SANDIA NATIONAL LABORATORIES

WASTE ISOLATION PILOT PLANT

TEST PLAN, TP 99-04

Disturbed Rock Zone Characterization Test Plan

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1. APPROVALS PAGE

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3. REVISION HISTORY

The following text is the first revision of this test plan (TP) which incorporates a section on sample (core) control, added and deleted references, reference to the current procedure (SP 13-2) for drilling, logging, and transportation of core, the addition of Attachment A and minor editorial changes. Changes to this plan, other than those defined as editorial changes per Nuclear Waste Management Program (NWMP) quality assurance (QA) procedure NP 20-1, *Test Plans*, shall be reviewed and approved by the same organizations as those who performed the original review and approval. All TP revisions will have at least the same distribution as the original document.

4. PURPOSE AND SCOPE

4.1. Importance of the DRZ

The Waste Isolation Pilot Plant (WIPP) near Carlsbad, New Mexico, is a mined, underground repository recently certified by the Environmental Protection Agency (EPA) for the management, storage, and disposal of transuranic (TRU) radioactive wastes generated by US government defense programs. The waste will be emplaced in panels excavated at a depth of around 650-m in the Permian-age Salado Formation. Following emplacement of waste and backfill, the panels will be closed with an approved concrete closure. The excavations are linked to the surface by four shafts that ultimately will be decommissioned and sealed.

The Salado Formation comprises a thick sequence of bedded evaporites, dominated by halite, but with interbedded sulfate-rich anhydrite and polyhalite layers and some thin clay seams. The repository is excavated in a nearly pure halite layer, just above one of the thicker anhydrite layers, known as Marker Bed (MB) 139. Compared to crystalline rocks, halite or salt creeps readily in response to stress differences. Stress-field alteration and creep closure will create a disturbed rock zone (DRZ) around the excavation. Compared to the intact salt, the DRZ will exhibit increased porosity, increased permeability, and decreased load-bearing capacity. Over time, the DRZ extent, shape, and properties will change as salt creep continues.

4.2. Overall Strategy of the Test Plan

A series of technical meetings have been held with the WIPP Managing and Operating Contractor (MOC; currently Westinghouse TRU Solutions, Inc. LLC) and contractor personnel to define the existing data and data needs associated with monitoring of the DRZ in the underground and in the Air Intake Shaft. The following areas of interest emerged:

- Refined definition of the extent of the DRZ.
- Availability of brine in the DRZ.
- Damage and healing around panel closures.

- Evaluation of geotechnical methods to improve and monitor changes in the DRZ.
- Assurance that the commitment to the EPA to compare monitored parameters to performance assessment models, and provides interpretation regarding expected versus measured behavior is met.

By focusing on the characteristics, extent, and transient behavior of the DRZ, this test plan will be complementary to, rather than duplicating, both extant data and the activities currently being performed under the DCTP. This test plan proposes to evaluate field and laboratory techniques that can be used to obtain data for the parameters shown in Table 4-1. These Test Plan parameters will allow measurement and evaluation of the DRZ and correlations between the fluid flow and geomechanical properties of the DRZ to be established.

Table 4-1: Summary of Planned Field and Laboratory Test Measurements.

TEST PLAN PARAMETER	FIELD MEASUREMENT	LABORATORY MEASUREMENT
Permeability	Fluid Flow Measurements	Laboratory Permeameter and/or Nuclear Magnetic Resonance Testing
Porosity	Resistivity; Gamma Ray Tomography	Laboratory Porosimeter and/or Nuclear Magnetic Resonance Testing
Density	Acoustic Velocity	Laboratory Analysis
Resistivity	Geophysical Measurement	Complete; see Borns, 1990
Acoustic Velocity	Geophysical Measurement	Complete Brodsky, 1990
Storativity	Fluid Flow Measurements	Nuclear Magnetic Resonance
Microstructural Analysis	Core extraction	Petrography
Mineralogy	Core extraction	Petrography and Geochemical Analysis
Hole Closure	Field Measurements	Not Applicable
Pore Water Saturation	Neutron Probe Testing	Low Temperature Desaturation

4.3. Status of DRZ Characterization

A considerable body of experimental and operational data already exists concerning the DRZ, including fluid flow measurements (see Table 4-2). These data provided the basis for the modeling of the DRZ in both the panels and around the shaft seals in the compliance certification application (CCA). This test plan is not intended to repeat previous data collection or duplicate ongoing monitoring. However, there is still considerable uncertainty concerning the DRZ, particularly with regard to its transient behavior, its role in brine inflow, and the likely heterogeneous distribution of the DRZ permeability. SNL and the MOC are currently working jointly to review all data relevant to the presence and characteristics of the DRZ and brine occurrences. This critical review of the data and analyses described in these tables will provide the status of knowledge regarding the halite DRZ. In addition, this will aide in the interpretation of new field data, and will be used in the evaluation of test methods incorporated in Phase II testing.

Table 4-2. Summary of Field and Laboratory Investigations of Salado Halite DRZ.

TEST PROGRAM DESCRIPTION	PRINCIPAL INVESTIGATOR(S)	DISCUSSION
Crack Closure and Healing Studies in WIPP Salt	D.E. Munson, N.S. Brodsky	Laboratory studies evaluating ultrasonic velocity of damaged salt (Brodsky, 1990).
Air Intake Shaft DRZ Study	L.D. Hurtado	Fluid flow measurements in the AIS; laboratory analysis of AIS cores (Dale and Hurtado, 1996).
Hydraulic Testing around Room Q	R.L. Beauheim	Effects of mining on hydraulic properties (Domski, et al., 1996).
DRZ Characterization around a Rigid Inclusion in Salt	M.K. Knowles	Field and laboratory studies on the DRZ around a concrete seal in WIPP Room D (Knowles and Howard, 1996).
Experimental Determination of the Relationship Between Permeability and Micro-fracture Induced Damage in Bedded Salt	D.E. Munson	Laboratory studies of permeability of damaged WIPP salt (Pfeifle, et al., 1998).
Permeability of Natural Rock Salt from the WIPP During Damage Evolution and Healing	L.D. Hurtado	Laboratory studies of permeability during damage and healing of salt (Pfeifle, et al., 1998).
Dilatancy of Rock Salt in Laboratory Tests	F.D. Hansen	Laboratory studies demonstrating the stress-invariant criterion for dilatancy in salt (Van Sambeek, et al., 1993)
A Constitutive Model for Inelastic Flow and Damage Evolution in Salt	D.E. Munson	Theoretical studies of damage evolution in salt (Chan, et al., 1992; Chan, et al., 1994; Chan, et al., 1995a,b; Chan, et al., 1996a,b,c; Chan, et al., 1997;)
Delineation of the DRZ at WIPP	D. Borns	Field studies on extent of the DRZ (Borns and Stormont, 1988).
Brine Sampling and Evaluation Program	D. Deal	Monitoring and analysis of brine inflow into underground boreholes
Excavation Effects Program	C. Francke	Modeling and observations of roof fracture in the underground (Francke et al., 1993)
Ground Control Monitoring Program	R. Carrasco	Extensometer, load cell, and borehole offset monitoring

4.3.1. Visual Observations

The development of a DRZ has been visually observed at WIPP and is evident as fracture and movement along bedding planes in various configurations depending upon rock type, excavation age, and geometry (Francke et al., 1993). At the panel horizon, the dominant fracture pattern to emerge is elliptical/circular around the rectangular openings, except at intersections. Observable fractures in the predominantly halite ribs are parallel to the excavation surfaces. Separations and offsets are common in the floors and backs and tend to be primarily associated with interbeds of anhydrite and clay.

4.3.2. Geophysical Measurements

Geophysical techniques have been used to delineate the DRZ in WIPP halite at the repository horizon. Cross-hole ultrasonic measurements around a new excavation showed dramatic increases of compressional wave attenuation, which was attributed to the existence of a dilated rock zone (Holcomb, 1988). Disturbances were measurable to a depth of at least 3-m next to an excavation with a 5.5-m width. Electromagnetic coupling surveys of mine pillars indicated that rock within 2 m of the excavation surface had significantly less free water content than rock more than 2 m into the pillar (Borns and Stormont, 1989). Seismic surveys conducted on older WIPP pillars indicated the seismic velocity associated with the pillar skin (1 m depth) was noticeably lower than velocities measured deeper in the pillars (Borns and Stormont, 1989), indicating a decrease in formation density. Brodsky (1990) showed an inverse correlation between compressional wave velocity and total strain of WIPP salt specimens in laboratory tests. Cross-hole ultrasonic measurements made in the Air Intake Shaft (AIS) reveal a zone of decreased compressional wave velocity within 0.10 radius of the shaft wall. This evidence suggests that the formation porosity has increased in the shaft wall.

4.3.3. Fluid Flow Measurements

Gas flow measurements were conducted in the rock mass immediately surrounding WIPP excavations. Measurements taken in small diameter boreholes (5 to 15 cm) showed that the rock in the first 1 to 2 m into the ribs generally offered little resistance to gas flow. Tracer tests demonstrated flow paths consistent with the elliptical fracture pattern used to describe fracture distribution around excavations (Stormont et al., 1987; Stormont, 1990). Stormont et al, (1991) reported a substantial increase in halite permeability during drilling of a 0.97 m diameter horizontal borehole immediately upon borehole creation. The zone of enhanced permeability extended radially outward nearly 0.5 m from the newly created borehole.

4.3.4. The Geotechnical Monitoring Program

The EPA requires operational period monitoring of a number of parameters associated with performance assessment. Four of these compliance-monitoring parameters are reported as part of the Geotechnical Monitoring Program (GMP): creep closure and stresses; extent of deformation; initiation of brittle deformation; and displacement of deformation features. Compliance monitoring will be used to detect any substantial deviations from predicted repository performance.

The ongoing GMP activities that are potentially relevant to this test plan include:

- Measurements from geomechanical instrumentation in the shafts and the underground, including: tape extensometer stations, convergence meters, borehole extensometers, piezometers, embedment strain gages, stress gages, inclinometers, load cells, and crackmeters.
- Routine inspections of selected borehole arrays to detect and quantify the occurrences of discontinuities such as fractures and bed separations.

- Geologic mapping in newly excavated areas.
- Brine sampling from underground boreholes to monitor the origins, hydraulic characteristics, extent, and composition of brine occurrences.

Results of these activities are reported annually by the MOC. SNL will evaluate their impact on PA. See Section 6.2 for further discussion.

4.4. Intended Use of Data

Data collected within the scope of this Test Plan may be used in three principal areas:

4.4.1. Performance assessment (PA).

The importance of the DRZ in predictions of repository performance was demonstrated in the recent Annual Sensitivity Analysis (ASA98) and the Near Field Systems Analysis (NFSA). Considerations of the DRZ in PA include: brine inflow, performance of the panel closures, the long-term distribution of fluid in the repository, the mechanical evolution of the repository near-field, and the effectiveness of the seal system through the Salado.

4.4.2. Seal and rock mechanics studies.

The shafts through the Salado also exhibit a DRZ as a result of their excavation and associated salt creep. This DRZ, if not appropriately evaluated or mitigated in seal system design, provides a potential fluid pathway around any shaft seal. Similarly, in the disposal area, the DRZ provides a potential fluid pathway around the panel closures. As a result, EPA selected a panel closure design that involves removal of essentially all the potential DRZ around the closures. Current models predict that any further development of the DRZ will be prohibited by the resistance to further deformation of the surrounding salt provided by the rigid closure.

4.5. Structure of this Test Plan

The test plan is divided into three phases:

I. Initiation of an observational program in the underground workings and Air Intake Shaft (AIS); establishment of a set of underground and laboratory tests to fully characterize the selected underground region, evaluation of methods potentially capable of better definition of the DRZ.

II. Continuation of the program initiated in Phase I; performance of selected underground test techniques positively evaluated that were derived as part of Phase I; continuation of laboratory testing designed to correlate the relevant geotechnical and compliance parameters to PA.

III. Continuation of the program initiated in Phase I; performance of a final set of underground tests to verify findings of Phase II; installation of instrumentation in the AIS and selected underground locations.

Each phase will depend on results of earlier phases and will be modified according to results obtained. Therefore, this version of the test plan presents the experimental program description for Phase I only (Section 5). The description of Phases II and III will be provided in subsequent revisions of this test plan. The experimental program description for each activity includes a background discussion, the experimental process, test and equipment needs, test requirements, and a data acquisition plan. The relative roles of SNL and the MOC are also presented.

Phase I of the test plan is intended to establish a technical baseline for the full characterization program to be undertaken in Phase II. The techniques and evaluations that will be performed under Phase I are discussed in detail in Section 5. In Section 6, Table 6-1 shows how the Test Plan parameters shown in Table 4-1 link to the reporting of the compliance monitoring parameters and, subsequently, how they integrate with the relevant PA models and parameters.

5. PHASE I: EXPERIMENTAL PROCESS DESCRIPTION

5.1. Observational Program

The observational program provides a continuous, methodical means to record the presence of any fluids on the rock surface. Observations will be recorded and, when possible, related to climatic or other events. A formal survey of the Salado stratigraphy in the underground region selected for testing will be conducted by cognizant staff. A detailed mapping of this area will be performed prior to fielding of any tests. Boreholes will be drilled into the footwall and ribs of the region. These boreholes will be monitored on a monthly basis for the occurrence of any flow. Observations of the Salado in the AIS will be conducted on a quarterly basis during Phase I conducted in concert with the MOC inspection schedule. Modification to this frequency may be made for Phases I and II. To the extent feasible, observations will be made in existing excavations and in all newly mined areas.

5.1.1. Experimental Process Description

The survey will adhere to the descriptive procedure described in Holt (1993), such that meaningful and reproducible correlations between lithologies and the presence of fluids can be made. This descriptive technique divides the evaporites in the Salado Formation into sulfate and halite rock types. Sulfate rock types will be classified by their primary mineral constituents (i.e., gypsum, anhydrite, or polyhalite). Halite rocks are classified by the crystal-to-crystal relationships and by the nature of the crystal margins. The crystal-to-crystal relationships are designated by Roman numerals, and the crystal margins/matrix relationships are indicated by lower case letters. If additional detail is added to this fundamental classification system, such description will be included in the scientific notebook.

5.1.2. Instrumentation/Test Equipment and Facilities

Scientific notebooks and digital video equipment will be used for sketches and photographic evidence of the lithotypes to ensure this information is contained in the data record and will be maintained as QA records. Additional equipment needs will be identified as part of the development of procedures for collection and transfer of observations.

5.1.3. Test Requirements

Adherence to the prescribed descriptive procedure is required. A methodical and complete documentation of fluids must be noted. Collection of brines for analysis is discussed in Section 5.4. Uncertainty regarding proper description of any type will be resolved through consultation with cognizant technical staff.

5.2. Core Studies and Sample Control

Characterization of a DRZ in WIPP salt through microstructural analyses has been previously documented (Hansen et al., 1988; Knowles and Howard, 1996). The present program encompasses the techniques used in those analyses and, in addition will use mineralogical studies to establish the nature of flow in the DRZ. These studies will rely heavily upon comparisons between specimens taken at various depths into the rib of the test region, and to those which are assumed to be representative of all undamaged regions in the rock mass. Nuclear magnetic resonance (NMR) imaging of cores will be evaluated for feasibility in characterizing the DRZ. This topic is described separately in Section 5.3.

Samples derived from coring activities will follow the labeling nomenclature described in SP 13-2 “Logging and Management of WIPP Core Samples.” SP 13-2 also addresses logging, transfer, re-coring, sawing, storage, shipping and environmental controls for the core. Two core diameters will be extracted for analysis: 4” and 1.25”. The 4” core is representative of current conditions of the WIPP DRZ and the majority will be consumed in the analysis. The remaining core will be archived for potential future investigation. The 1.25” core will be used to assess the adequacy of the neutron probe measurements (section 5.5.1.3), if this core proves not to be useful (broken/rubble) it will be offered (disposed) to institutions for demonstrative purposes. Archived core will either be stored in the WIPP underground or will be transferred to the SNL Carlsbad Programs Group facilities. Both facilities are environmentally controlled and secure which will prevent deterioration and tampering of the core.

5.2.1. Experimental Process Description

Cores for analysis will be extracted in a nominally horizontal orientation at locations determined “near field” and “far field.” Core diameters should be approximately 10 cm. The exact orientations and locations of the cores will be determined following description of the test area(s) by the MOC. It will be necessary to obtain specimens from the same location over time so that comparisons can be made. The cores will be airdrilled with special care to achieve optimal recovery and condition. The procedure for drilling, logging, and transportation of cores is described in SP 13-2”. The MOC may elect to use this procedure directly, or to develop a similar procedure. Each core will be individually photographed and uniquely identified. Selection of core intervals for analysis will be made after visual observation of the core. The core log forms (SP 13-2-1) and associated photographs will be maintained as QA records.

Existing damage (e.g., microcracks) will be characterized using standard optical, laser-confocal, and scanning electron microscopy (SEM) techniques on appropriately prepared subsamples. An extensive discussion on techniques for microcrack characterization is provided in a previous SNL/WIPP test plan

(Pickens, 1995) and by Carter (1995). Identification of microstructural features including dislocation density and subgrain diameters associated with deformation are also described in the literature (Carter and Hansen, 1983; Spiers et al., 1998) and are not repeated here.

Zones in the underground test area in which weeps were initially abundant will be selected for cores used in mineralogical studies. If the flow of brine continued with evaporation in the DRZ this could be evidenced by deposition of secondary salts on DRZ fracture surfaces. Unlike halite, some of these salts, carnallite and bischoffite in particular are anisotropic and are detectable by petrographic studies. Also, because the weep fluids are much richer in K, Mg, and sulfate than the rock salt, the evaporation of these brines should leave a distinctive elemental signature in the bulk chemistry of the rock. At least three techniques may be used to define chemical precipitate: optical petrology, scanning electron microscopy and bulk chemistry. Of these techniques, scanning electron microscopy provides the most detailed direct measurements of element composition at high magnification. If sufficient deposition for optical microscopy occurs, thin and thick micrograph sections can be prepared and examined. The technical procedure for visual and petrographic description, photography, and subsampling of clay-size materials from core samples will be developed using SNL TOP 554 as a template.

5.2.2. Instrumentation/Test Equipment/Facilities

Petrographic analyses require (as appropriate) the following equipment and instrumentation:

- Leitz petrographic microscope with photomicrography capability;
- Olympus Vanox universal microscope with photomicrography capability;
- Nikon zoom stereoscope with photomicrography capability;
- JEOL JSM T300 scanning electron microscope;
- Zeiss confocal system with appropriate laser source;
- Color print film for optical micrographs; and
- Polaroid Type 553-b/w film for scanning electron photomicrographs.

Scientific notebooks will be used to document the results of the analysis including calibration and standards used tests along with list of equipment used, make, model and operating system (if applicable) and the associated micrographs will be maintained as QA records. This equipment is currently available at Sandia National Laboratories in the Geosciences Center. Equivalent equipment may also be available within the SNL Carlsbad Programs Group facilities. The final determination regarding staff and location of petrographic studies will be made as deemed appropriate by cognizant technical staff of the MOC and SNL.

5.2.3. Test Requirements

It is implicitly assumed in the above discussion that the samples selected for microscopy are representative of the *in situ* material. The coring process results in immediate unloading of the *in situ* stresses. This issue, as well as others related to the fragile nature of salt cores and any appropriate remedial actions, will be addressed at the time of core retrieval and examination. Documentation of the petrographic studies will include a discussion regarding these issues.

Mineralogical studies of DRZ core specimens are scoping in nature, since it is unknown whether the expected mineralogies will be observed. In addition, this exercise represents the first attempt to characterize deposition of salts over approximately a ten-year period in the DRZ fractures. Therefore, the success of the approach is unknown.

5.3. Nuclear Magnetic Resonance (NMR) Tests

The majority of elements in nature have a magnetic moment. When a magnetic field is applied to the nuclei, they align parallel to the field, corresponding to the lowest energy state and are then polarized. The greater the magnetic field, the more nuclei are polarized and the higher the magnetization of the sample. The macroscopic magnetization can be influenced by electromagnetic fields as specific radio frequencies (RF). By transferring energy with such fields into the sample, the magnetization can be turned away from its aligned orientation. This re-orientation generates a radio frequency signal, which is detected. This signal is too weak to be detected for most nuclei, but hydrogen with its large magnetic moment and high abundance has a strong signal. The magnitude of the hydrogen signal is a measure of liquid-filled porosity (SIPM, 1995).

The high magnetic fields created by NMR instruments result in a high signal-to-noise ratio that enables the application of the pulse-echo NMR technique. By this method, the magnetization can be measured over long time periods (approximately 0.1 s). This decay (relaxation) is caused by the interaction of hydrogen atoms in the fluid with their surroundings and has an exponential character. In bulk water, the surroundings consist only of other hydrogen atoms and the relaxation can be described by a single relaxation time, approximately 3 seconds.

This situation is changed in a porous media. Characteristic relaxation times are much shorter (fraction of a second), since the interaction between the fluid and the pore wall introduces an additional means of relaxation. By measuring this relaxation, information about pore size is obtained, which is related to permeability of the media.

A magnetic field gradient is also present and the diffusion coefficient of the liquid can be measured with NMR as a function of increase in relaxation rate. The diffusion coefficient is directly related to the fluid viscosity (Kenyon, et al., 1988).

5.3.1. Experimental Process Description

Extraction of core will follow the process described in Section 5.2. Since the NMR spectroscopy of these kinds of samples has not been reported in the literature, the initial set of experiments will be used to explore the instrumental and interpretative issues associated with these samples. This approach has been developed during discussions with E. Fukushima, president of New Mexico Resonance and author of "Experimental Pulse NMR: A Nuts and Bolts Approach" (Fukushima and Roeder, 1981).

5.3.2. Instrumentation/Test Equipment Facilities

NMR of halite cores will be conducted at a selected commercial facility. Necessary equipment includes:

- Spectrometer with appropriate bandwidth;
- Probe(s) designed to scan cores having a minimum diameter of 5 cm.

Two potential commercial facilities have been identified: the University of Texas at Austin, and the NM Resonance in Albuquerque, New Mexico. Selection of the facility will be based on ability of the contractor to meet necessary Quality Assurance and technical requirements.

5.3.3. Test Requirements

Correlations are required between measured conductivity and the relaxation times of the salt specimens. If scoping tests on core samples are successful; QA records and procedures will be developed describing additional test requirements. At this time, the following calibrations and tests are planned for the Phase I program:

- Conductivity
- Probe tuning and matching
- Free induction decay vs. spin-echo experiments, effects of instrumental response time
- Skin effects of the sample, comparison to standards
- Homogeneous vs. inhomogeneous broadening

Relaxation measurements:

- Appendix A is a memorandum describing preliminary scoping studies on subsamples of Salado halite and anhydrite, which defines T2 and T1.
- Observation of fastest component of T2 decay, free induction decay vs. spin-echo
- Long component of T2 decay, free induction decay vs. spin-echo to determine homogeneous vs. inhomogeneous line broadening
- Pulse saturation to determine range of T1
- Inversion recovery measurement of T1, one to two decades of decay

5.4. Collection of Samples for Geochemical Analysis

Any seepage of brine from test boreholes, newly mined zones, and the AIS may be sampled for testing. These occurrences cannot be predicted in advance. This section assumes that such weeps and seeps will occur, and provides guidelines for retrieval of samples. Analysis of brine samples is conducted by qualified subcontractors to the MOC. It is assumed that samples collected will be analyzed by the MOC subcontractors so that laboratory consistency is maintained. These samples will be controlled according to the MOC technical and quality assurance procedures.

5.4.1. Experimental Process Description

Two general methods exist for collection of brine from samples. A pre-weighed, absorbent laboratory tissue is wrapped in an external, absorbent tissue. The two tissues are pressed against the wet zone, such that fluid is absorbed. The tissues are sealed and marked prior to transport to a laboratory facility for geochemical analysis. At the laboratory, the external tissue is discarded, and the internal tissue is weighed to assess the mass of brine collected. The second method utilizes a pre-weighed capillary tube. Fluid is collected in the capillary tube, which is marked and transported for analysis.

Selection of the appropriate method for brine collection will be made on the success of these methods based on current MOC procedures.

5.4.2. Instrumentation/Test Equipment Facilities

In addition to pre-weighed tissues and capillary tubes, equipment is necessary for geochemical analysis of the fluids. The location and lithology of sampling zone will be recorded as part of the observational program discussed in Section 5.1.

5.4.3. Test Requirements

The mass of brine collected must be accurately assessed. Weighing of tissues and samples will adhere to ASTM Standards. Documentation of the location of the sampled site will conform to the requirements described in Section 5.1.3.

5.5. Moisture Content Analysis

The Salado DRZ bulk moisture content has been measured indirectly through electromagnetic resistivity measurements and directly from laboratory samples. Moisture contents from resistivity measurements range from 0.5 to 1% (by weight) at the rib surface to 2 to 3% at several meters depth (Borns and Stormont, 1988). The lower value at the surface is attributed to dehydration by the mines ventilation system, the upper values are thought to be representative of the in-situ moisture contents. Resistivity measurements will be repeated in Phase I (see Section 5.7). In addition, neutron probe testing will be evaluated for suitability in the determination of *in situ* moisture content (5.5.1). Cores will be analyzed for moisture content using standard laboratory procedures as described in a paragraph in Section 5.5.1.3

5.5.1. Neutron Moisture Probe Tests

5.5.1.1. Experimental Process Description

Troxler™ neutron gages will be employed to measure the moisture content (Φ) of the Salado repository strata. The primary purpose of these measurements is to observe changes in the water content in the *in situ* rock as a function of location and of time since excavation. These measurements will be made in single boreholes drilled into the rib, floor, and, if feasible, back.

This technique of measuring the water content is standard in the environmental, agricultural, and construction fields, and involves measuring hydrogen in water contained in the material surrounding a borehole. The gages work on the principal of neutron thermalization. Fast neutrons emitted by an Americium 241:Beryllium neutron source are thermalized (slowed) by hydrogen in the tested material. Some thermalized neutrons will be bounced back to the probe and detected. Neutron counts measured are proportional to the water content of the material.

The gage contains a microprocessor that allows the instrument to compute and display the moisture content. The display is in units of kg/m^3, lb/ft^3, percent volume (%), count ratio, in/ft, mm/m, or cm/m. Provisions are made to alter the calibration to account for hydrogen (other than free water) and change the slope of the calibration to account for materials that absorb thermal neutrons (Troxler, 1983).

5.5.1.2. Instrumentation/Test Equipment Facilities

The equipment required for these tests is a Troxler Model 3233 Depth Moisture Gage. The gage has a LCD display for manual recording of data. The gage operates through its own internal battery source, which is recharged either by connecting to a 115 volt AC or 12 volt DC negative ground power supply.

The probe obtains readings by dropping a combined source/detector assembly down an access tube to the desired depth. The access tube is permanently installed in a drilled hole. Three types of tubing are commonly in use: aluminum, steel, and polyethylene pipe. Steel is preferred, since aluminum corrodes in the salt and polyethylene with its high hydrogen content decreases the gage sensitivity. Standard, 1.5" nominal, metallic thin-wall electrical conduit, (EMT) is the preferred material due to its strength, ready availability, low cost, and standardized use elsewhere.

5.5.1.3. Test Requirements

Access tube installation: Four steps are required for access tube installation. First, a 1.25" or smaller diameter core is taken dry. This core is used to define the hole lithology and water content with depth. On removal from the core barrel it will be quickly broken into pieces about 15 cm long and sealed in airtight containers. Exact core size will be determined by available core barrel size, but in no case will the coring produce a hole greater than 1.6". The second step will be to carefully ream the hole to 1.5" using either a drag bit, or a reverse taper core bit. The liner will then be gently positioned. Once placed, the liner will be cut so that only 30 cm protrudes out of the rock face. Finally, the borehole location will be surveyed. The details of placement accuracy and sources of error will be described in survey and location sections of test reports.

Core analysis: Within seven days of collection, each core segment will be inspected and visually described. Its ends will be square cut, if needed, and overall dimensions (diameter and length) measured to ± 0.1 mm. The entire core will then be used for a gravimetric water-content measurement. That will require a weight before and after oven drying at 105^{o} C. Volumetric water content will be calculated from gravimetric content and core dimensions. Finally, the core will be stored in a core library in case of future need.

Neutron Probe Calibration: Due to the high neutron adsorption by chloride, the neutron probe will require field calibration to the WIPP salts. Neutron probe measurements are based on a linear relationship between the count in the rock and the volumetric water content in the material being tested. Immediately after each access tube is installed 20, one-minute counts will be taken at 6" intervals along the entire length of the hole. Readings for different lithologies will be separated. Then all readings from similar lithologies will be plotted versus the measured volumetric content to provide a single meter-specific calibration for that rock type. Linear calibration fits will be calculated with Microsoft Excel.

Independent calibration standards will be constructed to test the performance of the probes once a year. The standards will be sealed, and packed in 55-gallon drums of rock salt with an access tube in the center. Constant water content within the drums will be simulated by an ammonium salt. Use of a dry salt is common for neutron-probe calibration and prevents gravity redistribution of water within the standard. If a probe calibration is found to change significantly, the field calibration procedure will be repeated.

Monitoring Sensitivity: Due to the relatively low count rate in salt, all measurements should be at least four minutes or longer. The built-in statistical test routine of the Troxler probes, which performs 20, one-minute counts is a convenient method that will provide a total of 7,000 to 8,000 counts per reading.

All data including calibrations, standards, list of equipment used, make, model and operating system (if applicable) from the moisture content analysis will be recorded in a scientific notebook which will be the QA record for the tests.

5.6. Cross-Hole Acoustic Wave (V_p and V_s) Tests

5.6.1. Experimental Process Description

Piezoelectric transducers will be used to measure V_p, the velocity of compressional waves, and V_s, the velocity of shear waves. The purpose of the tests is to evaluate differences in wave velocities along differing paths through the DRZ. These tests are made by measuring the acoustic wave velocity between two boreholes. Borehole location will be selected to complement other *in situ* tests.

The technique for measurement of acoustic velocity is standard (Mattaboni and Schreiber 1967), and a SNL procedure (TOP 489) was written for the WIPP underground. This procedure may be used as a template for a procedure for measurement of cross-hole velocity measurements.

5.6.2. Instrumentation/Test Equipment Facilities

The equipment required is an electrical pulse generator to drive the transmitter, a switch box to select the transmitter and receiver, an amplifier for the received signal, and a digital oscilloscope to display and record the transmitted and received signal. Standard, off the shelf equipment is readily available for these purposes.

The transducers are piezoelectric crystals glued to the inner surface of the emplacement housing. The transducers are emplaced with a special tool that compresses the spring-loaded housing until it fits easily inside the borehole. After the housing is inserted to the proper depth, as indicated by a scale on the tool itself, the spring can be released. The housing expands and presses tightly against the borehole wall, ensuring good acoustic coupling. This set up has been used successfully for other WIPP *in situ* tests (Holcomb, 1988).

5.6.3. Test Requirements

The primary test requirement is to accurately position the transducers. Accuracy of the velocity measurements is controlled by the uncertainty in the relative locations of the transducers. On the basis of expected changes in velocity as a result of the formation of a DRZ, it is desirable that velocities be determined to an accuracy of 0.5%, which means that the positions of the transducers must be known within 0.5% as well.

Positioning accuracy is affected by the initial survey of the room and the actual placement of the transducers. The details of placement accuracy and sources of error will be addressed in the discussion of results.

All data including calibrations and standards, list of equipment used, model make and operation system (if applicable) from the acoustic wave tests will be recorded in a scientific notebook that will be the QA record for the analysis.

5.7. Resistivity Tests

5.7.1. Experimental Process Description

The resistivity tests will be used to measure the apparent resistivity for the halite along the electrical current path between selected locations in the test region. The precise configuration to be used will be detailed in the MOC workplans. The electrical resistivity of a rock depends on the rock's porosity, pore saturation and cementation, and the chemical composition of the pore fluid. The resistivity values determined *in situ* by this program will provide a measure of variations in these parameters as they change due to development of the DRZ.

5.7.2. Instrumentation/Test Equipment Facilities

The instrumentation for resistivity tests includes a power supply, cabling, electrodes, and voltage and current meters. Equipment used in this type of testing is readily available, and described in previous WIPP test plans (Pickens, 1995). The power supply, and voltage and current meters are combined into one unit. The power supply may be direct current or low frequency alternating current. For underground WIPP applications, the power supply is battery-based. Subsurface electrodes used at WIPP are either porous pots, driven nails or bolts, or a metal weight lowered into a brine-bearing portion of the borehole. Resistivity testing equipment is standard commercial equipment. Because the resistivity system is designed to reduce the electrical interference and ground problems, it is operated independently of the underground power system.

5.7.3. Test Requirements

Because resistivity techniques will be used to assess change in rock characteristics as a function of time, early access after mining is important to establish the baseline. Thereafter, periodic resistivity measurements will be made, with appropriate lengths of time between measurements based on experience.

5.8. In-Situ Gamma Ray Densitometry and Tomography

Gamma-ray density and tomography are well-established technologies. The only physical factor limiting their application is the need to obtain adequate source count rates at the detector. If the system is not adequately sized, results may not have adequate precision for the intended purpose. Primary design specifications of concern are the source energy, source strength, detector-source separation, and detector type.

5.8.1. Experimental Process Description

Figure 1 shows a typical application of gamma densiometer. Two parallel holes are drilled and a liner installed. The liner is only needed to keep the borehole open, and will probably not be necessary at WIPP. A gamma ray source is inserted in one tube and a string of one or more detectors in the other.

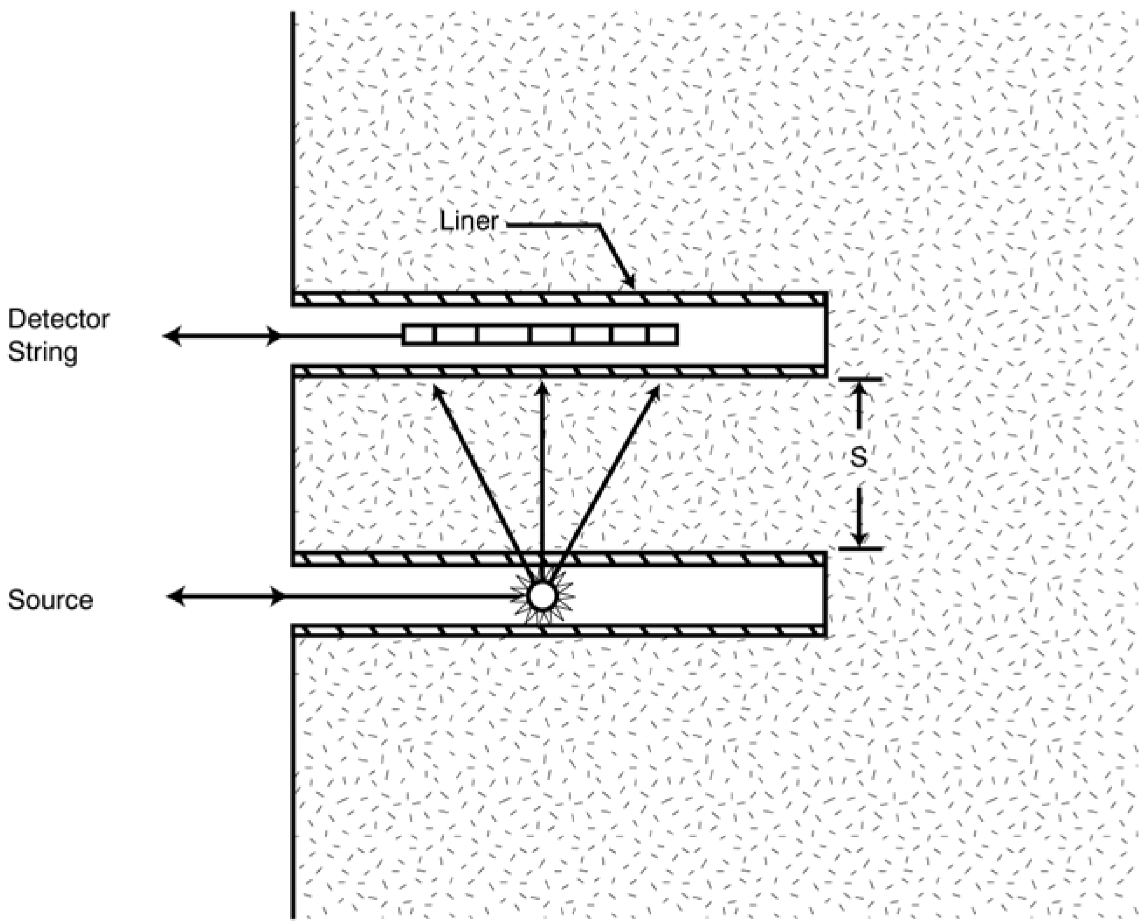

Figure 1. Schematic of in-situ gamma densiometer.

Source and detectors are positioned at known points and gamma rays are counted for an interval of a few seconds to a few minutes. When the source energy is in the range of 200 KeV to 1.2 MeV, the number of gamma rays detected will be proportional to the total mass of rock and fluids between source and detector. Source and detector collimators are usually not used due to space limitations. Thus, the spatial resolution of a reading is roughly equal to the area of the detector normal to the gamma ray path.

In density mode, the total mass between the source and detector is computed from a single count. By taking counts at various positions along the tubes, the linear variation along the tubes is computed. This is a relatively rapid measurement even with only one detector. With one-minute counts, a two-meter deep installation could be measured at 1 cm (linear) resolution in less than four hours.

In-situ tomography is also possible with this setup. Tomography requires taking additional counts at various angles between the holes and multiple detectors become essential. Two-meter deep tubes with 40-cm separation would require about 13 hours to image at 1 cm^2 (area) resolution with a ten-detector string.

5.8.2. Instrumentation/Test Equipment Facilities

Phase I will evaluate the feasibility of *in-situ* gamma ray tomography using a single detector system.

Instrumentation required for the Phase I system includes:

- Sources: 100 and 1000 mCi , ^{137}Cs sealed source (Source gamma ray: 662 KeV at 85% yield);
- Detector crystal: 1 x 1 x 3 cm BGO;
- Photomultiplier tube, tube base and battery powered high voltage supply;
- Laptop computer;
- Multichannel analyzer card;
- Multichannel analyzer spectrum analyzer software;
- Liner tubes: 1.75" O.D, thin wall plastic tubing;
- Source holder and shield; and
- Detector holder.

All components are available from two or more suppliers, with the exception of the holders and shield.

Those components are simple mechanical systems and will be designed and constructed by SNL following appropriate WIPP QA/QC.

5.8.3. Test Requirements

Requirements for access tube installation and core analysis are identical to those described in section 5.5.1.3.

Calibration: The instrument is calibrated at each measurement site by four standard counts. Before and after each borehole survey the source and detector will be positioned just outside the rock face. Counts through air, *(I_o)* and through a standard sample, *(I_s)* will be recorded. The standard sample can be any uniform, convenient material with an attenuation published by the National Institute of Standards and Technology (NIST, 1997). The path standard's length should be adjusted so that the count rate through it is approximate equal to the count rate through the rock salt. The before and after counts are averaged to provide a mean value. The air counts provide the value of I_o for both the standard sample and the measurement. Instrument readings are then calibrated by multiplication of the ratio of the true standard-sample attenuation to the measured.

Monitoring Sensitivity: Radioactive isotopes emit gamma rays randomly and any single count will be an estimate of the long-term mean count. For total counts larger than about 30, the count will be a random normal variable with a standard deviation equal to its mean. This random emission is the primary source of error.

Additional requirements relate to the use of a radioactive source in the underground. This is addressed in Sections 8 and 9.

6. DATA IDENTIFICATION AND USE

Prior to collection of new data in the WIPP underground, a clear understanding of the use of any new data is required. This section presents those activities that address this requirement.

6.1. Data Acquisition Systems for Experimental Programs

6.1.1. Observational Program in the Salado

The classification system will be implemented by the descriptive procedures listed below:

- Graphic Log
- Rock Type Log
- Percent Insoluble Material Curve
- Percent Sulfate/Clay Curve
- Crystal Size Curve
- Written Description
- Photographic Log

Sample forms and discussion of these procedures are found in Holt (1993). The final procedures for the observational program will be developed by SNL which will follow appropriate SNL QA procedures (i. e., NP 12-1 “Control of Measuring and Test Equipment” and NP 20-2 “Scientific Notebooks”.

6.1.2. Petrographic and Mineralogical Studies

Quantitative measurements of the damage structure may or may not be performed depending upon the level of open microcracks observed. Any data obtained will be recorded in a tabular form in a permanent laboratory scientific notebook. Data regarding crack density will be obtained using standard stereological techniques. Photomicrographs, if taken, will be indexed with respect to sample identification and orientation.

These studies are observational in nature. Visual evidence, as well as results from X-ray diffraction (XRD) analysis will be recorded in a permanent scientific notebook. Electronic storage of visual evidence, written notes regarding observations and all other data will be developed as part of this activity.

6.1.3. Geochemical Analysis

Current MOC procedures for data collection and reporting and geochemical analysis of retrieved brine samples will be used.

6.1.4. Moisture Content Analysis: Neutron Probe Measurements

Two types of data are required for the neutron probe measurements: access tube specifications and measurement values. The access tube information need only be recorded at installation. At the installation of each access tube the following will be determined:

- Time and date of installation
- Depth of core sample, hole and access tube installed
- Access tube locator number
- Survey coordinates of tube and relevant stratigraphy

For each measurement event the following will be recorded:

- Time and date of measurement
- Access tube locator number
- Instrument serial number and four-minute standard count value (Once per access hole)
- Neutron counting time interval(s)
- Insertion depth of neutron probe for each measurement point
- Neutron count rate (up to 20 individual measurements at each point)

Survey coordinates will be recorded using MOC procedures. Neutron probe moisture readings will be recorded and reviewed in a timely manner. The procedure for recording of neutron probe moisture measurements will be developed as part of the Phase I program.

The sources for error in the data recordings are primarily human error in operating the control panel settings, and in setting and recording neutron probe positions. Experience has shown that operational errors are the result of missed measurements, accidental erasure of measurements, or mislabeling of disks. Transcribing the LCD display measurements into the field notebook immediately after the measurement will eliminate missed or erased measurements. Any problems with the measurements will be revealed by this procedure. Digital data storage, reduction and analysis will be performed using a Microsoft Excel spreadsheet. No custom programming or macros will be required.

The MOC will be responsible for installation of access tubes, surveying, and data collection and entry. SNL will be responsible for fabrication of instrument standards, instrument calibration in accordance to NP 12-1 “Control of Measuring and Test Equipment” and collection and analysis of core samples.

6.1.5. Cross-Hole Acoustic Velocity Testing

Three types of data are necessary for this test: survey coordinates, transducer locations, and waveform recordings. Survey coordinates will be recorded using MOC procedures. For each measurement location, the following will be recorded:

- Transmitter locator number
- Insertion depth of transmitter
- Receiver locator number
- Insertion depth of receiver
- Oscilloscope data disk number
- Oscilloscope data disk track(s) used
- Travel time (quick look data).

Waveforms will be recorded on floppy disks for later transfer to permanent media and hard copy.

The sources for error in the data recordings are primarily human error in switching the transducer pairs, operating the oscilloscope, and in setting and recording transducer positions. Experience has shown that operational errors are the result of missed recordings, accidental erasure of recordings or mislabeling of disks. Missed or erased recordings will be eliminated by replaying to the oscilloscope screen each of the recorded waveforms for a given transducer location, before the transducers are moved. Any problems with the recordings will be revealed by this procedure. Errors in setting and recording transducer positions will be controlled by internal consistency checks using the quick-look results for the travel time, read directly from the oscilloscope. For each transducer pair, the travel time will be read from the replayed waveform record before the transducer is moved. Unusual waveforms or large discrepancies in the travel times, as compared to the previous position, will be an indication that an error may have been made and the data should be reexamined.

Collection of this data is the responsibility of SNL and will follow appropriate SNL QA procedures (i. e., NP 12-1 “Control of Measuring and Test Equipment” and NP 20-2 “Scientific Notebooks”).

6.1.6. Resistivity Measurements

The data acquisition steps are as follows:

- Place electrodes and determine the distance r(m) between sampling locations.
- Place external ground or reference electrode (see note below).
- Setup and calibrate resistivity system.
- Induct a current by the use of two 12-Volt sealed dry cell batteries (measure current, I, in mA)

- Measure the induced and background potential differences between electrodes, and measurement of the electrical current (measure potentials, V, in mV).
- Calculate apparent resistivity using Ohm's Law.
- Document implementation, data collection and significant occurrences in logbooks.

Collection of this data is the responsibility of the MOC and some training of MOC technical staff will be required. SNL will provide technical staff capable of training underground workers in all necessary procedures associated with data collection.

6.1.7. Gamma Ray Tomography

Two types of data are required for the gamma ray measurements, access tube specifications and measurement values. The access tube information need only be recorded at installation. At the installation of each access tube the following will be determined:

- Time and date of installation
- Depth of core sample, hole and access tube installed
- Access tube locator number
- Survey coordinates of tube and relevant stratigraphy

For each measurement event the following will be recorded:

- Time and date of measurement
- Access tube locator number
- Air count value (Once at the beginning and end of the borehole survey)
- Standard sample count value (Once at the beginning and end of the measurement)
- Gamma counting time interval
- Insertion depth of source and detector for each measurement point
- Gamma count rate

Survey coordinates will be recorded using MOC procedures. Neutron probe moisture readings will be recorded and reviewed in a timely manner. The procedure for recording of gamma ray measurements will be developed as part of the Phase I program.

The sources for error in the data recordings are primarily human error in operating the control panel settings, and in setting and recording instrument positions. Experience has shown that operational errors are the result of missed measurements, accidental erasure of measurements, or mislabeling of disks. Transcribing the display measurements into the field notebook immediately after the measurement will eliminate missed or erased measurements. Any problems with the measurements will be revealed by this procedure. Digital data storage, reduction and analysis will be performed using a Microsoft Excel spreadsheet. No custom programming or macros will be required.

The MOC will be responsible for installation of access tubes, surveying, and data collection and entry. SNL will be responsible for fabrication of instrument standards, instrument calibration, and collection and analysis of core samples according to NP 12-1 “Control of Measuring and Test Equipment.”

6.2. Management of Data

The data produced as a result of this test plan has two potential uses: to augment the compliance monitoring commitments; and to extend the understanding of the DRZ to support PA modeling. Some uses will be qualitative (to help support and/or evaluate conceptual models), while other uses will be quantitative (to develop parameter values and/or verify mathematical and numerical models). The data will be applied across a range of current SNL technical activities, as set out in the SNL Compliance and Recertification Program Strategy document (Rev. 0, 1/15/99).

Table 6-1: Mapping relating test plan parameters to potential data uses.

TEST PLAN PARAMETER	RELATED COMPLIANCE MONITORING PARAMETER	DIRECTLY-RELATED PA PARAMETER	DIRECTLY-RELATED FEP INCLUDED IN THE 1996 CCA PA	DIRECTLY-RELATED MODELLING ASSUMPTION
Permeability Density	Creep closure Extent of deformation	DRZ intrinsic permeability Shaft DRZ intrinsic permeability	Disturbed rock zone Salt creep	The DRZ has constant permeability higher than intact halite.[1] The shaft is surrounded by a DRZ that heals with time. The change in permeability across the shaft DRZ is log-linear.[2]
Porosity	Creep closure Extent of deformation	DRZ effective porosity	Salt creep Disturbed rock zone	The DRZ porosity is constant and equal to the porosity of pure halite plus 0.29%.[1]
Resistivity Acoustic velocity	Extent of deformation	DRZ thickness (set in BRAGFLO grid)	Disturbed rock zone	The dimensions of the repository geometry [including the DRZ] are constant.
Mineralogy	-	-	Fracture infills	Only advection is modelled in the Salado.
Pore water saturation Storativity	-	DRZ initial brine saturation DRZ residual brine saturation and other two-phase flow parameters DRZ; specific storage	Brine inflow	Initial pressure conditions: excavation and waste emplacement result in partial drainage of the DRZ. Initial saturation conditions: the shaft is fully saturated.

TEST PLAN PARAMETER	RELATED COMPLIANCE MONITORING PARAMETER	DIRECTLY-RELATED PA PARAMETER	DIRECTLY-RELATED FEP INCLUDED IN THE 1996 CCA PA	DIRECTLY-RELATED MODELLING ASSUMPTION
Microstructural analysis	Creep closure Extent of deformation Initiation of brittle deformation Displacement of deformation features	DRZ pore size distribution DRZ matrix tortuosity DRZ thickness (set in BRAGFLO grid)	Salt creep Changes in the stress field Excavation-induced changes in stress Disturbed rock zone Consolidation of seals	-
Hole closure	Creep closure	DRZ outer radius at each shaft Air Intake shaft, salt-handling shaft, waste-handling shaft, air-exhaust shaft radii	Salt creep Changes in the stress field Excavation-induced changes in stress Disturbed rock zone Consolidation of seals	The amount of creep closure is a function of time, gas pressure, and waste matrix strength.

1) These assumptions form the basis for screening the following FEPs as accounted for in the CCA PA through the modeling of the DRZ: rock falls, seismic activity, gas explosions, underground boreholes. If the CCA PA DRZ model is changed and these assumptions are removed, then the listed FEPs will need to be re-screened. 2) This assumption was not actually stated in the list of modelling assumptions in Appendix MASS of the CCA. It is stated in Appendix PAR.

In order to ensure the integration of the test plan data across all the activities they concern, the reporting of the test plan results will be done within the framework of the DOE's Research Data Management (RDM) model. Indeed, data collection and analysis described in this test plan will be performed on a timescale that will provide a useful test of the capability, flexibility and functionality of the developing RDM model. The RDM model is intended to provide a networked relational database that will improve traceability and communication across the WIPP project. It is currently developed at a conceptual level that integrates information across Carlsbad Field Office (CBFO) and CBFO prime contractor functions, and consists of a set of data entities (with associated tables) and relationships between the entities, plus definitions of the entities and relationships. SNL has begun development of a set of the conceptual RDM components, including components intended to manage the far-field monitoring database. The RDM is expected to be fully implemented prior to the first recertification.

Implementation of the RDM model will continue throughout the period in which this test plan is running. As they are developed, RDM components will be used for retention, control and communication of data generated as part of this test plan. Some of the required components are expected to be developed prior to initiation of Phase 1, as part of the RDM implementation project focused on the management of far-field monitoring data. Other RDM components are expected to be developed in the course of the test plan.

7. TRAINING

Data generated under this test plan may be used in the SNL WIPP Performance Assessment calculations. Therefore, all activities performed under this test plan will be performed under quality assurance (QA) procedures which are consistent with the requirements specified in the CBFO Quality Assurance Program Description (QAPD), CAO-94-1012. All personnel associated with this test plan will be qualified in accordance with all applicable QA requirements prior to performing any quality affecting work.

Participants affiliated with the MOC will follow the approved MOC QA Program. The qualifications of the Sandia participants will be documented on Nuclear Waste Management Program (NP) Form NP 2-1-1, *Qualification and Training Form* as per NP 2-1, "Qualification and Training." Sandia National Laboratory personnel who perform work under this test plan shall receive SNL WIPP QA training in accordance with NP 2-1. Activity/project specific (SP) procedures will be developed for repetitive operations and personnel will be trained to these procedures.

Non-Sandia participants under contract to Sandia will follow the same training and qualification processes as Sandia participants. Non-MOC participants under contract to MOC follow the same training and qualification procedures as the MOC participants.

Participants working with the neutron probe and gamma ray tomography required the following, or equivalent, training:

- RAD 102, General Employee Radiological Training
- RAD 210, Radiological Worker I Training
- RAD 230 Radiological Worker II Training
- RAD250, Management of Radiological Operations (Managers and supervisors of those involved in SNL radiological work activities)
- ELC 105, Basic Electrical Safety Awareness

8. HEALTH AND SAFETY

All the health and safety requirements relevant to the proposed monitoring activities will be addressed in the procedures related to the work activity. The work described in this Test Plan will require that personnel use radiation-generating devices (RGD). Operating and handling these RGD devices will require personnel appropriately qualified as Radiological Workers. All hazards, both chemical and radiological, will be identified and evaluated before personnel are exposed to these hazards. All employees have the authority to stop work if they have a safety concern. Other required ES&H training includes General Employee Training, General Employee Radiological Training, and Federal Metal and Nonmetal Mine Safety and Health Training Regulations, as appropriate to task and location.

9. PERMITTING/LICENSING

Permits and licenses required will be obtained by the MOC prior to testing initiation.

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11. APPENDIX A: NMR SCOPING STUDY OF SALADO HALITE AND ANHYDRITE

Sandia National Laboratories

Operated for the U.S. Department of Energy by
Sandia Corporation

Albuquerque, New Mexico 87185-1407

date: March 26, 1999 March 26, 1999

to: David Guerin, MS 1395, Org 6811

from: Roger A. Assink, MS 1407, Org 1811

subject: Characterization of Salt and Anhydrite samples by NMR Spectroscopy

We performed NMR spectroscopy measurements on 8 samples that were prepared by Tom Pfeifle of Repec. The following table duplicates the short description provided with the samples:

Sample Number	Description
L4X01-7/1-2/1-7/T-1	Argillaceous salt
C1X06-3/1-1/2-7/1-2	Clean WIPP salt
C1X06-3/1-1/2-7/1-3	Clean WIPP salt
C1X06-3/1-1/2-7/1-4	Clean WIPP salt
C1X06-3/1-1/2-7/1-5	Clean WIPP salt
C1X06-3/1-1/2-7/1-6	Clean WIPP salt (glass ampoule)
TV10-3-2/1-1	WIPP Anhydrite (red)
MX05	WIPP Anhydrite (white)

All measurements were conducted at room temperature on a Bruker AMX 400 spectrometer with a 10 mm broadband probe. The probe tuned quite easily for each of the samples. Very little adjustment for either the match or tune capacitors was required. The 90° pulse width was 42 μs. Since there was some interference from the background of the probe, spectra of the empty probe were recorded under identical conditions. In order to reduce the background, a pulse cycling program called "aring" was implemented. The program applies an effective 90° pulse by the vector addition of three 90° pulses. The sample, which experiences a relatively homogeneous rf field, provides a signal approximately 80% of the signal achieved with a single 90° pulse, while the background hydrogens outside of the coil are attenuated by a factor of 3. After suitable adjustments for the background, both approaches gave similar results.

Results

The integrals from the samples were compared to the signal from a sample of 50 µl of 1:19 H_2O:D_2O or a total volume of 2.5 µl of H_2O. The results for the single pulse sequence, "zg" and the "aring" sequence are shown in the following table. Spreadsheets showing the samples (identified by the last two digits), file names, weights, raw signal areas, the signal areas minus the blank area, and the calculated water percents are attached. Also attached are the spectra for representative single pulse spectra, "zg", and all of the "aring" spectra. All spectra were plotted on the same vertical scale with the exception of sample T-1 whose vertical scale was divided by a factor of 4. The 42 µs pulse provides a spectral width of $\pm$ 30 ppm over which the signal intensity is greater than 80 % of its theoretical value. Inspection of the spectra indicates that this power level appears sufficient to excite the entire linewidth of the water signal.

Sample Number	Wgt (%), zg	Wgt (%), aring
L4X01-7/1-2/1-7/T-1	0.36	0.38
C1X06-3/1-1/2-7/1-2	0.13	0.13
C1X06-3/1-1/2-7/1-3	0.12	0.13
C1X06-3/1-1/2-7/1-4	0.08	0.09
C1X06-3/1-1/2-7/1-5	0.10	0.09
C1X06-3/1-1/2-7/1-6	0.10	0.10
TV10-3-2/1-1	0.05	0.05
MX05	0.05	0.05

The T_1 and T_2 relaxation times were recorded for samples 1-2 and 2/1-1. An "aring" modification of the inversion recovery method was used for the T_1 measurement to reduce background effects. A normal spin-echo pulse sequence was used for the T_2 measurement since the background hydrogens outside of the probe were not refocused by the pulse sequence. The following tables show the relaxation times calculated for each of the samples. Single component and two component fits were applied to each of the measurements. Also shown is the chi-square (Chisq) value for each fit. Attached are the relaxation plots for each of the samples.

T_1	Intensity (%)	Time (s)	Chisq
C1X06-3/1-1/2-7/1-2			
1 comp	100	0.68$\pm$0.02	0.006
2 comp			
comp A	59	0.48$\pm$0.05	0.0007
comp B	41	1.15$\pm$0.17	

TV10-3-2/1-1			
1 comp	100	0.33±0.02	.02
2 comp			
comp A	11	0.035±0.03	.008
comp B	89	0.37±0.03	

T_2	**Intensity (%)**	**Time (ms)**	**Chisq**
C1X06-3/1-1/2-7/1-2			
1 comp	100	9.1±0.6	.014
2 comp			
comp A	53	3.7±0.4	.0004
comp B	47	17±2	
TV10-3-2/1-1			
1 comp	100	1.9±.02	.00006

The T_2's are significantly smaller than the T_1's for each of the samples. Using the single component fits, the ratio is 75 for sample 1-2 and 174 for sample 2/1-1. A two component fit gave better results for sample 1-2. This is seen by the large reductions in chi-square for both T_1 and T_2 and by the fits on the attached plots. A two component fit did not give appreciably better results for the 2/1-1 sample. The chi-square was only modestly better for the T_1, while the chi-square was already quite small for the 1 component fit for the T_2. The scatter in the data does not justify using a function with additional degrees of freedom.

Discussion

The NMR measurements on the WIPP samples presented no unusual experimental difficulties. Probe tuning and matching were routine and varied little from sample to sample. The free induction decays for the salt samples were relatively long. The 42 μs 90° pulse should have sufficient power to excite the full spectral range. The free induction decays of the Anhydrite samples were shorter than those of the salt samples, but still appeared well within the range of the instrument. Unfortunately, the samples appear to have little water remaining in their pores. We detected only 0.1% water for the salt samples, while these samples were believed to have originally contained 0.5 to 2.0% water. Apparently they were exposed to the ambient for an indefinite period of time during which they lost much of the initial water. We crushed two of the salt samples, 1-3 and 1-6, and then dried them for 24 hours at 95 °C. No weight lost could be detected for either sample. We attempted to rehydrate these samples by exposing them to 98% relative humidity at room temperature for 44 hours. The signal intensity increased by only 25%.

The relaxation measurements were successful. The 1-2 sample gave a two component fit for both the T_1 and T_2 measurement, while the 2/1-1 sample gave a single component fit for each relaxation time. The two components indicate that the salt sample contains a distribution of pore sizes. The large T_1/T_2 ratio indicates that paramagnetic impurities play a significant role in the relaxation of water in these samples [Foley et al. J. Magn. Resonance A **123**, 95 (1996)].

Conclusions/Proposals

The following items should be considered if additional NMR measurements are pursued:

1. Water Content, Porosity. It is most important to examine samples containing water in the 0.5 to 2 % range. Higher levels of water mean that interference from the background signal becomes much less difficult to address. We need to compare our values with weight loss measurements to determine the accuracy of NMR. We could also crush a sample and perform the experiment with a 5 mm probe which provides a spectral width of greater than $\pm$ 200 ppm to ensure that we are observing the entire water contribution.
2. Permeability. Since the relaxation times are much shorter than pure water, presumably, the water is an effective probe of the pore size and pore size distribution. Again, it is important to look at sample that is fully hydrated. Higher water levels may change the character of the relaxation results. It should make the experimental requirements less stringent. Straley et al. [Transactions of the SPWLA Thirty-Second Annual Logging Symposium, 1991] have shown that the relaxation times decrease as water is removed by centrifugal force from the samples. Presumable the larger pores with the longest relaxation time are the easiest to drain. Similarly, we may have lost water first from the larger pores by evaporation. I would expect that samples with additional water would exhibit a wider distribution of relaxation times than we observed for the nearly dry samples. An increase in signal intensity and a wider distribution of relaxation times would make the analysis and interpretation less difficult.
3. Sample Size. Presently we are limited to 10 mm diameter sample tubes that limit the core size to 9mm. We have several probes from previous instruments that could be adapted to larger core samples. The maximum sample size would be approximately 25 mm diameter by 30 mm long. Adaptation of the probe to our present spectrometer by a commercial source would probably cost a few thousand dollars and require a couple months to implement.
4. Diffusion Measurements. The fast diffusion times expected for water in these samples may provide the means to measure the diffusion constants by a pulse gradient technique. The T_2 relaxation time for the 1-2 sample combined with the 50 G/cm gradients available on our spectrometer and assuming a diffusion constant of $10^{-6}cm^2/s$ indicates that the experiment would be feasible. Longer relaxation times for more hydrated samples would simplify the experiment. Shorter diffusion constants would make the experiment more difficult. These measurements would provide a very direct measurement of the water content and diffusion constant of the samples. Temperature studies would be possible.

These proposals are written in the order in which I would recommend that they be implemented. The first two items would essentially be a repeat of the measurements described in this paper. The experiments would require that samples be placed immediately in an air-tight container. If preparation of cores to fit the NMR tubes is difficult, perhaps fresh samples could be crushed and placed immediately in the NMR tubes. It would be useful to have weight loss or some other analysis of the water content for comparison with the NMR results.

The order in which the last two items are implemented are interchangeable. Diffusion measurements could be implement with existing probes with a modest level of effort.