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Sandia National Laboratories
Waste Isolation Pilot Plant (WIPP)
Test Plan TP 00-07

**Experimental Study of WIPP MgO backfill
at Sandia National Laboratories Carlsbad Facility**

Task 1.1.09.01.01.02

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APPROVAL PAGE

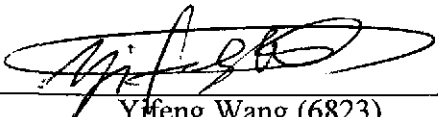
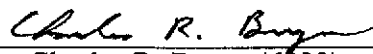
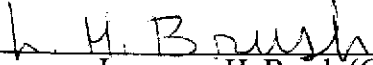
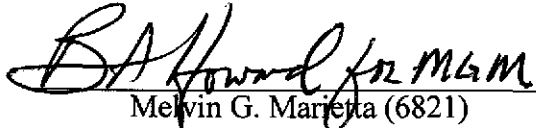

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1.0 DEFINITION OF ABBREVIATIONS AND ACRONYMS

ASTM	American Society for Testing and Materials
B.E.T.	Brunauer, Emmett, and Teller
CCA	Compliance Certification Application
CAO	DOE Carlsbad Office
DAS	Data acquisition system
DOE	Department of Energy
EBSD	Electron backscatter diffraction
GC-MS	Gas chromatography mass spectrometer
ICP	Inductively-Coupled Plasma Optical Emission Spectrometer
ISA	Isosaccharinic acid
LANL	Los Alamos National Laboratory
M&TE	Measuring and Test Equipment
NIST	National Institute of Standards and Technology
NBS	National Bureau of Standards
NP	Nuclear Waste Management Program Procedure
NWMP	Nuclear Waste Management Program
RWP	Radiological Work Permit
SEM	Scanning Electron Microscope
SNL	Sandia National Laboratories
SP	NWMP Activity/Project Specific Procedure
STTP	Source term test program
TDIC	Total dissolved inorganic carbon
TOP	Technical operating procedure
TP	Test Plan
WID	Westinghouse Waste Isolation Division
WIPP	Waste Isolation Pilot Plant
XRD	X-ray Diffraction

2.0 REVISION HISTORY

This is the first issue of this Test Plan (TP). Any future modifications to this Test Plan will be documented in a Test Plan Addendum, which will be issued to all project participants on controlled document distribution.

3.0 PURPOSE AND SCOPE

MgO backfill is being used in the Waste Isolation Pilot Plant (WIPP) to mitigate the effect of microbial CO₂ generation on actinide mobility in a post-closure repository environment (Bynum et al., 1998; Papenguth et al., 1998). MgO will sequester CO₂ and buffer brine pH at a moderately basic level, thus minimizing actinide solubilities in WIPP brines. In the past few years, a series of experiments have been conducted at Sandia National Laboratories (SNL) to verify the efficacy of MgO as a chemical control agent in the WIPP (See Test Plans TP 97-01 and TP 98-05). Some of these experiments are still in progress in the SNL-Albuquerque laboratories.

With the development of a laboratory facility at the SNL Carlsbad Operations Office, MgO experiments will be transferred to Carlsbad and new experiments will be initiated. This Test Plan provides guidance only for the MgO backfill studies to be conducted at SNL-Carlsbad Laboratory. The following tasks are planned:

- *Further study of the kinetics of Mg carbonate formation and conversion under WIPP-relevant conditions.* MgO reaction with CO₂ can produce a series of Mg carbonate minerals with magnesite as the most stable phase (Königsberger et al., 1999). Different Mg carbonate and hydroxycarbonate minerals can have different buffering capabilities for controlling CO₂ fugacity and brine pH. In the WIPP Compliance Certification Application (CCA), it is assumed that metastable Mg carbonate minerals will be ultimately converted to magnesite. The objective of this task is to gain a more mechanistic understanding of MgO carbonation and provide additional data to support the CCA assumption. This task will also support the Department of Energy (DOE) Carlsbad Area Office's (CAO) effort to simplify the existing MgO backfill system.
- *Evaluation of the possibility of chemical degradation of cellulose materials and isosaccharinic acid (ISA) production in MgO-buffered systems.* ISA has been produced by chemical degradation of cellulose materials in a cement-buffered system (Greenfield et al., 1992; Glaus et al, 1999). Because its ability to form stable metal complexes, ISA could have a significant impact on actinide mobility (Greenfield et al., 1992). The objective of this task is to evaluate the possibility of ISA production in a MgO backfill-buffered system and its possible impact on WIPP actinide source term calculations.
- *Investigation of MgO backfill interactions with dissolved actinides.* Little is known about the interactions of dissolved actinides with MgO backfill mineral phases. Theoretical considerations indicate that magnesite is a good metal sequester through coprecipitation (Wang and Xu, 2000). This task is to investigate the sorption and coprecipitation of actinides on Mg carbonate and brucite mineral surfaces. The data obtained from this task will support the interpretation of the experimental data obtained from the WIPP Source Term Test Program (STTP) at Los Alamos National Laboratory (LANL).

4.0 EXPERIMENTAL PROCESS DESCRIPTION

4.1 Overall Strategy and Process

Task 1. Further study of the kinetics of Mg carbonate formation and conversion

This activity is to determine (a) the controlling factors of solution chemistry on Mg carbonate formation, (b) the conversion rates of metastable Mg carbonate phases to magnesite under anticipated WIPP conditions, and (c) the capability of various carbonate minerals in controlling brine $p\text{CO}_2$ and pH. Since MgO hydration has been previously studied, the experiments planned here will be focused on the formation of Mg carbonate minerals from the reaction of brucite with CO_2 . Brucite will be purchased or synthesized using MgO backfill pellets. Brucite crystals will be crushed into fine-grained powder, and the specific surface area of brucite powders will be determined using B.E.T. (Brunauer, Emmett, and Teller) method.

In the first set of experiments, brucite powder will be added to various solutions and allowed to react at fixed $p\text{CO}_2$ in plastic bottles. Bottles will be continuously mixed to avoid any mass transport effect on reaction rates. The chemistry of the solutions will vary with respect to $p\text{CO}_2$, dissolved Mg concentration, sulfate concentration, and ionic strength. WIPP brines such as Brine A and ERDA-6 (Brush, 1990) will be used. During the experiments, an appropriate number of brine samples will be taken for the analysis of pH, Mg concentration, and total carbonate concentration, in order to monitor changes in solution chemistry due to carbonation. After sufficient time (perhaps weeks), the solids in each bottle will be filtered, and the resulting mineral phases will be identified using Scanning Electron Microscopy (SEM) and the associated Energy Dispersive System (EDS) and electron Backscatter Diffraction (EBSD). EBSD is able to obtain X-ray diffraction patterns on a scale of $<1 \mu\text{m}$ and therefore provides a better mineral identification capability than the X-Ray Diffraction (XRD) technique used in the previous MgO work (TP 96-01 and TP 98-05). XRD will be used to quantify the fractions of Mg carbonates formed in bulk samples, from which the rates of Mg carbonate formation rates will be calculated.

In the second set of experiments, the metastable Mg carbonate mineral phases will be incubated in various solutions at elevated temperatures from $25 - 90^\circ\text{C}$, and the fractions of a given metastable mineral converted after a specified incubation time period will be determined by XRD. The conversion rates can be then calculated. Using the Arrhenius equation (Lasaga, 1981), the conversion rates will be extrapolated to WIPP-relevant temperatures. The metastable Mg carbonate phases used in this set of experiments will be either purchased or synthesized.

In the third set of experiments, WIPP MgO pellets will be first reacted with CO_2 in WIPP brines for a certain period of time in plastic bottles. The bottle will be tightly capped and maintained at a room temperature for long enough time to let the solution to reach equilibrium with buffering minerals (brucite and Mg carbonate). Because of slow kinetics of magnesite formation at room temperatures, magnesite and brucite will be directly added to at least two tightly-capped plastic bottles, which will be first incubated at an elevated temperature and then gradually cool down to let the solution to equilibrate with magnesite and brucite. Finally, brine samples will be taken for the analysis of total dissolved inorganic carbon (TDIC) and pH. The

measured pH and TDIC will be compared with EQ3/6 (Version 7.2a) predictions (Wolery, 1992; Wolery and Daveler, 1992).

In the fourth set of experiments, the CO₂ uptake rate by MgO under humid conditions will be determined. A certain amount of MgO pellets will be placed a glass bottle. The bottle will be bubbled with CO₂ gas for a short period of time and then will be tightly closed. The CO₂ uptake rate will be determined by measuring CO₂ concentration changes in the bottle headspace. To maintain appropriate humidity in the bottle, a small beaker partially filled with synthetic WIPP brine will be placed together with MgO pellets. Care should be taken to avoid any spilling of brine on MgO pellets. To ensure data quality, control samples will be included that have the same configuration as other samples but without MgO pellets.

In the fifth set of experiments, MgO materials from new suppliers will be evaluated in terms of chemical reactivity according to the MgO acceptance criteria and the related procedure developed by Krumhansl et al. (1997). This set of experiments will be performed as needed whenever a new MgO material is acquired by the M&O, Currently Westinghouse Waste Isolation Division (WID).

Task 2. Evaluation of the possibility of chemical degradation of cellulose materials and isosaccharinic acid (ISA) production in MgO-buffered systems.

The cellulose materials used in this task will include laboratory filter paper, paper towels, tissue wipes (KimwipesTM), cotton, and wood. These materials comprise the typical cellulosic wastes of laboratory and transuranic process activities. The materials will be shredded and mixed into small pieces before being used in the following experiments. The electrolytes will range from dilute solutions to synthetic WIPP brines.

In the first set of experiments, equal grams of cellulose materials will be added to plastic bottles containing the solutions of interest. To compare the effects of two types of buffering systems, half of samples will be incubated with MgO, while another half will be incubated with CaO. Within each set of samples, the solution chemistry will vary from sample to sample in ionic strength, Mg concentration, or other parameters. Each sample equilibrated under MgO buffered conditions will have a corresponding sample in the CaO set. Under alkaline conditions, cellulose materials are degraded through a peeling-off reaction (Van Loon and Glaus, 1997), which strongly depends on the concentration of OH⁻, and so the solution pH of samples will be monitored during the course of the experiments. To avoid the interference of atmospheric CO₂, all sample bottles will be tightly closed. Small volumes of liquid samples will be periodically withdrawn from each bottle and analyzed for ISA and other organic compounds using Gas Chromatography Mass Spectrometer (GC-MS) or other methods. The extents of ISA production in both CaO and MgO-buffered systems will be determined.

The second set of experiments is to determine the solubility of ISA in WIPP brines. NaISA crystals will be synthesized following the procedure proposed by Van Loon et al. (1997). NaISA will be added to synthetic WIPP brines, and the concentration of dissolved ISA in brine will be

measured. The precipitates from the brine will be analyzed with XRD or EBSD to identify possible solubility-limiting mineral phases.

In the third set of experiments, the sorption of ISA on MgO backfill minerals will be studied with the batch technique. In the sorption experiments, the concentrations of ISA in the solution must be maintained below the solubility limit. These experiments will be focused on sorption by brucite and magnesite. Mineral powder will be prepared and its specific surface area will be determined by the N₂ B.E.T. method. Both mineral powder and ISA will be added to solution in plastic bottles and then mixed for a sufficient time (minutes to hours) for the sorption to reach chemical equilibrium. A small volume of solution will be withdrawn, filtered, and analyzed for ISA. The amount of ISA sorbed on mineral will be then calculated from the difference in concentration before and after sorption.

Task 3. Investigation of MgO backfill interactions with dissolved actinides

Since the SNL Carlsbad facility does not have the facilities to perform work involving radioactive materials, this task will start with non-radioactive divalent and trivalent heavy metals and use them as chemical analogs for actinides. The experiments will be focused on two Mg minerals – brucite and magnesite.

In the sorption experiments, a limited residence time reactor will be used, in order to minimize the effect of mineral dissolution and precipitation on pH control (Wersin et al., 1989). A saturated mineral slurry will be continuously pumped to a reaction vessel where it will be mixed with a mineral-free solution containing a heavy metal. The pCO₂ and pH in the vessel will be controlled by continuously bubbling CO₂-N₂ gas mixtures through the solution and adding HCl or NaHCO₃. A steady state is assumed to exist in the reaction vessel when the pH reading stabilizes. The outflow solution will be analyzed for the heavy metal, TDIC, and Mg²⁺. Based on the experimental data and literature data (e.g., Pokrovsky et al., 1999), a surface complexation model for heavy metal sorption on brucite and magnesite will be established.

In the coprecipitation experiments, the initial solution will be spiked with a heavy metal (e.g., Zn²⁺, Pb²⁺, Nd³⁺) to be coprecipitated. To precipitate brucite, MgO powder will be added to the spiked solution and mixed in a tightly closed plastic bottle. Similarly, to precipitate magnesite, brucite powder will be added to the solution and then continuously bubbled with CO₂ gas at an elevated temperature for a sufficiently long time to ensure all brucite has been converted to magnesite. In both cases, at the end of the experiments, the heavy metal concentrations in both precipitates and solution will be analyzed. The partition coefficients of heavy metal between mineral and solution will be then determined and compared with model predictions (Wang and Xu, 2000).

4.2 Sample Control

The sample control for the work under this Test Plan will follow WIPP Nuclear Waste Management Program (NWMP) Procedure NP 13-1. Each sample will be appropriately labeled. Sample preparation, utilization, and final disposition will be documented in scientific notebooks.

When samples are not in the possession of individual designated with their custody, they shall be stored in a secure area with associated documentation (Chain of Custody).

4.3 Data Quality Control

4.3.1 Measuring and Test Equipment (M&TE)

A calibration program will be implemented for the work described in this test plan in accordance with NP 12-1, "Control of Measuring and Test Equipment." This M&TE calibration program will meet the requirements in NWMP procedure NP 12-1 for: (1) receiving and testing M&TE; (2) technical operating procedures for M&TE; (3) the traceability of standards to nationally recognized standards such as those from the National Institute of Standards and Technology; (4) maintaining calibration records. In addition, NP 13-1 and SP 13-1 identify requirements and appropriate forms for documenting and tracking sample possession.

4.3.2 Data Acquisition Plan

Data collection procedures are specific to individual instruments. For details of the data acquisition for a particular instrument, see the Specific Procedures (SP) or Users Manual for that instrument. Any data acquired by a data acquisition system (DAS) will be attached directly to the Scientific Notebook or compiled in separate loose leaf binders with identifying labels to allow cross reference to the appropriate Scientific Notebook. If the instrument allows data to be recorded electronically, copies of the data disks will be submitted to the NWMP Records Center according to NWMP procedure NP 17-1 "Records." For instruments that do not have direct data printout, the instrument readings will be recorded directly into the scientific notebook. Current versions of the DAS software will be included in the SNL WIPP Baseline Software List, as appropriate.

Quality control of the Scientific Notebooks will be established by procedures described in NWMP procedure NP 20-2 "Scientific Notebooks." Methods for justification, evaluation, approval, and documentation of deviation from test standards and establishment of special prepared test procedures will be documented in the Scientific Notebooks. Procedures including use of replicates, spikes, split samples, control charts, blanks and reagent controls will be determined during the development of experimental techniques.

The numerical data will be transferred from data printouts and scientific notebooks to Microsoft Excel (Office 97 version or later) spreadsheets. Data transfer and reduction shall be performed in such a way to ensure that data transfer is accurate, that no information is lost in the transfer, and that the input is completely recoverable. Data transfer and reduction shall be controlled to permit independent reproducibility by another qualified individual. A copy of each spreadsheet will be taped into the scientific notebook, and a second person will compare the data recorded in the notebook and that on the spreadsheet to verify that no transcription errors have occurred during technical and/or QA review of the notebook. This verification will be documented in the notebook when it is "signed off" by the reviewer.

4.3.3 *Data Identification and Use*

All calculations performed as part of the activities of TP 00-07 will be documented in a scientific notebook. The notebook will be technically reviewed periodically by a second person, who will note concurrence by co-signing the examined material. If a discrepancy is found, that discrepancy and its resolution will be documented in the notebook. In addition, there will be periodic quality assurance reviews of the notebook to ensure that the requirements of NWMP procedure NP 20-2, "Scientific Notebooks" are addressed.

4.4 **Equipment**

A variety of measuring and analytical equipment will be used for the work described in this test plan. This equipment includes that listed below, as well as equipment not yet purchased. A complete equipment list, including serial numbers, will be maintained in the scientific notebook. Much of the instrumentation to be used for this project is newly purchased, and operating procedures have not yet been developed or written. Scientific notebooks will be used to record all laboratory work activities.

4.4.1 *Weighing Equipment*

Several balances are present in the facility and may be used for this project. These include a Mettler AT-261 five-decimal place electronic balance, an ANC three-decimal place balance, and top loading balances and scales with maximum ranges of 2 to 30 kilograms. Balance calibration checks will be performed routinely using the following NBS-traceable weight sets, which, in turn, are calibrated by the SNL Calibration Laboratory every 3 years:

- Troemner Calibration weight set, ASTM Class 1, Serial number 22803, 1 mg – 100 g, calibration expires 12/16/02.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42795, 100 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42797, 100 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42799, 100 g, calibration expires 11/19/01.
- *Troemner Calibration weight*, NBS-Class 1, Serial number 42800, 100 g, calibration expires 11/19/01.
- *Troemner Calibration weight*, ASTM-Class 1, Serial number 47824, 200 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, ASTM-Class 1, Serial number 55335, 1000 g, calibration expires 11/19/02.
- *Troemner Calibration weight*, ASTM-Class 2, Serial number I-12, 10 kg, calibration expires 12/17/02.

Balance accuracy and precision will be checked daily or prior to use (whichever is less frequent), using the calibration weight sets listed above. Calibration checks will be recorded in the scientific notebook.

4.4.2 Liquid Measuring Equipment

Standard Laboratory Class A glassware (pipettes, volumetric flasks, etc.) will be used at all times. In addition, several adjustable Eppendorf pipettes are available for use in the laboratory. The calibration of pipettes will be checked routinely against a calibrated balance, and will be recorded in the scientific notebook.

4.4.3 Other Analytical Equipment

- *pH Meters and Autotitrators* – solution pH may be measured using pH meters and/or autotitrators. A *Mettler Model MA235 pH/Ion Analyzer* and a *Mettler Model DL25 Autotitrator* will be used for this purpose. The range for all pH meters is 0.00 to 14.00. Electrodes will be calibrated before each use or daily (whichever is less frequent) with pH 4, 7, and 10 buffers manufactured by Fisher Scientific with unique lot numbers and expiration dates; traceable to the National Institute of Standards and Technology (NIST). The accuracy of the buffers is ± 0.01 pH units; buffer values will be adjusted for laboratory temperatures as per buffer instruction sheets if necessary. Calibration checks will be recorded in the scientific notebook. Measuring pH in concentrated brines is difficult, and a procedure will be developed to calibrate pH meters.
- *Equipment for Chemical Analysis* – Three instruments may be used for chemical analyses. The first is a Perkin Elmer Optima 3300 DV Inductively-Coupled Plasma Optical Emission Spectrometer (ICP); the second is a Cary 300 UV-Visible Spectrophotometer; and the third, is a UIC, Inc. Carbon Analyzer, consisting of an acidification module, a furnace module, and a CO₂ coulometer. These instruments will be user-calibrated each time they are used and documented in the scientific notebook.
- *Equipment for Mineralogical, and Textural Characterization* – The mineralogy and texture may be characterized using either an Olympus BX60 Polarizing Microscope or a JEOL JSM 5900LV scanning electron microscope (SEM). Calibration standards will be used to verify instrument magnification when these instruments are used. Bulk sample mineralogy will be determined using a Bruker AXS D-8 Advance X-Ray Diffractometer (XRD). A mineral standard will be run periodically to verify diffraction line positions. Calibration results will be documented in the scientific notebook.

NMWP Activity/Project Specific Procedures (SPs) will be written for these instruments as necessary. Until that time, detailed procedure descriptions will be documented in laboratory notebooks.

5.0 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 NWMP procedure NP 2-1, "Qualification and Training." Evidence of training to assigned NPs, SPs, TOPs, TP 00-07, ES&H procedures, and any other required training will be documented through Form NP 2-1-1 *Qualification and Training*. Annual Refresher QA training will ensure on-site personnel are trained to the NWMP QA Program. Specifically, the following Nuclear Waste

Management Program Procedures (NPs) and Activity/Project Specific Procedures (SPs) are applicable:

- SOP-C001 – “Standard Operating Procedure for Activities in the SNL/Carlsbad Laboratory Facility.”
- SP 13-1 – “Chain of Custody”
- NP 6-1 – “Document Review Process”
- NP 13-1 – “Sample Control”
- NP 12-1 – “Control Of Measuring And Test Equipment”
- NP 20-2 – “Scientific Notebooks”
- NP 2-1 – “Qualification and Training”
- NP 17-1 – “Records”

In addition, SPs will be written for use of the ICP, SEM, XRD, Carbon analyzer, UV-Vis spectrophotometer, and assorted balances and scales used in the laboratory. Sample preparation procedures, which may vary from sample to sample as work scope evolves, will be detailed in Scientific Notebooks, in accordance with NWMP procedure NP 20-2.

6.0 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 ES&H standard operating procedures. SP-C001 describes the non-radiological hazards associated with these experiments and describes the procedures to deal with those hazards, including all the training requirements for personnel involved in conducting the experiments. In addition, a Radiological Work Permit (RWP) will be written for procedures involving use of the X-Ray Diffractometer. Additional SPs and RWPs may be mandated by SNL ES&H requirements and their issuance will not require revision of this Test Plan.

7.0 Permitting/Licensing

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

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