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SANDIA NATIONAL LABORATORIES
WASTE ISOLATION PILOT PLANT

AP-172
Revision 0

Analysis Plan for Experimental Investigation of Absence or Presence of Mineral Fragment Colloids in the GWB and ERDA-6 Brines Under the WIPP Relevant Conditions at Sandia National Laboratories Carlsbad Facility

Task 4.4.2.2.1

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1 Introduction and Objectives

1.1 Introduction

The Waste Isolation Pilot Plant (WIPP) is a deep geologic repository developed by the U.S. Department of Energy (DOE) for the permanent disposal of transuranic (TRU) radioactive waste. The repository is located within the bedded salts of the Permian Salado Formation, consisting mainly of rock salt with thin interbedded layers of anhydrite and polyhalite. Containment of TRU waste at the WIPP is regulated by the U.S. Environmental Protection Agency (EPA) according to requirements set forth in Title 40 of the Code of Federal Regulations (CFR), Part 191. WIPP PA calculations estimate the probability and consequence of actinide releases from the repository to the accessible environment for a regulatory period of 10,000 years after facility closure. The WIPP PA models indicate that actinides are released by instantaneous dissolution and colloidal mobilization. According to the 2009 Compliance Recertification Application Performance Assessment Baseline Calculation (CRA-2009 PABC) the colloidal actinides cover a significant portion of mobile actinides (Fig.1) except U(VI). Actinides with oxidation state of +4 are apparently absorbed on mineral fragments colloidal particles (Fig.2). The residual colloid number population examinations showed that mineral fragment type colloidal particles are kinetically destabilized by brines similar to the WIPP brine, and the mineral fragment estimated from hematite, goethite, and bentonite after about 13 aging days were in ranges of 10^{-7} – 10^{-10} moles/L dispersion. Accounting for the possible sorption of the dissolved actinides onto indigenous mineral fragments within the Culebra in an intrusion scenario, the WIPP PA parameter CONCMIN (concentration of mineral fragments) has been conservatively set to the geometric mean value of the estimated mineral fragment concentrations multiplied by a factor of two. Recently, Altmaier et al. (2004) observed the formation of $Mg_2Cl(OH)_3{\cdot}4H_2O$ (phase 3) in 4.5M $MgCl_2$ solutions. Xiong et al. (2008a) observed the formation of $Mg_3Cl(OH)_5{\cdot}4H_2O$ (phase 5) in industrial grade MgO added GWB (Generic Weep Brine). WIPP PA parameter CONCMIN has not taken the formation of phase 3 or phase 5 in the WIPP-relevant brines into account, because the WIPP colloidal actinide source term program did not experimentally identify phase 3 or phase 5 mineral fragment colloids. As a result, this analysis plan is to investigate the potential formation of phase 5 colloids under the WIPP relevant conditions.

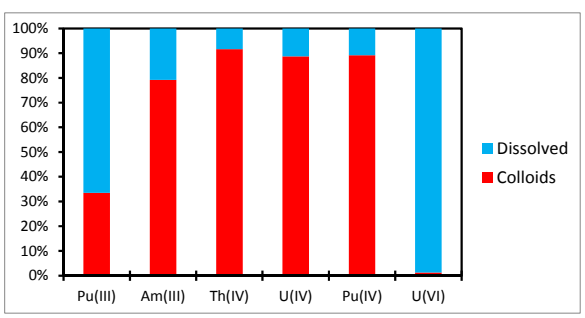

Fig.1. 100% stacked column plot showing proportions of the colloidal actinides and the dissolved actinides in the CRA-2009 PABC.

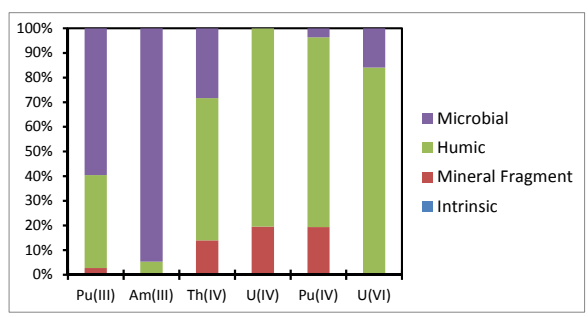

Fig.2. 100% stacked column plot showing proportions of microbial, humic, mineral fragment, and intrinsic colloidal actinides in the CRA-2009 PABC.

This analysis plan guides the experimental assurance of formations of mineral fragment colloids in the WIPP relevant brines. This analysis plan is considered under the Test Plan, TP12-01

Revision 1 (Xiong et al., 2014a): "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." Outputs of this analysis plan would make WIPP PA parameters updates. Therefore, this analysis is a Compliance Decision analysis per NP 9-1.

1.2 Objectives

It is uncertain that the formation of phase 5 colloids and their actinide pseudocolloids could be viable under the WIPP relevant conditions. Therefore, Test Plan TP12-01 Revision 1 (Xiong et al., 2014a) was written in response to the question of whether mineral fragments (phase 5 mineral fragments) will form in WIPP relevant conditions. The purpose of TP 12-01 Revision 1 is to determine experimentally if phase 5 mineral fragments are absent or present in WIPP relevant conditions.

2 Approach

According to Brush et al. (2009a), the WIPP repository is expected to define the long-term chemical conditions: (1) entrance of fluids from the Salado Formation and the Castile Formation; and (2) instantaneous, reversible equilibria among these brines, major Salado minerals, and MgO hydration and carbonation products.

Based on the issues identified in Section 1.2 and the long-term chemical conditions in the WIPP repository, the first objective of this study is to evaluate the existing experiments involving MgO under WIPP relevant conditions (initially executed under TP 06-03, Deng et al., 2006) to demonstrate whether colloids of phase 5, are, or are not, present in those experiments. The second objective is to assess the role of Na^+ and Cl^- in stabilization of phase 5 in additional experiments. In addition, metallic lead is used in the waste stream as radiation shielding material. When metallic lead is corroded under the WIPP conditions, lead corrosion products such as lead ion species can be generated and could form lead-containing minerals. The lead-containing mineral fragments could be produced from some of these minerals. The likely lead-containing minerals that could be formed are lead oxalate and lead carbonate. Therefore the third objective is to assess the potential formation of mineral fragments related to these two minerals. Three objectives will be accomplished with the following four tasks:

- *Task 1 - 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.

- *Task 2 - 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 et al., 2006). In the similar carbonation experiments with Premier MgO, phase 5

has been also observed (Xiong et al., 2008a). Therefore, MgO carbonation experiments in GWB and simplified GWB are also selected for determination of absence or presence of colloids of phase 5.

- *Task 3 - 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.

- *Task 4 - Experimental determination of potential formation of lead-containing mineral fragments in ERDA-6 and GWB.* When lead is corroded in the WIPP brines, ERDA-6 and GWB, lead oxalate, lead carbonate, or both, are expected to form under the WIPP conditions. The WIPP brines have high oxalate concentrations up to 1.7×10^{-2} M (Brush et al., 2009b; Brush et al., 2013), and microbial consumption of cellulosic, plastic and rubber (CPR) material produce CO_2 gas. Consequently, lead oxalate and lead carbonate are predicted to be the stable phases in the high ionic strength brines (Xiong, 2013, 2014b).

3 Software List

The software to be used for this analysis is EQ3/6 Version 8.0a with execution platforms with Microsoft Windows 95, 98, 2000, NT4, XP, Vista, and Window 7.

4 Tasks

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

Experiments under Tasks 1 of TP 12-01 were already initiated under Test Plan TP 06-03. Samples for hydration experiments were prepared utilizing three types of brines along with three different sizes of Martin Marietta MgO (Scientific Notebook WIPP-MM-MgO-5 pgs. 59-73). The used brines were Simplified GWB, GWB, and ERDA-6. Three different sizes of Martin Marietta MgO were Large (L) 1.0-2.0 mm, Small (S) less than 75 µm, and a Mix of the two (M) (Milestone Report, Deng et al., 2009). The sample names reflect the experimental set-up, for example; in order from left to right the sample name reflects the brine type, the MgO-to-brine ratio and the MgO particle size. The prefix GW (GWB), ER (ERDA-6 brine) and MgCl (Simplified GWB) represent the brine type. The next number, either 3 or 20, represents the MgO-to-brine ratio, 3 g/11 mL (3) or 3.1 g/77 mL (20). The last letter (L, S or M) represents the MgO size: Large, Small or Mixed. Finally, the last number is the individual sample identifier within a specific group.

For Task 1 of TP 12-01, only the 3.1 g/77 mL samples in Simplified GWB and GWB brines will be analyzed because the 3 g/11 mL samples and the samples with small size MgO particles do not contain enough solution for testing. ERDA-6 samples will not be used because phase 5 is unlikely to form in ERDA-6 brine. Long-term MgO hydration experiments samples in GWB and simplified GWB will be analyzed for determination of absence or presence of colloids of phase 5. Samples for Task 1 of TP 12-01 will be GW20M#, GW20L#, MgCl2-20M#, and

MgCl2-20L#, where # represents the sample number. Sample processing and analyses methods are described in the first milestone report by Xiong et al. (2014c).
The responsible individuals for analysis of the results from Task 1 of TP 12-01 are Yongliang Xiong and Leslie Kirkes. The estimated completion date for analysis of the results from Task 1 of TP 12-01 is 12/31/2016.

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

Under TP 06-03, samples for carbonation experiments were set up in a similar way to that for hydration experiments (Scientific Notebook WIPP-MM-MgO-12 pgs. 21-50). Three types of brines (Simplified GWB, GWB, and ERDA-6) were utilized. Three different sizes of Martin Marietta MgO (Large, Small, and Mixed) were utilitzed. The MgO and brine descriptions are the same as seen in Section 3.1. Additional experiment samples were also prepared with normal MgO as well as Hydrated MgO (MgOH). In these experiments, the humidity, temperature and CO_2 concentration were carefully controlled through the use of a Multi-flow Gas Control System (MFGCS) designed and constructed by Sandia National Laboratories. Two different CO_2 concentrations were utilized such as 350 and 3500 ppm. The prefix in the sample set-ups 1:3 or 1:20 represents the MgO-to-brine ratio, 3 g/11 mL (1:3) or 2 g/50 mL (1:20). The experiments with 3 g/11 mL (1:3) will not be selected for testing because of insufficient solution volume. Samples prepared with ERDA-6 will not be selected for testing because phase 5 is unlikely to form. In carbonation experiments, in addition to phase 5, hydromagnesite (5424), which has a chemical formula of $Mg_5(CO_3)_4(OH)_2{\bullet}4H_2O$ (Xiong et al., 2008a), also forms. In the first milestone report (Xiong et al., 2014c), samples prepared under the CO_2 concentration of 3500 ppm were analyzed and reported. These samples are 1:20 GWBB #, 1:20 GWBD #, and 1:20 SGWBB # for the non-hydrated MgO carbonation experiments, and 1:20 GWBMgOHA #, and 1:20 SGWBMgOHB # for the hydrated MgO carbonation experiments. Samples prepared under the CO_2 concentration of 350 ppm will be analyzed in the same way as the 3500 ppm samples. Similar to Task 1, sample processing and analyses methods are described in the first milestone report by Xiong et al. (2014c).

The responsible individuals for analysis of the results from Task 1 of TP 12-01 are Yongliang Xiong and Leslie Kirkes. The estimated completion date of for analysis of the results from Task 2 of TP 12-01 is 12/31/2016.

4.3 Task 3 - Experimental Assessments of the Role of Na^+ in Stabilization of Phase 5.

The formation of phase 3 or phase 5 depends on activities of Mg^{2+}, Cl^-, H^+ and water. Activity of Na^+ could play a role in stabilization of phase 5, as GWB has high concentrations of Na^+. GWB contains low concentrations of B^{3-}, Ca^{2+}, K^+, SO_4^{2-} and Br^- ions (Xiong et al., 2008a). These ions are expected not to play important roles in stabilization of phase 5. Simplified GWB excludes the above ions. Consequently, Simplified GWB enables the exploration of the role of Na^+ ion in the stabilization of phase 5. For Task 3 of TP 12-01, the following experiments have been started:

1.0 M $MgCl_2$ + Martin Marietta MgO
1.0 M $MgCl_2$ + 1.0 M NaCl + Martin Marietta MgO
1.0 M $MgCl_2$ + 2.0 M NaCl + Martin Marietta MgO
1.0 M $MgCl_2$ + 3.0 M NaCl + Martin Marietta MgO
1.65 M $MgCl_2$ + Martin Marietta MgO
1.65 M $MgCl_2$ + 1.0 M NaCl + Martin Marietta MgO
1.65 M $MgCl_2$ + 2.0 M NaCl + Martin Marietta MgO
1.65 M $MgCl_2$ + 3.0 M NaCl + Martin Marietta MgO

Numerous Martin Marietta MgO sieve sizes were utilized including mesh numbers: 10 (2.0 mm), 30 (600 μm), 50 (300 μm), 100 (150 μm), and 200 (75 μm). All of the samples were prepared in duplicate. The samples were named to reflect the experimental set-ups and named from left to right: MMgO-Sieve Size-Concentration of $MgCl_2$/Concentration of NaCl-Sample Number (1 or 2). For example the following sample name reflects a sample that contains MgO sieve size 10, 1 M $MgCl_2$, no NaCl and it is the first sample of 2: MMgO-10-1.0 Mg/0 Na-1. The subset of experiments with 1.0 M $MgCl_2$ contains the same magnesium concentrations as that of GWB. The magnesium concentration in the subset with 1.65 $MgCl_2$ is close to that of the invariant composition of phase 3 and phase 5 predicted by Xiong et al. (2010). Experimental solutions were spiked with 1.0m CsCl and 1.0m KI solutions. The reason for spiking CsCl and KI is that if colloids are present, Cs^+ and I^- have tendency to sorb to colloids. Sample processing and analyses methods are described in the first milestone report by Xiong et al. (2014c). Solid samples will be analyzed for phase identification by using the XRD and SEM.

The responsible individuals for analysis of the results from Task 3 of TP 12-01 are Sungtae Kim, Yongliang Xiong and Leslie Kirkes. Specially, Yongliang Xiong and Leslie Kirkes will analyze the results regarding solution chemistry, and Sungtae Kim and Leslie Kirkes will analyze the results regarding solid phases. The estimated completion date of for analysis of the results from Task 3 of TP 12-01 is 12/31/2017.

4.4 Task 4. Experimental determination of potential formation of lead-containing mineral fragments in ERDA-6 and GWB.

Due to lead in the waste stream and the radiation shielding materials, lead-containing mineral fragments could be formed from lead ion species under the WIPP conditions. The lead-containing mineral fragments could be lead oxalate (PbC_2O_4) and lead carbonate ($PbCO_3$). These lead-containing mineral fragments are expected to form, when lead is corroded in the WIPP brines (ERDA-6 and GWB). Therefore, Task 4 of TP 12-01 will be to experimentally assess the potential formation of lead oxalate and lead carbonate in un-equilibrated ERDA-6 and GWB that are supersaturated with lead oxalate and lead carbonate, respectively. The experimental procedure will be similar to that in Tasks 1 and 2, except that the target element in Task 4 will be lead.

The Task 4 experiments can serve dual purposes. Firstly, the presence or absence of lead-containing mineral fragments in un-equilibrated ERDA-6 and GWB will be evaluated. Secondly, the dissolved lead concentrations determined from the Task 4 experiments would be used as validation tests for the thermodynamic model for lead developed at SNL.

The responsible individuals for analysis of the results from Task 4 of TP 12-01 are Yongliang Xiong and Leslie Kirkes. The estimated completion date of Task 4 of TP 12-01 is 12/31/2017.

4.4.1 Potential Formation Experiments of Lead Oxalate in ERDA-6 and GWB.

Specifically, both ERDA-6 and GWB will have a background concentration of 0.01 M $Na_2C_2O_4$. For ERDA-6, the pmH will be controlled by equilibrium reactions with brucite ($Mg(OH)_2$). To prevent brucite from complicating the formation of potential lead-containing mineral fragments, brucite powder will be loaded into silver capsules, and the capsules will be tightly crimped at both ends, but not welded shut, to allow it communicate with the brine (Xiong et al., 1999). For GWB, the pmH will be controlled by equilibrium reactions with phase 5 ($Mg_3Cl(OH)_5 \bullet 4H_2O$). The phase 5 powder will be loaded into silver capsules, and the capsules will be crimped. In both ERDA-6 and GWB, lead oxalate will be formed from supersaturation by drop-wise addition of a 0.1 M $PbCl_2$ solution, as precipitation of lead oxalate can be observed when the 0.1 M $PbCl_2$ is added. The sampling of solution sample for filtration will start after about one week of initial precipitation of lead oxalate.

4.4.2 Potential Formation Experiments of Lead Carbonate in ERDA-6 and GWB.

Both ERDA-6 and GWB will have a background concentration of 0.01 M $NaHCO_3$. The pmH will be controlled by equilibrium reactions with brucite ($Mg(OH)_2$) for ERDA-6 and phase 5 for GWB, respectively. Brucite and phase 5 powders will be loaded into silver capsules, respectively, and the capsules will be crimped. In both ERDA-6 and GWB, lead carbonate will be formed from supersaturation by drop-wise addition of a 0.1 M $PbCl_2$ solution. The sampling of solution sample for filtration will start after about one week of initial precipitation of lead carbonate.

5 Special Considerations

5.1 ERDA-6 and GWB Brines

All supporting solutions for Task 4 will be the un-equilibrated ERDA-6 and GWB brines without organic ligands. Their recipes and synthesis are addressed the below.

<u>Un-equilibrated GWB Brine Without Organic Ligands</u>
GWB is a saturated brine containing 3.98m sodium, 1.15m magnesium, 0.53m potassium, 6.32m chloride and 0.20m sulfate. This brine is used in the WIPP geochemical conceptual model and experiments for the brine from the Salado formation (SP 20-4; Xiong, 2008b). Table 1 gives the synthetic composition of the GWB brine. The experimentally measured density for the GWB brine composition is 1.2368 g/mL (SP 20-4).

Table 1. Synthetic GWB Brine Recipe on a Molality Scale (SP 20-4 Appendix C).

Compound	Grams for 1000g H_2O
$MgCl_2 \bullet 6H_2O$	233.23
NaCl	202.32

Compound	Grams for 1000g H_2O
KCl	39.25
Na_2SO_4	28.42
$Na_2B_4O_7$•$10H_2O$	16.96
$CaCl_2$•$2H_2O$	2.29
NaBr	3.09
LiCl	0.21

Un-equilibrated ERDA-6 Brine Without Organic Ligands

ERDA-6 is a saturated brine containing primarily sodium chloride. ERDA-6 contains 5.44m sodium, 0.02m magnesium, 0.11m potassium, 5.20m chloride and 0.19m sulfate. This brine is used in the WIPP geochemical conceptual model and experiments for brine from the Castile Formation. In Table 2, the synthetic composition of ERDA-6 is listed. The experimentally measured density for the ERDA-6 brine is 1.1918 g/mL (SP20-4; Xiong, 2008b)

Table 2. Synthetic ERDA-6 Brine Recipe on a Molality Scale (SP 20-4 Appendix C)

Compound	Grams for 1000g H_2O
$MgCl_2$•$6H_2O$	4.33
NaCl	293.38
KCl	8.11
Na_2SO_4	26.58
$Na_2B_4O_7$•$10H_2O$	6.73
$CaCl_2$•$2H_2O$	1.97
NaBr	1.27
LiCl	None

5.2 Solid starting materials

Mg phase 5 starting material for Task 4 will be synthesized using11.1275 g MgO (Fisher), 11.2697 g $MgCl_2$:$6H_2O$, and 7.4286 g DI water.

5.3 Investigation of Colloids

It has been known that suitable methods of investigating colloids are centrifugation/ultracentrifugation, filtration/ultrafiltration, electrophoresis, and spectroscopy (Zhao and Steward, 1997). The present analysis plan will use filtration/ultrafiltration in combination with centrifugation to separate the possible mineral fragments, if there are any present, from the dispersing media. Conventionally, aquatic colloids are operationally defined as the particles with a size between 1 nm and 1 μm (Guo et al. 1997, Guo et al. 2007). Aquatic colloids are isolated from aquatic system by filtration of samples through 0.2 μm pore-size filters. The lower limit for colloids is usually between 1 and 10 kDa, equivalent to 1 and 3 nm (Guo et al. 1997). An ultra-filtration device under TP 12-01 will be used in ranges of 10, 30, 50, and 100 kDa, equivalent to pore sizes of 3, 4, 5, and 7 nm. Table 3 shows pore sizes

corresponding to the ultra-filtration filters (10, 30, 50, and 100 kDa), and 0.2 μm membrane filter.

The samples will be successively filtered through 5 different filters with decreasing pore sizes (eg. Millipore or other brand ultra centrifugal filters), whose sizes range from 200 nm to 3 nm (Table 3). The absence or presence of phase 5 and hydromagnesite(5424) colloids will be determined through the magnesium and spiked element(s) (such as Cs^+) concentration analyses. Phase 5 has a chemical formula of $Mg_3Cl(OH)_5 \bullet 4H_2O$, and hydromagnesite(5424) has a chemical formula of $Mg_5(CO_3)_4(OH)_2 \bullet 4H_2O$. If phase 5 or hydromagnesite(5424) mineral fragments are present in the solution, they will be retained by the filters, and the magnesium and spiked element concentrations in the aliquots will decrease with decreasing filter pore size. A constant value for magnesium and spiked element concentration, within the experimental uncertainty, demonstrates the absence of phase 5 or hydromagnesite(5424) colloids.

Table 3. Ultra-Filtration devices and filtration of samples used in this work, with their corresponding nominal molecular weight limit and pore sizes.

Ultra-filtration Device	Nominal Molecular Weight Limit (Da)	Pore Size (nm)
10k	10,000	3
30k	30,000	4
50k	50,000	5
100k	100,000	7
Filtration of Samples		**Pore Size (nm)**
0.2μm		200

6 Applicable Procedures

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"

- SP 20-4 – "Preparing Synthetic Brines for Geochemical Experiments"
- NP 17-1 – "Records"
- NP 20-2 – "Scientific Notebooks"

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