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Sandia National Laboratories  
Waste Isolation Pilot Plant (WIPP)  
Test Plan TP 12-01

**Experimental Investigation of Absence or Presence of Colloids  
of Magnesium Chloride Hydroxide Hydrate (Phase 5) in the WIPP Generic Weep  
Brine (GWB) Under the WIPP Relevant Conditions at  
Sandia National Laboratories Carlsbad Facility**

**Task 1.4.2.2**

Rev. 0

Effective Date: 12/15/11

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## 1.0 DEFINITION OF ABBREVIATIONS AND ACRONYMS

ASTM	American Society for Testing and Materials
CBFO	US DOE Carlsbad Field Office
CO <sub>2</sub>	Carbon dioxide
CRA	Compliance Re-Certification Application
DAS	Data acquisition system
DOE	Department of Energy
EBSD	Electron backscatter diffraction
EDS	Energy dispersive system
ERDA-6	Energy Research and Development Administration well 6
GC-MS	Gas chromatography mass spectrometer
GWB	Generic Weep Brine (synthetic Salado Formation brine)
IC	Ion chromatograph
ICP-AES	Inductively-coupled plasma atomic emission spectrometer
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 Procedure
PA	Performance Assessment
SEM	Scanning electron microscope
SNL	Sandia National Laboratories
SP	Activity/Project Specific Procedure
TP	Test Plan
TRU	Transuranic
WIPP	Waste Isolation Pilot Plant
XRD	X-ray diffraction

## 2.0 REVISION HISTORY

This is the original version of this Test Plan (TP).

## 3.0 PURPOSE AND SCOPE

The purpose of this test plan is to investigate the potential formation of colloids of  $\text{Mg}_3\text{Cl}(\text{OH})_5 \cdot 4\text{H}_2\text{O}$ , termed as phase 5 in cement literature, under the WIPP relevant conditions, and, if formed, the capacity of such colloids to sorb Th(IV) as pseudocolloids in the WIPP source term. The investigation of presence or absence of  $\text{Mg}_3\text{Cl}(\text{OH})_5 \cdot 4\text{H}_2\text{O}$  colloids under the WIPP relevant conditions is necessary, as the presence of such colloids could have an effect on the actinide source term. In a recent study by Altmaier et al. (2004), these authors mentioned that they observed the formation of colloids of magnesium chloride hydroxide hydrate,  $\text{Mg}_2\text{Cl}(\text{OH})_3 \cdot 4\text{H}_2\text{O}$ , which is termed as phase 3 in cement literature, in their experiments in 4.5 M  $\text{MgCl}_2$ . When dissolved Th(IV) was sorbed onto colloids of phase 3, pseudocolloids could form, and such pseudocolloids could facilitate the transport of actinides. Furthermore, in the U.S. EPA Technical Support Document (TSD) related to CRA-2009, EPA stated that “although the mineral-fragment colloids reported in the recent literature are not expected to be stable in WIPP brines, examination of the data used to develop the colloidal actinide source term model has shown that possible formation of mineral fragment colloids by MgO and its hydration and carbonation products under WIPP-relevant conditions has not been evaluated.” (U.S. EPA, 2010).

In the WIPP, there are two brines that are important to Performance Assessment (PA), i.e., ERDA-6 (Energy Research and Development Administration Well 6) from the Castile Formation, a Na-Cl dominated brine, and GWB (Generic Weep Brine) from the Salado Formation, a Na-Mg-Cl dominated brine (Xiong, 2008). The compositions of these brines are significantly different from 4.5 M  $\text{MgCl}_2$ . Therefore, it is highly uncertain that these kinds of pseudocolloids could form under WIPP relevant conditions, based on the reasoning detailed in Xiong et al. (2010a). The main reasons listed by Xiong et al. (2010a) include the following. First, the WIPP colloidal actinide source term program supporting the WIPP CCA did not identify intrinsic or mineral-fragment colloids. Second, Altmaier et al. (2004) concluded that the high [Th(IV)] concentrations from pseudocolloids observed in their laboratory experiments have not to be expected in real systems. Third, pseudocolloids in the experiments of Altmaier et al. (2004) were produced by phase 3 in 2.5 and 4.5 M  $\text{MgCl}_2$  solutions. Fourth, the phase 3 used in experiments of Altmaier et al. (2004) was in fine-grained powder.

In contrast to the experiments of Altmaier et al. (2004) in concentrated  $\text{MgCl}_2$  solutions with phase 3, under the WIPP relevant conditions,  $\text{Mg}_3\text{Cl}(\text{OH})_5 \cdot 4\text{H}_2\text{O}$ , instead of phase 3, will form, in the GWB, as it has been experimentally observed (Xiong and Lord, 2008). Additionally, the experiments of Altmaier et al. (2004) were from undersaturation with respect to phase 3, and the phase 3 used by Altmaier et al. (2004) was in fine-grained powder form. Therefore, when phase 3 was in contact with their solutions, dissolution and re-precipitation probably occurred. During this dissolution and re-precipitation process, Th(IV) may have sorbed onto the phase 3 colloids, forming pseudocolloids. Under the expected WIPP conditions used in the Sandia National Laboratories (SNL) study of MgO, the reaction path is that MgO partially converts to phase 5 in GWB (Xiong and Lord, 2008), and the phase 5 produced is a well cemented form of magnesium chloride hydroxide hydrate. Neither phase 3, nor phase 5, will be formed in ERDA-6 (Xiong and Lord, 2008).

The above brief review indicates that there is a very low probability of formation of colloids from phase 5 under WIPP relevant conditions. However, whether phase 5 could produce or will not produce colloids under WIPP relevant conditions has not been experimentally demonstrated. Additionally, as mentioned before, existing experimental work indicates that pseudocolloids could form from phase 3, and we are not aware of any experiments that have produced pseudocolloids from phase 5. Consequently, if the reaction product of MgO with GWB is stabilized as phase 5 instead of phase 3, the possibility of formation of pseudocolloids would be further reduced. Therefore, the relative stability of phase 3 and phase 5 could be significant. It has been known that formation of phase 3 and phase 5 depends on activities of  $Mg^{2+}$ , hydrogen ion ( $H^+$ ) and water (Xiong et al., 2010b). In the work of Xiong et al. (2010b), the role of  $Na^+$  in stabilization of phase 5 has not been explored. As GWB is a multi-component brine with high concentrations of  $Na^+$ , it could play a role in stabilization of phase 5 in addition to activities of  $Mg^{2+}$ ,  $Cl^-$ , hydrogen ion ( $H^+$ ) and water.

Based on the issues identified above, the objective of this study is to evaluate the existing experiments involving MgO under WIPP relevant conditions (initially executed under TP 06-03, Deng, Nemer, and Xiong, 2006) to demonstrate whether colloids of phase 5, are, or are not, present in those experiments. As a secondary objective, we will assess the role of  $Na^+$  and  $Cl^-$  in stabilization of phase 5 in additional experiments. The results will be used to constrain any revision of the colloid source-term model, and could potentially directly support re-certification and the PA. The following tasks focused on experiments in GWB are planned because formation of phase 5 has been observed in GWB. As described above, neither phase 3, nor phase 5, will form in ERDA-6, and therefore no tasks are planned for experiments on MgO in ERDA-6 to determine the absence or presence of colloids of phase 5.

- *Experimental determination of absence or presence of phase 5 colloids in long-term hydration experiments with MgO in GWB.* In long-term hydration experiments with Martin Marietta MgO in GWB and simplified GWB (1 M  $MgCl_2$  + 3.6 M  $NaCl$ ), phase 5 has been observed (Deng et al., 2009). Therefore, long-term MgO hydration experiments in GWB and simplified GWB are selected for demonstration of absence or presence of colloids of phase 5.
- *Experimental determination of absence or presence of phase 5 colloids in carbonation experiments with MgO in GWB.* Carbonation experiments with Martin Marietta MgO in GWB and simplified GWB have  $CO_2$  partial pressures ranging from  $10^{-3.46}$  to  $10^{-3.00}$  atm (Deng, Nemer, and Xiong, 2006). In the similar carbonation experiments with Premier MgO, phase 5 has been also observed (Xiong and Lord, 2008). Therefore, MgO carbonation experiments in GWB and simplified GWB are also selected for determination of absence or presence of colloids of phase 5.
- *Experimental assessments of the role of  $Na^+$  and  $Cl^-$  in stabilization of phase 5.* GWB is a multi-component brine with high concentrations of  $Na^+$ . Therefore, the role of  $Na^+$  in stabilization of phase 5 will be experimentally explored.

## 4.0 EXPERIMENTAL PROCESS DESCRIPTION

### 4.1 Overall Strategy and Process

The overall strategy is to determine magnesium concentrations in experiments in which phase 5 forms as a function of pore sizes of Millipore® or other brand ultra centrifugal filters, ranging from 220 nm to 10 nm. If there is no significant difference in terms of magnesium concentrations between two

widely different filter sizes, this indicates the absence of colloids of phase 5. This is because phase 5 has a chemical formula of  $Mg_3Cl(OH)_5 \cdot 4H_2O$ . Therefore, should colloidal phase 5 be present in solution, the aliquot filtered at 220 nm would have higher magnesium concentrations in comparison with that filtered at much smaller pore sizes.

#### **4.1.1 Experimental Procedure**

##### ***Task 1. Experimental Determination of Absence or Presence of Colloids of Phase 5 in Long-term MgO Hydration Experiments in GWB and Simplified GWB.***

Long-term MgO hydration experiments in GWB and simplified GWB are selected for determination of absence or presence of colloids of phase 5. For each experiment, a solution sample will be withdrawn and filtered with 450 nm pore size filter to remove solids. Then, this solution sample will be weighed, and split into two equal samples. One sample will be kept as a back-up, and the other sample will be further split into four aliquots. These four aliquots will be subject to four pore sizes filtration corresponding to 10,000, 30,000, 50,000, and 100,000 rotation velocities, respectively. After filtration, these samples will be acidified to 5%  $HNO_3$ . Then they will be further diluted for analyses of magnesium concentrations. Magnesium concentrations will be determined for diluted samples by using the Perkin Elmer Optima DV 3300 inductively coupled plasma atomic emission spectroscopy (ICP-AES).

##### ***Task 2. Experimental Determination of Absence or Presence of Colloids of Phase 5 in MgO Carbonation Experiments in GWB and Simplified GWB.***

MgO carbonation experiments in GWB and simplified GWB are selected for determination of absence or presence of colloids of phase 5. In carbonation experiments, in addition to phase 5, hydromagnesite (5424), which has a chemical formula of  $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$  (Xiong and Lord, 2008), also forms. Similar to Task 1, for each experiment, a solution sample will be withdrawn and filtered with 450 nm pore size filter to remove solids. Then, this solution sample will be weighed, and split into two equal samples. One sample will be kept as a back-up, and the other sample will be further split into four aliquots. These four aliquots will be subject to four pore sizes filtration corresponding to 10,000, 30,000, 50,000, and 100,000 rotation velocities, respectively. After filtration, these samples will be acidified to 5%  $HNO_3$ . Then they will be further diluted for analyses of magnesium concentrations. Magnesium concentrations will be determined for diluted samples by using the ICP-AES.

If the experimental results indicate the presence of colloids in carbonation experiments in GWB and simplified GWB, the following reasoning and actions will be applied in consideration of the results from long-term hydration experiments. If the results from long-term hydration experiments indicate the absence of colloids of phase 5, the positive results from carbonation experiments would suggest that the colloidal contributions must be from colloids of hydromagnesite (5424) only. Pseudocolloids of hydromagnesite (5424) have not been mentioned in literature. If the results from long-term hydration experiments indicate the presence of colloids of phase 5, the positive results from carbonation experiments would suggest that the colloidal contributions could be from colloidal phase 5 alone, or a mixture of colloids of phase 5 and hydromagnesite (5424). Under such a circumstance, the contribution from colloids of hydromagnesite (5424) will be determined by analyses of total carbon concentrations via a  $CO_2$  coulometer. Samples for  $CO_2$  coulometer analyses will be prepared separately in a way similar to that for ICP-AES analyses, but without acidification. In addition, should

colloids of hydromagnesite (5424) in carbonation experiments in GWB and simplified GWB be positively identified, experimental activities would also be extended to carbonation experiments in ERDA-6, as hydromagnesite (5424) also forms in those experiments.

### ***Task 3. Experimental Assessments of the Role of Na<sup>+</sup> in Stabilization of Phase 5.***

As mentioned before, the formation of phase 3 or phase 5 depends on activities of Mg<sup>2+</sup>, Cl<sup>-</sup>, hydrogen ion (H<sup>+</sup>) and water, and Na<sup>+</sup> could play a role in stabilization of phase 5, as GWB has high concentrations of Na<sup>+</sup>. In GWB, boron, Ca<sup>2+</sup>, K<sup>+</sup>, SO<sub>4</sub><sup>2-</sup> and Br<sup>-</sup> are also present, but at much lower concentrations (Xiong and Lord, 2008). Therefore, they are not expected to play important roles in stabilization of phase 5. In simplified GWB, the above ions at low concentrations are not present. Consequently, only the role of Na<sup>+</sup> ion in stabilization of phase 5 needs to be experimentally explored. In this set of experiments, the following experiments are planned:

1.0 M MgCl<sub>2</sub> + Martin Marietta MgO  
1.0 M MgCl<sub>2</sub> + 1.0 M NaCl + Martin Marietta MgO  
1.0 M MgCl<sub>2</sub> + 2.0 M NaCl + Martin Marietta MgO  
1.0 M MgCl<sub>2</sub> + 3.0 M NaCl + Martin Marietta MgO  
1.65 M MgCl<sub>2</sub> + Martin Marietta MgO  
1.65 M MgCl<sub>2</sub> + 1.0 M NaCl + Martin Marietta MgO  
1.65 M MgCl<sub>2</sub> + 2.0 M NaCl + Martin Marietta MgO  
1.65 M MgCl<sub>2</sub> + 3.0 M NaCl + Martin Marietta MgO

In the above experiments, the subset of experiments with 1.0 M MgCl<sub>2</sub> will contain the same magnesium concentrations as that of GWB. The magnesium concentration in the subset with 1.65 MgCl<sub>2</sub> is close to that of the invariant composition of phase 3 and phase 5 predicted by Xiong et al. (2010b).

Solution samples from the above experiments will be analyzed for hydrogen ion concentrations by using a pH electrode via applying correction factors determined by Xiong et al. (2010b), for chloride concentrations by using IC or a chloride electrode, and for magnesium and sodium concentrations by using the ICP-AES. Solid samples will be analyzed for phase identification by using the XRD.

#### ***4.1.2 Supporting Solutions***

All supporting solutions will be prepared from reagent grade chemicals from Fisher Scientific or its associated vendors.

## **4.2 Sample Control**

The sample control for the work under this Test Plan will follow WIPP 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 individuals designated as responsible for 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 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. The spread sheet and other computer based data handling will follow NP 9-1.

#### ***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 Records Center according to procedure NP 17-1 "Records." If possible, data files may be transferred to ZIP disks or CD ROM for submittal to the records center. 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 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.

#### ***4.3.3 Data Identification and Use***

All calculations performed as part of the activities of TP 12-01 will be documented in a scientific notebook. The notebook will be technically and QA reviewed periodically to ensure that the requirements of procedure NP 20-2, "Scientific Notebooks", are addressed. If a discrepancy is found, that discrepancy and its resolution will be documented during the review on a Document Review and Comment (DRC) Form NP 6-1-1.

## 4.4 Equipment

A variety of measuring and analytical equipment will be used for the work described in this test plan. A complete equipment list, including serial numbers, will be maintained in the scientific notebook. 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. Balance calibration checks will be performed daily or prior to usage, using the following NBS-traceable weight sets, which, in turn, are calibrated by the SNL Calibration Laboratory. Calibration checks will be recorded in Balance Calibration Records.

### 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. The accuracy of pipettes will be within  $\pm 1\%$ .

### 4.4.3 Other Analytical Equipment

- *Ovens and Furnace*- Six Precision Telco Lab ovens are being used to hold samples at elevated temperatures. Temperature is monitored, maintained, and recorded on a daily schedule.
- *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 a minimum of two pH buffers manufactured by chemical companies with unique lot numbers and expiration dates; traceable to the National Institute of Standards and Technology (NIST). Calibration checks will be recorded in the scientific notebook.
- *Equipment for Chemical Analysis* – Four instruments may be used for chemical analyses. The first is a Perkin Elmer Optima 3300 DV Inductively-Coupled Plasma Atomic Emission Spectrometer (ICP-AES); 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 CO<sub>2</sub> coulometer, and the fourth is a DIONEX Ion Chromatograph (IC) 3000. These instruments will be user-calibrated per instrument requirement.
- *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). 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.

The usage of these instruments will follow Activity/Project Specific Procedures (SPs).

## 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 procedure NP 2-1, "Qualification and Training." Specifically, the following Nuclear Waste Management Procedures (NPs) and Activity/Project Specific Procedures (SPs) are applicable:

- SOP CPG-CHEM-TWD-2011-001 – "ES&H Standard Operating Procedure (ES&H SOP) for Activities in the Sandia National Laboratories/Carlsbad Program Group Laboratory, Building NPHB (U)."
- TWD 001 – "Safety Management of Bruker Analytical X-ray Systems D8 Advance X-ray Diffractometer"
- Standard Operating Procedure (SOP) for Oxygen Deficiency Hazard Alarm Response for Carlsbad (Building NPHB)
- NP 2-1 – "Qualification and Training"
- NP 6-1 – "Document Review Process"
- NP 12-1 – "Control of Measuring and Test Equipment"
- NP 13-1 – "Control of Samples and Standards"
- SP 13-1 – "Chain of Custody"
- NP 17-1 – "Records"
- NP 20-2 – "Scientific Notebooks"

## 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. ES&H SOP 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. Additional SOPs 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.

## 8.0 References

Altmaier, M., Neck, V., and Fanghänel, Th., 2004. Solubility and colloid formation of Th(IV) in concentrated NaCl and MgCl<sub>2</sub> solution. *Radiochimica Acta*, **92**, 537–543.

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