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Sandia National Laboratories
Waste Isolation Pilot Plant
Revised Test Plan TP97-01

Confirmation of the Ability of a Designed Backfill to Control the Chemical Environment in the WIPP

WBS 1.1.10.1.1
Rev. 1, effective date: 4-15-97

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WPO # 42951

1. Approval Page

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2. Table of Contents

3. Addendum History

Rev. 0 was effective 10/1/96.
Rev. 1 was issued to address the finding ISR97-06-QAF-1. Specifically, the Rev. 0 did not comply with all of the requirements of QAP20-1 whereby the Principal Investigator must be clearly identified and the SNL PI, SNL Project Manager, and the SNL Safety Representative must review and approve the Test Plan. Additionally, the distribution list was incomplete, the effective date of Rev. 0 was listed as being before all of the approval signatures had been obtained, and additional information was required in the references list.

4. Definition of Acronyms

BET	Brunauer-Emmett-Teller
CFR	Code of Federal Regulations
DAS	Data Acquisition Software
DOE/CAO	Department of Energy/Carlsbad Area Office
ES&H	Environment, Safety, & Health
NIST	National Institute of Science and Technology
PA	Performance Assessment
PI	Principal Investigator
QA	Quality Assurance
QAP	Quality Assurance Procedure
SEM	Scanning Electron Microscope
SNL	Sandia National Laboratories
SOP	Standard Operating Procedure
TOP	Technical Operating Procedure
WID	Westinghouse Waste Isolation Division
WIPP	Waste Isolation Pilot Plant

5. Summary

A "backfill" system has been designed for the Waste Isolation Pilot Plant (WIPP) which will control the chemical environment of the post-closure repository to a domain where the actinide solubility is within its lowest region. The actinide solubility is highly dependent on the chemical species which constitute the fluid, the resulting pH of the fluid, and the oxidation state of the actinide which is stable under the specific conditions expected in the repository. Within some scenarios of the WIPP Performance Assessment (PA), there will be a significant quantity of carbon dioxide (CO_2) generated as a result of microbial degradation of carbon-containing waste material (i.e. cellulosics, plastics, and rubbers). The formation of CO_2 has a significant impact on the solubility of the actinides through one or both of the following processes: 1.) Upon contact with water, carbon dioxide reacts with water forming carbonic acid. Carbonic acid, although a relatively weak acid, is capable of driving the pH of the repository into the acidic range where the solubility of the actinides is typically at its highest point. 2.) Carbonic acid undergoes dissociation in water forming carbonate and bicarbonate species. The carbonate ion is known to bind very strongly to the actinides forming stable, relatively highly soluble species. The presence of carbonate in any significant quantity therefore drives the actinide solubility to much higher values due to both lowering pH and forming soluble actinide carbonate complexes.

To mitigate these two detrimental effects of CO_2 generation in the repository, a material is required to maintain the pH of the brines in the alkaline region *and* to remove CO_2 thus minimizing the ability of the carbonate ion to participate in complexation reactions. The alkaline earth oxides (e.g. MgO) were identified as fulfilling these two functions.

The alkaline earth oxides react with water according to the following equation:

$$MgO(s) + H_2O \leftrightarrow Mg(OH)_2(s) \qquad (1)$$

The hydroxide formed, in this case brucite, is then available to react with any carbonic acid which may be available,

$$Mg(OH)_2(s) + H_2CO_3 \leftrightarrow MgCO_3(s) + 2H_2O$$

thus effectively removing the carbonate from the system due to $MgCO_3$'s relative insolubility. The mineralogy of the resulting solid magnesium carbonate phase(s) is one subject of this investigation. Although modeling studies have shown that the exact composition will not significantly impact the pH buffering capacity, this study will determine the exact mineralogy formed and confirm the resulting chemical conditions. Through the implementation of MgO backfill, the range of chemical conditions pertinent to the PA of the WIPP is constrained, and additional assurance of the repository's compliance with the applicable disposal regulations should be obtained.

The experiments briefly described in this Test Plan will confirm the above described reactions and provide data to predict the efficacy of using alkaline earth oxides as backfill. In recognition of the complexity of the WIPP system, additional studies and experiments may be performed as part of this Test Plan to confirm the impact (or lack thereof) of backfill on the balance of the system (e.g. waste strength and transport). This work may also include, as required, support to the Department of Energy/Carlsbad Area Office (DOE/CAO) and Westinghouse Waste Isolation Division (WID) on issues relating to materials specifications and emplacement operations. This additional work, and additional experimental details of the described work will be described in a Test Plan Addendum as required.

6. Regulatory Requirements

There are no unique regulatory requirements applicable to the conduct of this work. All work is expected to be performed at Sandia National Laboratories' (SNL's) facilities under existing Standard Operating Procedures (SOPs)(governing the Environment, Safety, and Health [ES&H] aspects of the work) and Technical Operating Procedures (TOPs) that address the laboratory procedures for conduct of the experiments and operation of the required analytical equipment.

7. Compliance Justification

In the governing regulation for the WIPP (40 Code of Federal Regulations [CFR] 191), measures to provide additional assurance of the repository's ability to comply with the regulation are required. The control of the chemical environment through the implementation of a backfill (e.g. MgO) is one such assurance measure. The work described in this test plan will confirm that the backfill will perform as designed, while not imposing any significant detrimental effects on the balance of the system.

The lack of relevant experimental data confirming the ability of the backfill to function as designed was highlighted as an issue in two of the Peer Reviews performed as required by 40 CFR 194[1,2]. While noting that our theoretical arguments are well founded and presented, the members of the Peer Review Panels still felt the need for direct experimental observation and confirmation.

8. License/Permit Requirements

There are no special license or permit requirements for the work described in this Test Plan.

9. Objective

The objective of this study is to confirm the ability of a backfill (e.g. magnesium oxide) to exert direct control over the chemical conditions that may be expected in the disposal room environment. This control has the objective of providing additional assurance of the repository's compliance with 40 CFR 191. Secondary objectives are to confirm the lack of significant impact of the backfill on the balance of the system and to support DOE/CAO and WID on any issues relating to the emplacement of the backfill.

10. Experimental Process Description

There are four critical questions to be answered by these experiments which will provide the confirmation of the performance of the backfill material:

1. At what rate does $MgO/Mg(OH)_2$ react with CO_2 under dry, humid, and brine inundated conditions?
2. What are the reaction products formed by $MgO/Mg(OH)_2$ reacting with CO_2 under dry, humid, and brine inundated conditions?
3. What are the critical chemical conditions (e.g., brine composition, pH, $[CO_3^{2-}]$) expected in the backfilled repository for scenarios where there is not significant microbially-generated CO_2 and for scenarios where there is significant microbially-generated CO_2?
4. Are there any significant impacts to the balance of the system from the implementation of a backfill?

The experiments necessary to answer the above questions are described in the following sections.

10.1. General Experimental Provisions

These requirements apply to all of the following experiments.

- MgO will be obtained as samples from vendors of industrial quantities of the material. This is in order for the material used for testing to be similar to that which may actually be used as the backfill.
- The term 'brine' or 'brines' refers to synthetic Salado and/or Castile brine prepared by TOP-544. All experiments should be performed for both Salado and Castile brine.
- Where an understanding of the precision of a measurement can be obtained by replicates of an individual measurement, those measurements shall be performed in triplicate.
- Record in the Scientific Notebook the source of all reagents, their lot number (if available), and the purity (if known).

10.2. Development of Procedure for Analysis of Carbonate Formation in $MgO/Mg(OH)_2$ Solids and Slurries

Most of the following experiments require the determination of the amount of carbonate in some other matrix (e.g., $MgO/Mg(OH)_2$ solids and slurries). This task will involve the development of that procedure and its required TOP. The development leading up to the writing of the TOP will be documented in the Scientific Notebook.

10.3. Reaction Rates

The rate at which MgO dissolves has been previously studied[3], but not under conditions similar to those expected to exist in the post-closure repository. Additionally, no data exists for the reaction rate of $MgO/Mg(OH)_2$ with CO_2 for conditions similar to those expected to exist in the post-closure repository (e.g., high ionic strength, high pH). The results of the following experiments will produce the required dissolution rate.

10.3.1. Dissolution Rate of $MgO/Mg(OH)_2$ in Brine

The first activity in this part of the program is to determine the overall dissolution rate of $MgO/Mg(OH)_2$ in brines. Although MgO is known to react with water to form $Mg(OH)_2$[4], most studies of these reactions have taken place in acidic to neutral pH solutions, and none are known to have involved high ionic strength brines. To determine the dissolution rate of MgO, a weighed amount of MgO will be added to brine in a closed, stirred reactor. A sealed reactor is used to minimize the influence of atmospheric CO_2 on the reaction. The small amount of CO_2 trapped in the reactor will not have a significant impact on the experiment. The pmH will be monitored as a function of time until such time as there in no noticeable change in pmH (e.g., the change in pmH is less than 0.1 pmH unit over 1 hour). Since $MgO/Mg(OH)_2$ dissolution raises the pmH, the increase in pmH is directly correlatable to the dissolution rate.

10.3.2. Intrinsic Rate of $MgO/Mg(OH)_2$ Neutralization

The intrinsic reaction rate is the rate at which $MgO/Mg(OH)_2$ will react at a fresh mineral surface. To develop an understanding of this process, MgO in each of the brines and also in deionized water will be added to a sealed, stirred reactor. A sealed reactor is used to minimize the influence of atmospheric CO_2 on the reaction. The small amount of CO_2 trapped in the reactor will not have a significant impact on the experiment. This mixture will be titrated with a weak acid buffer (e.g., acetic acid buffer) and the reaction rate followed by measuring (via automatic titrator) the rate of addition of weak acid buffer necessary to maintain a specific pH. The specific pH maintained should be approximately neutral;the exact value chosen must be documented in the Scientific

Notebook. Additionally, the stir rate will be varied during the experiment to determine whether the reaction is transport dominated.

10.3.3. Reaction Rate Of $MgO/Mg(OH)_2$ With CO_2 Under Dry Conditions

The MgO will be prepared for the experiment by drying the powder for approximately 1 hour at 60° C. The dried powder will be analyzed by x-ray diffraction to confirm the mineralogy (i.e. periclase or brucite) and the surface area determined by Brunauer-Emmett-Teller (BET) analysis.

The dried MgO will be suspended in a fine mesh bag in a gas-tight reaction vessel through which dry CO_2 can be continuously fed. As an alternative to utilizing a mesh bag, this experiment may be performed with the MgO in a column and the gas passed through the column. The choice of configuration will be documented in the Scientific Notebook. The gas inflow and outflow must be monitored and recorded. The gas inflow and outflow may be simply measured using a bubble gauge and the readings manually entered into the Scientific Notebook. More elaborate monitoring methods may be used if documented in the Scientific Notebook. At 24 hour intervals, the MgO/CO_2 reaction product will be sampled, via a grab sample, and analyzed to determine the amount of carbonate formation as a function of time. The intervals for analysis may be adjusted based on the initial observation of the rate of reaction. Any adjustments to the sampling interval will be approved by the Principal Investigator (PI) and documented in the Scientific Notebook.

The solid reaction product(s) will be retained for further analysis.

10.3.4. Reaction Rate of $MgO/Mg(OH)_2$ With Air Under Dry Conditions

The MgO will be prepared for the experiment by drying the powder for approximately 1 hour at 60° C. The dried powder will be analyzed by x-ray diffraction to confirm the mineralogy (i.e. periclase or brucite) and the surface area determined by BET analysis.

The dried MgO will be suspended in a fine mesh bag in a gas-tight reaction vessel through which dry air can be continuously fed. The gas inflow and outflow must be monitored and recorded. The gas inflow and outflow may be simply measured using a bubble gauge and the readings manually entered into the Scientific Notebook. More elaborate monitoring methods may be used if documented in the Scientific Notebook. At 24 hour intervals, the MgO/CO_2 reaction product will be sampled, via a grab sample, and analyzed to determine the amount of carbonate formation as a function of time. The intervals for analysis may be adjusted based on the initial observation of the rate of reaction. Any adjustments to the sampling interval will be approved by the PI and documented in the Scientific Notebook.

The solid reaction product(s) will be retained for further analysis.

10.3.5. Reaction Rate Of $MgO/Mg(OH)_2$ With CO_2 Under Humid Conditions

The MgO will be prepared for the experiment by drying the powder for approximately 1 hour at 60° C. The dried powder will be analyzed by x-ray diffraction to confirm the mineralogy (i.e. periclase or brucite) and the surface area determined by BET analysis.

The dried MgO will be suspended in a fine mesh bag in a gas-tight reaction vessel through which humidified CO_2 can be continuously fed. To humidify the CO_2, the gas will be bubbled through a container of brine prior to being introduced into the reaction vessel. The gas inflow and outflow must be monitored and recorded. The gas inflow and outflow may be simply measured using a bubble gauge and the readings manually entered into the Scientific Notebook. More elaborate monitoring methods may be used if documented in the Scientific Notebook. At 24 hour intervals, the MgO/CO_2 reaction product will be sampled, via a grab sample, and analyzed to determine the amount of carbonate formation as a function of time. The intervals for analysis may be adjusted based on the initial observation of the rate of reaction. Any adjustments to the sampling interval will be approved by the PI and documented in the Scientific Notebook.

The solid reaction product(s) will be retained for further analysis.

10.3.6. Reaction Rate of $MgO/Mg(OH)_2$ With Air Under Humid Conditions

The MgO will be prepared for the experiment by drying the powder for approximately 1 hour at 60° C. The dried powder will be analyzed by x-ray diffraction to confirm the mineralogy (i.e. periclase or brucite) and the surface area determined by Brunauer-Emmett-Teller (BET) analysis.

The dried MgO will be suspended in a fine mesh bag in a gas-tight reaction vessel through which humidified air can be continuously fed. To humidify the air, the gas will be bubbled through a container of brine prior to being introduced into the reaction vessel. The gas inflow and outflow must be monitored and recorded. The gas inflow and outflow may be simply measured using a bubble gauge and the readings manually entered into the Scientific Notebook. More elaborate monitoring methods may be used if documented in the Scientific Notebook. At 24 hour intervals, the MgO/CO_2 reaction product will be sampled, via a grab sample, and analyzed to determine the amount of carbonate formation as a function of time. The intervals for analysis may be adjusted based on the initial observation of the rate of reaction. Any adjustments to the sampling interval will be approved by the PI and documented in the Scientific Notebook.

The solid reaction product(s) will be retained for further analysis.

10.3.7. Reaction Rate Of $MgO/Mg(OH)_2$ With CO_2 Under Brine Wicked Conditions

The brine wicked reaction rate experiments will be performed similarly to that described in 10.3.5. above with the following modifications. The mesh bag of MgO should be suspended in the reaction vessel such that only the bottom of the bag is immersed in the brine. In this way, the brine can wick up into the powder. Prior to gas entering the reaction vessel, presaturate the CO_2 with water vapor by bubbling the gas through a container filled with brine, and then introducing the vapor saturated CO_2 into the reaction vessel beneath the brine liquid level. The gas inflow and outflow and reaction progress (as measured by carbonate formation) will be monitored every two hours for the first eight hours, and then every twenty four hours until no further signs of reaction are evident (i.e. the carbonate concentration change between the last two samples is less than 5%). The intervals for analysis may be adjusted based on the initial observation of the rate of reaction. Any adjustments to the sampling interval will be approved by the PI and documented in the Scientific Notebook.

Note whether the brine remains clear or becomes cloudy. A clear brine solution may indicate that the reaction products are confined to the MgO surface, while a cloudy solution may indicate that at least part of the CO_2 is undergoing reaction in the brine solution.

At least one experiment under these conditions will be performed utilizing a mesh size of MgO large enough that single particles may be removed, sectioned, and analyzed by Scanning Electron Microscopy (SEM). The SEM analysis will allow the determination of whether a reaction rind forms on the particles, thus diminishing the particles' effectivity.

Retain the solid reaction product(s) for further analysis.

10.3.8. Reaction Rate Of $MgO/Mg(OH)_2$ With CO_2 Under Inundated Conditions

The rate of reaction experiments for inundated conditions will be performed as described in section 10.3.7. above with the exception of the MgO will be immersed in the brine without a bag. The intervals for analysis may be adjusted based on the initial observation of the rate of reaction. Any adjustments to the sampling interval should be approved by the PI and documented in the Scientific Notebook.

10.4. Identification of Reaction Product(s)

To confirm that the predictions previously made by the thermodynamic models are valid, it is necessary to determine the chemical and mineralogical composition of the reaction products of $MgO/Mg(OH)_2$ with CO_2. This will be accomplished through a combination of analytical techniques, which may include separation of phases. As a minimum, the solid phase from each experiment described above will undergo x-ray diffraction and major elements analysis (e.g., Mg, Ca, C, O, H). The exact suite of analyses required will be specified upon completion of the x-ray diffraction analysis and will be documented in the Scientific Notebook consistent with Quality Assurance Procedure (QAP) 20-2 (Preparing, Reviewing, and Approving Scientific Notebooks).

Petrology may also be utilized to augment the identification of the reaction products and gain an understanding of the process(es) taking place.

Determination of Resulting Chemical Conditions

This task will involve measuring and assessing any changes in the brine chemistry as a result of the equilibration with MgO. To perform the equilibration, add an excess of MgO to each of the brines (i.e. Salado and Castile) under an inert atmosphere. Stir for approximately 48 hours. Take a sample of the solid and liquid phases for analysis. Specific items to be measured are the concentrations in the brine(s) of the major cations/anions (i.e., Na^+, K^+, Cl^-, Ca^{2+}, Mg^{2+}, CO_3^{2-}, B^{3+}, Br^-, SO_4^{2-}) and pmH. The solid remaining after the equilibration will be analyzed by x-ray diffraction to determine the mineralogical phases present and will undergo a major elements analysis (e.g., Mg, Ca, C, O, H).

To the equilibrated solid and liquid phases from above, add a sub-stoichiometric quantity of CO_2 and allow the equilibrium to be re-established. Take a sample of the solid and liquid phases for analysis. Specific items to be measured are the concentrations in the brine(s) of the major cations/anions (i.e., Na^+, K^+, Cl^-, Ca^{2+}, Mg^{2+}, CO_3^{2-}, B^{3+}, Br^-) and pmH. The solid remaining after the equilibration will be analyzed by x-ray diffraction to determine the mineralogical phases present and will undergo a major elements analysis (e.g., Mg, Ca, C, O, H).

10.5. Assessment of the Impact of Backfill on the Balance of the System

By design, the implementation of a backfill impacts the WIPP system. Most, if not all, of these impacts clearly lead to enhanced repository performance. This task will involve a systematic evaluation of the impacts of a backfill on the repository system and documentation of the results. Where there is a paucity of data or supporting information, additional studies and/or experiments may be defined and included in this Test Plan as a Test Plan Addendum.

10.6. Support of DOE/CAO and WID Materials Specifications and Emplacement Issues

As DOE/CAO and WID develop materials specifications and emplacement strategies for the backfill, issues may arise which require technical evaluations and/or experiments. For example, it may be required to measure the CO_2 permeability of the container material or verify the effective surface area and/or reactivity of a specific backfill product. Such studies will be performed as part of this Test Plan and documented in a Test Plan Addendum.

Instrumentation/Test Equipment/Facility

The following instrumentation/equipment, located in SNL Building 823, is anticipated to be needed to carry out the experiments described in this Test Plan.

Principle Task	Instrument/Device	Manufacturer
Quantification of surface area of solids	Micromeritics ASAP 2000 BET Surface Area Analyzer	Micromeritics Instrument Corp., Norcross, Georgia
Quantification of organic and inorganic ions concentrations	Beckman Model DU-640 UV/Visible light spectrophotometer	Beckman Instruments, Inc., Fullerton, California
Generation of controlled atmospheres for experiments	Labconco Model 50801 Controlled-Atmosphere Glovebox	Labconco Corp., Kansas City, Missouri
Quantification of masses	Mettler AE-163 Balance	Mettler-Toledo, Inc., Hightstown, New Jersey
Quantification of inorganic ion concentrations	TJA AtomScan 25 sequential inductively coupled argon plasma atomic emission spectrometer	Thermo Jarrell-Ash Corp., Franklin, Massachusetts
Quantification of carbon concentrations	UIC Model CM5012 Carbon Analyzer	UIC Inc. Coulometrics, Joliet, Illinois
Quantification of carbon concentrations in solutions	OI Corp. Model 524 D Carbon Analyzing Unit;	OI Corp. College Station, Texas

	Horrida PIR2000 General Purpose IR Gas Analyzer	
Determination of solid phase composition	JEOL T-300 Scanning Electron Microscope	JEOL Tokyo, Japan
Determination of solid phase composition	Philips 3100 X-ray Generator	Philips Electronic Instruments, Mahwah, NJ
Measurement of pH	Orion Model 940 pH meter with Ross Combination Semi-Micro pH Electrode	Orion Research Inc., Boston Massachusetts
Measurement of pH; control of pH	Orion Model 720A960 Autotitration System with Ross Combination Semi-Micro pH Electrode	Orion Research Inc., Boston Massachusetts
Measurement of weight for petrology	Sartorious Balance Model 1465	Great Britain, Ltd. Sutton, England
Measurement of weight for petrology	Mettler Balance PM1200	Mettler-Toledo, Inc., Hightstown, New Jersey
Measurement of weight for petrology	Mettler Balance PM60	Mettler-Toledo, Inc., Hightstown, New Jersey
Visualization of Petrology Samples	Zeiss Axioskop	Zeiss, Germany
Measurement of pH; control of pH	Corning Model 130 pH Meter with Corning Electrode	Corning Glass Works Co. New York

The experimental work will be performed in the laboratories located in SNL Buildings 823 and 6600. Data analysis, interpretation, and project management may also be performed in the Sandia Vista Building.

11. Test/Equipment Requirements

Reaction rates are typically directly tied to temperature. All reactions will be performed at room temperature.

All calibrations will be performed with National Institute of Science and Technology (NIST) traceable standards. This requirement, along with the requirements for instrument calibration, verification of continuing calibration, and specific measurement units are specified in the TOP (listed in Section 20.1) for the specific instrument.

12. Data Acquisition Plan

The means to collect the data varies for each individual instrument being used. Each of the instruments, with the exception of the Mettler AE-163 Balance and the JEOL SEM, have direct data printout as part of their Firmware Data Acquisition Software (DAS). These data printouts will be attached directly to the Scientific Notebook(s) or submitted to the Sandia WIPP Central Files. For the instruments which do not have direct data printout as part of their DAS, the instrument reading will be directly recorded into the Scientific Notebook. For details of the data acquisition for a particular instrument, see the specific TOP for that instrument. A listing of the relevant TOPs is provided in Section 20, References.

From the Scientific Notebook(s), the numerical data will be transferred into a Microsoft Excel (Excel) spreadsheet. A second person will check the data as it exists in the Excel spreadsheet to verify that no data transcription errors have occurred. This verification will be documented via memorandum with a copy of the spreadsheet attached. The data will be directly analyzed from the spreadsheet.

13. Data Identification and Use

The data resulting from these activities will be recorded directly into the Scientific Notebook(s) being used for these Tests. In cases where an instrument provides a printed output, that output will be attached to a page within the Scientific Notebook. All calculations performed as part of the activities covered by this Test Plan will be verified by a second person who will note their concurrence by co-signing the Scientific Notebook. If a discrepancy is found, that discrepancy and its resolution will be documented in the Scientific Notebook. Additionally, there will be periodic Quality Assurance (QA) reviews of the Scientific Notebooks to ensure that the requirements of QAP 20-2 (i.e., use of a single line through errors and initialing and dating of the correction).

Data reflecting the use of triplicate experiments will be directly, mathematically averaged. In the event that some data are determined to be missing or unusable, this fact will be documented in a memorandum along with an assessment of whether the analysis may be continued in the absence of the missing or unusable data or whether the experiment must be repeated.

14. Concept and Hardware Design Analysis

No separate design review is required for this work. The general design of the experiments are being reviewed as part of this Test Plan review.

15. Provisions for Significant Events

There are no significant events anticipated for the work described in this Test Plan.

16. Quality Assurance

All work conducted by SNL and its contractors in support of this Test Plan will be conducted in accordance with the QA requirements described in the SNL WIPP QAPs. Contractors and other laboratories who are anticipated to support this Test Plan are Science Applications International Corporation, TAD, and Los Alamos National Laboratories (performed under their own QA Program under direct contract to DOE/CAO).

16.1. Training

All personnel involved in the experiments described in this Test Plan will be trained and qualified for their assigned work. This requirement will be implemented through QAP 2-2 (Orientation and Training Program) for those working under the SNL WIPP QA Program. Suppliers working under their own QA Plans will be required to maintain equivalent records. All qualification and QA training records will be submitted to, and maintained by the SNL WIPP Central File. The training records for the following ES&H courses are maintained in the SNL Laboratory Information System training database TEDS:

-ESH 100:	ESH Awareness
-PRS 102:	Basic Pressure Safety
-RAD 102:	Radiological Worker Training (if employee is not RAD210 trained and needs access to a Radiological Control Area)
-RAD 210:	Radiological Worker Training

Those personnel working under the SNL WIPP QA Program will be required to submit the following forms to the SNL WIPP Central File prior to beginning work:

Form 404	Certificate of Personnel Qualification
Form 439	WIPP Personnel Database Entry Form
Form 405-B	Training Assignment (including training to TOPs)

Evidence of training to assigned QAPs, TOPs, this Test Plan, ES&H procedures, and any other required training are needed prior to beginning work.

16.2. Procurement Document Control

The procurement documents associated with any procurement of items or services in support of the proposed experiments will be prepared following the guidelines provided in QAP 4-1 (WIPP Supplier Quality Assurance Program Requirements). The review and approval requirements for the procurement documents and any changes to them are also provided in QAP 4-1. The QA review requirement for commercial grade items is waived if they meet the criteria specified in QAP 4-1.

16.3. Document Control

The preparation and issuance of this Test Plan, and any major changes to it in the future will be controlled through QAP 20-1 (Preparing, Reviewing, and Approving Test Plans). Any other document control issues will be governed by QAP 6-1 (Document Control System).

16.4. Control of Purchased Items and Services

Procurement of items and services required for the proposed experiments will be controlled by QAP 4-1 (WIPP Supplier Quality Assurance Program Requirements), which provides QA controls and guidelines for the following, as appropriate:

-source evaluation and selection;
-evaluation of objective evidence of quality furnished by the supplier;
-source inspection;
-audit; and
-examination of items or services upon delivery or completion.

Identification and Control of Items

The requirements for identification and control of data, materials, parts, samples, specimens, and components are provided in QAP 13-1 (Conducting and Documenting Sample Control and Chain of Custody) and will be followed during the conduct of the proposed experiments.

16.5. Control of Processes

The various SNL WIPP QAPs establish processes and procedures to ensure that activities conducted in support of the WIPP are completed in accordance with federal and industrial quality requirements. Any special processes affecting the quality of the activities described in this Test Plan will be controlled by Technical Operating Procedures according to the QA requirements specified in QAP 5-3 (Preparing, Reviewing, and Approving Technical Operating Procedures).

16.6. Inspections

In addition to periodic QA surveillances and audits covering any or all aspects of the experiments being performed subject to this Test Plan, routine safety inspections are anticipated as discussed in the ES&H Standard Operating Procedures for the laboratories where these tests will be performed.

16.7. Test Control

Experimental activities will be controlled and conducted in accordance with the approved Test Plan, a copy of which will be maintained in the laboratory(ies) where the work is being performed, utilizing approved (per QAP 5-3) TOPs (see Section 20.1) and Scientific Notebook(s) (prepared using QAP 20-2). With the written concurrence of the PI, responsible manager, and QA representative, experimental activities may be initiated based on a draft Test Plan pending approval.

16.8. Control of Measuring and Testing Equipment

The calibration requirements for measuring and testing equipment used in the experiments will be controlled according to QAP 12-2 (WIPP Calibration Quality Assurance Program). The calibration procedures for each instrument are described in the various TOPs (see Section 20.1, Relevant TOPs). These procedures will be followed and the calibration data will be kept in the data package for this activity. If these procedures are unavailable or deficient, a calibration procedure will be developed and reviewed following the guidelines provided in QAP 12-2. In addition, all requirements identified in QAP 12-2 regarding staff qualification and training, calibration records including calibration labels, and measurement standards will be observed.

16.9. Handling, Storage, and Shipment

SOPs or instructions for handling, storage, and shipping laboratory equipment and materials related to the proposed experiments have been documented in ES&H SOPs. Those SOPs also include guidance for safety related equipment and materials (e.g., radioactive materials). In addition, QAP 13-1 (Conducting and Documenting Sample Control and Chain of Custody) identify requirements and appropriate forms for documenting and tracking sample possession.

16.10. Inspection, Test, and Operating Status

Many of the instruments to be used in the proposed experiments will require periodic inspections and testing for their satisfactory operation. This requirement will be controlled through calibration stickers, tags, and markings on each item and also through notations in laboratory notebooks. The stickers, tags, or markings on each instrument will clearly show its operating status so that instruments that are due for an inspection/testing or do not pass the required inspection/testing are not inadvertently used.

The status of these experiments will be provided through monthly progress reports conveyed verbally to SNL WIPP Project Management, SNL reports, through presentations at meetings, workshops, and conferences, and through peer-reviewed journal publications.

16.11. Corrective Actions

Conditions adverse to data quality such as power failures during data acquisition, instrument malfunctions or failures, and defective items and their root cause will be immediately evaluated to assess their impact on the experiments and to determine what corrective actions are needed. This evaluation and any corrective actions taken will be documented in a Corrective Action Request using the guidelines of QAP 16-2 (Conditions Adverse to Quality and Corrective Action).

Quality Assurance Records

QA records that are planned to be generated through the activities of this Test Plan are:

- -Test Plan
- -Training and qualification records
- -Procurement documents
- -ES&H SOPs
- -Logbooks
- -Inspections/testing documents
- -Technical and operating procedures
- -Assessment reports
- -Calibration/certification reports
- -Uninterpreted data
- -Technical progress reports and SAND reports
- -Corrective Action Requests

These records will be submitted to the SNL WIPP Central File as required by QAP 17-1 (WIPP Quality Assurance Records Source Requirements). Document records and test data will be submitted to the SNL WIPP Central File when they are finalized or completed, respectively. During the test, any data collected using an automated data acquisition system will be downloaded and backed up in duplicate at least daily.

16.12. Computer Software

No specialized computer software will be developed as part of the work under this Test Plan. DAS that is part of the instrument is verified through the calibration program for that instrument. The DAS utilized with the instruments identified in Section 11 is classified as Firmware DAS per QAP 19-1 and is outside the scope of QAP 19-1 per Table A.1. Off-the-shelf software will be utilized in support of this Test Plan (e.g. Microsoft Excel 5.0 and Microsoft Word 6.0c). Their use will be governed by QAP 19-1 (WIPP Computer Software Requirements) which requires referencing and using vendor-supplied user instruction publications and formal verification of the outputs to be included in the analysis documentation.

17. Health and Safety

All of the health and safety requirements relevant to the work described in this Test Plan and the procedures that will be used to satisfy these requirements are described in the ES&H SOPs. Those ES&H SOPs identify the hazards associated with these experiments and describe the procedures to deal with these hazarding including all the training requirements for personnel involved in conducting these experiments.

18. Milestones

The following milestones are tentatively established for the work described in this Test Plan. The dates may have to be adjusted based on resource allocations and when the preliminary experimental data are obtained.

1. Develop and have approved any required TOPs (10.2)-Nov 15, 1996
2. Complete all personnel training-Nov 1, 1996.
3. Determine the chemical conditions resulting from the equilibration of $MgO/Mg(OH)_2$ with Salado and Castile brine in the absence of CO_2 and in the presence of substoiciometric CO_2 (10.5)-April 30, 1997
4. Determine the reaction rate of $MgO/Mg(OH)_2$ with CO2 under inundated conditions (10.3.8)-April 30, 1997
5. Determine the reaction products of $MgO/Mg(OH)_2$ with CO_2 (10.4)-May 1, 1997

19. References and Controlled Documents

19.1. Procedures and SOPs That May Be Used to Support This Work (Sandia National Laboratories WIPP Project Controlled Documents)

TOP-535 Calibration, Use, and Maintenance of pH Meters and Probes, authored by John Kelly, 01/05/96.

TOP-536 Calibration, Use, and Maintenance of the Atomscan-25 Inductively Coupled Plasma Emission Spectrometer, authored by John Kelly, 01/05/96.

TOP-537 Calibration, Use, and Maintenance of the ASAP-2000 BET Surface Area Analyzer, authored by John Kelly, 01/05/96.

TOP-538 Calibration, Use, and Maintenance of the Model 5012 Carbon Dioxide Coulometer, authored by John Kelly, 01/05/96.

TOP-539 Calibration, Use, and Maintenance of the N4MD Sub-Micron Particle Analyzer, authored by John Kelly, 01/05/96.

TOP-541 Calibration, Use, and Maintenance of the Eppendorf Model 4810 Autoclavable Pipettes, authored by John Kelly, 01/05/96.

TOP-542 Calibration, Use, and Maintenance of the Mettler AE-163 Balance, authored by John Kelly, 01/05/96.

TOP-544 Preparing Synthetic Brines for Chemical Retardation and Transport Experiments, authored by Karen Robinson, 01/05/96.

TOP-549 Calibration, Use, and Maintenance of the PMS-100 Micro Laser Particle Spectrometer, authored by John Kelly, 01/15/96.

TOP-550 Calibration, Use, and Maintenance of the Sequoia-Turner Model 450 Digital Fluorometer, authored by John Kelly, 01/17/96.

TOP-552 Calibration, Use, and Maintenance of Philips XRG 3100 X-Ray Generator, authored by Jim Krumhansl, 03/14/96.

TOP-559	Calibration, Use, and Maintenance of the Aminco-Bowman Series 2 Luminescence Spectrometer, authored by Sherri A. Doolittle, 07/22/96.
SP472887	ES&H Operating Procedure for Chemical Handling for Geochemical Research Department 6118 in Building 823, authored by H. L. Buddy Anderson, 08/06/93.
SP472729	Standard Operating Procedure for Column Experiments in the Chemical Processes Laboratory in Building 6600/Area III, authored by Dan Lucero, 05/04/95.
SP471436	Standard Operating Procedure for Research in the Deparment 6119 Chemcial Processes Laboratory Building 6600/Area III, authored by Charles Heath, 08/08/95.
SP472968	ES&H Standard Operating Procedure for Geochemical Research Department 6748 Water Chemistry Chemistry Laboratory in Building 823, authored by Karen Robinson, 07/18/95.
SP472799	ES&H Standard Operating Procedure for Geochemical Research in the Department 6119 Colloid- and Sorption-Chemistry Laboratory, Building 823, Room B45, authored by Karen Robinson, 01/24/95.

19.2. References

[1] Hrncir, D, and R. Knecht, 1996. "Waste Isolation Pilot Plant Waste Form and Disposal Room Data Qualification Peer Review Report (Final Draft)" Sandia WIPP Central Files

[2] Wilson, C., D. Porter, J. Gibbons, E. Oswald, G. Sjoblom, and F. Caporuscio, 1996. "Conceptual Models Peer Review Report (Final Draft)" Sandia WIPP Central Files

[3] Terry, B., 1983. "Specific Chemical Rate Constants for the Acid Dissolution of Oxides and Silicates", *Hydrometallurgy*, **11** (1983), 315-344.

[4] Vermilyea, D. A., 1969. "The dissolution of MgO and $Mg(OH)_2$ in aqueous solutions", *J. Electrochem. Soc.*, **116**, p1179-1183.