAPD-2014-05-30, Rev:4.0 May, 30, 2014

3 Sandia National Laboratories Used Fuel Disposition Campaign 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 Revision 1; February 15, 2013.

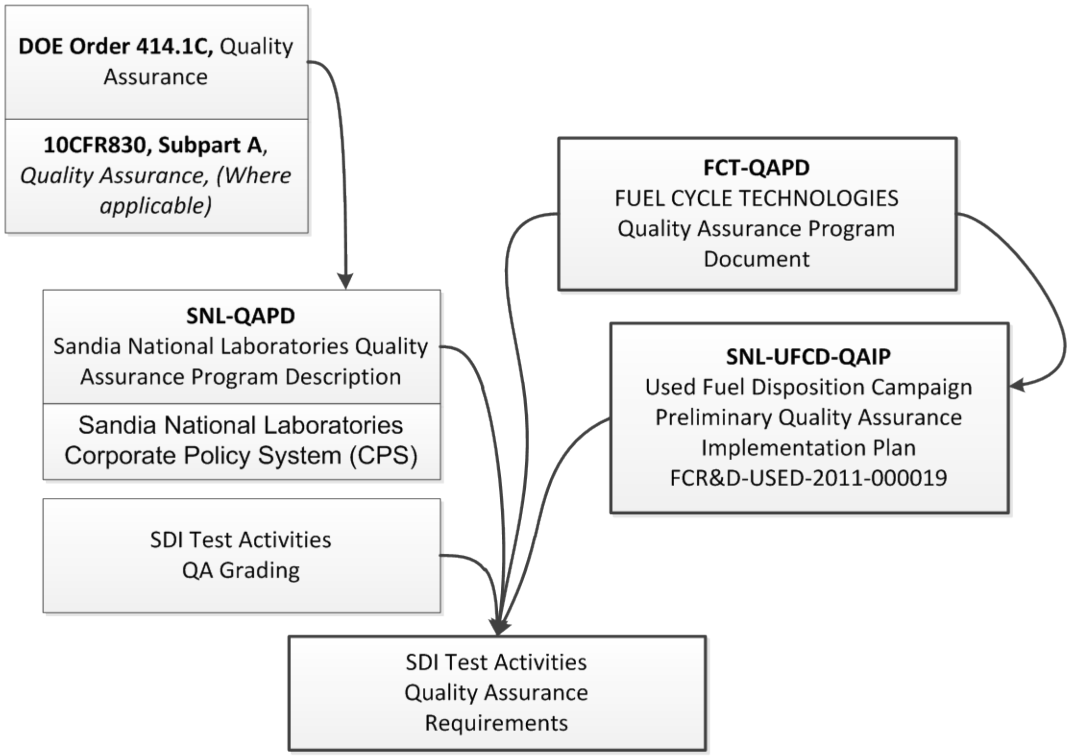

Figure A-1. General QA Requirements Flow Down for SDI Activities.

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

Table A-1. Graded Quality Assurance Requirements for SDI Activity

NQA-1 (2008) Requirement Excerpt[4]	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 **CG100.6.19** - Conduct Management Review
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.	**CG100.8.1 -** Perform Work **CG100.8.2 –** Manage Projects Throughout Their Lifecycle **CG100.8.3 -** Apply Configuration Management Principles to Documents and Physical Items **FCT QAP 20-1** Test Plans

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.

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.	**CG100.1.2 -** 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.	**ME100.3.1CG100.8.1 -** 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 CG100.8.1 -** 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 CG100.8.1 -** 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 CG100.8.1 -** 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, gauges, 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 CG100.8.1 -** 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 CG100.8.1 -** 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 **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 -** Determine and Take Action

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 **IM100.2.5-** 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** - Plan and Perform Assessments **CG100.6.7** - Conduct and Manage Audits