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Sandia National Laboratories
Waste Isolation Pilot Plant

Test Plan TP 14-04

**Experimental Investigation of Stability of Mineral
Colloids under WIPP Conditions**

Task 4.4.2.2.1

Revision 2

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1 ABBREVIATIONS, ACRONYMS AND INITIALISMS

CCA	Compliance Certification Application
DAS	Data Acquisition System
ERDA-6	Energy Research and Development Administration well 6 (Synthetic Castile Formation brine)
GWB	Generic Weep Brine (Synthetic Salado Formation brine)
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectrometer
ICP-MS	Inductively-Coupled Plasma Mass Spectrometer
M&TE	Measuring and test equipment
NP	Nuclear Waste Management Procedure
QA	Quality Assurance
SNL	Sandia National Laboratories
TP	Test Plan
UV-Vis	Ultraviolet-Visible
WIPP	Waste Isolation Pilot Plant

2 REVISION HISTORY

This is Revision 2 of this Test Plan (TP). Extensive changes were made to this TP. This current revision features additional characterization techniques to analyze lower colloid concentrations. In addition, all iron based colloids and equilibration with magnesium oxide (MgO) were removed from the TP.

In revision 1 of this TP, the task numbers were updated and a mineral classification chart was added.

3 PURPOSE AND SCOPE

The purpose of this test plan is to experimentally determine the potential formation and stability of mineral fragment-type pseudo-colloids that may be present in the Waste Isolation Pilot Plant (WIPP) repository environment in simulated Salado-Castile brines (Generic Weep Brine, GWB; Energy Research and Development Administration well 6, ERDA-6). The simulated brines (GWB and ERDA-6) will cover a broad range of salinity and represent the bulk composition of the intergranular brines from the Salado and Castile Formations at or near the stratigraphic horizon of the WIPP. The brines tested in these experiments will include simulants of Salado groundwater, "Culebra brine," and a NaCl-CaCl₂ solution. Each of the minerals will be tested in simplified GWB and ERDA-6 and in simplified GWB (1 M MgCl₂ and 3.6 M NaCl). In addition, in order to facilitate experimental work, the simulated brines will be equilibrated with halite (NaCl), anhydrite (CaSO₄), brucite (Mg(OH)₂), and hydromagnesite (Mg₅(CO₃)₄(OH)₂·4H₂O) as per SP 20-4.

Colloids are particles with diameters ranging from 1 nm to 1 μm that have high surface area that may affect contaminant transport which present high sorption capacity for many solutes. They are considered rapid transporters of potential heavy metals and radionuclides in natural media such as groundwater (Abdel-Salam and Chrysikopoulos, 1995a, Abdel-Salam and Chrysikopoulos, 1995b, Tatalovich et al., 2000 and James et al., 2005). Colloid-driven transport is a cause of concern in assessing the long-term performance of deep geological repositories of radioactive waste, especially those hosted in crystalline rock. At the WIPP, colloid particles form in the repository by a variety of process, including waste degradation, microbial activity, rock decomposition, and chemical condensation. In theory, WIPP colloids may react with actinides in the WIPP and therefore facilitate actinide mobility. Carrier colloids (also known as "pseudo-colloids," type II colloids) are far smaller than the pores in impermeable and fractured media, and their high surface area per unit mass means that they will be effective sorption substrates. A natural colloid becomes a mineral pseudo-colloid when a radionuclide adsorbs to it and, may undergo different sorption processes than dissolved species. The primary distinction between the dissolved species and that of the different colloidal particles is whether particles will diffuse into the groundwater matrix. This is controlled in part by the size difference and resultant concentrations amongst the colloid particles (Title 40 CFR Part 191 CCA). Currently, CCA actinide concentrations associated with mineral fragment-type pseudo-colloidal particles are estimated based on results from WIPP experiments designed to determine mobile concentrations in brines, coupled with site-binding densities of mineral substrates, not linked to the concentration of dissolved actinides (DOE 1996, SOTERM.6.3). Hence, the concentration of mineral fragment-type pseudo-colloidal particles will be determined if applicable.

Additional quantitative assessment, such as those listed in section 4.5.3, of the mineral-type fragment pseudo-colloids will be performed if the presence of such colloids is established. A low detection limit on the order of 10 µg/L) is needed for quantitative assessment of the colloidal concentration.. The investigation of presence or absence of mineral fragment colloids under the WIPP relevant conditions is necessary, as the presence of such colloids could have an effect on the actinide source term. 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.

Particular mineral fragments present in the stratigraphic horizon of the WIPP include:

- A. Colloid-size fragments of mineral materials tested for the Compliance Certification Application (CCA) (DOE 1996, SOTERM.6.3.1):
 - Bentonite, kaolinite, montmorillonite, vermiculite, illite, anhydrite, calcite, magnesite, hematite, limonite, goethite, magnetite, quartz, siderite, brucite, strontianite, diatomaceous earth, and pyrite.
- B. Colloidal fragments of water-insoluble minerals identified in the vicinity of the repository horizon:
 - Quartz, magnesite, anhydrite, gypsum, polyhalite, mixed-layer saponite-chlorite with lesser chlorite and illite, potassium feldspar, and corrensite (a chlorite mineral).
- C. Colloid-size fragments of minerals identified as potentially introduced in large quantities during pre-closure activities or during a post-closure event:
 - Periclase (MgO) and bentonite.

The minerals identified for study in this TP will be broken up into specific mineral classes to facilitate dissemination of information. This list reduces to the following 16 minerals and corresponding classes as seen in Table 1. The minerals that are not listed in Table 1 that are mentioned above in A, B, and C will be included in other WIPP SNL comparable ongoing, planned or completed laboratory studies involving GWB and ERDA-6.

Table 1. TP 14-04 Minerals and Classification

Phyllosilicate Class	Sheet Silicates
Bentonite	No defined chemical formula, primarily consisting of montmorillonite
Montmorillonite	$\text{Na}_{0.2}\text{Ca}_{0.1}\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2(\text{H}_2\text{O})_{10}$
Kaolinite	$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$
Vermiculite	$\text{Mg}_{1.8}\text{Fe}^{2+}_{0.9}\text{Al}_{4.3}\text{SiO}_{10}(\text{OH})_2 \cdot 4(\text{H}_2\text{O})$
Illite	$\text{K}_{0.6}(\text{H}_3\text{O})_{0.4}\text{Al}_{1.3}\text{Mg}_{0.3}\text{Fe}^{2+}_{0.1}\text{Si}_{3.5}\text{O}_{10}(\text{OH})_2 \cdot (\text{H}_2\text{O})$
Diatomaceous Earth	No defined chemical formula, mostly consisting of silica and traces of alumina and iron oxide.

Tectosilicate Class	Framework Silicates
Quartz	SiO ₂
Potassium Feldspar	KAlSi ₃ O ₈
Sulfate Mineral Class	
Anhydrite	CaSO ₄
Gypsum	CaSO ₄ ·2H ₂ O
Polyhalite	K ₂ Ca ₂ Mg(SO ₄) ₄ ·2H ₂ O
Carbonate Mineral Class	
Strontianite	SrCO ₃
Calcite	CaCO ₃
Magnesite	MgCO ₃
Oxide & Hydroxide Class	
Brucite	Mg(OH) ₂
Periclase	MgO

For the CCA, similar experiments were performed to measure the stability of humic mineral fragment and microbe particles in WIPP brine. As noted above, the minerals in A (DOE 1996, SOTERM.6.3.1) were tested by Papenguth and Behl (1996) in coagulation series experiments. The brines tested in these experiments included simulants of Salado groundwater, "Culebra brine," and a NaCl-CaCl₂ solution. These brines were diluted sequentially to produce a set of solutions covering a broad range of salinity. The maximum salinity of the Salado groundwater simulant was 10% of the salinity of groundwater in the Salado Formation. Among the particles tested, only that of bentonite showed some stability in the 10-fold diluted Salado brine simulant. Considering that all groundwater in the WIPP environment are brines, we are assessing the stability of mineral colloids in the brines likely to be involved in the transport of actinides in the WIPP.

Excluded from this study are minerals that are included in other comparable ongoing, planned, or completed laboratory studies involving simulated GWB and ERDA-6 brines. Degradation of iron materials in the repository may generate iron mineral colloids. However, iron mineral colloids are being studied in a separate test plan (TP 14-03) and therefore are not included in the planned experiments. In addition, the possible formation of mineral fragment colloids equilibrated with MgO added to simulated brines are being evaluated under TP 12-01 and therefore excluded. However, periclase will be included in the experiments since it is used in large quantities as an engineered barrier in the repository. In the future, exploratory drilling may occur in the vicinity of the repository. Drilling fluid commonly contains clay fragments such as bentonite. Therefore, bentonite, which was also tested in the CCA experiments, will be included in the set of experiments. Due to the inability to obtain Corrensite, the mineral is also omitted from this study. Corrensite and chlorite have been studied over the last several years with respect to the corrensite-to-chlorite conversion, and the debate of the nature of this conversion is still controversial. Therefore, chlorite was not used as an alternative to corrensite here. Tasks for this experimentation are as follows:

1. *Characterize starting materials: solids, brines etc.*
2. *Experimental determination and/or characterization of absence or presence of mineral-type fragment pseudo-colloids of Phyllosilicate Mineral Class*
3. *Experimental determination and/or characterization of absence or presence of mineral-type fragment pseudo-colloids of Tectosilicate Mineral Class*
4. *Experimental determination and/or characterization of absence or presence of mineral-type fragment pseudo-colloids of Sulfate Mineral Class*
5. *Experimental determination and/or characterization of absence or presence of mineral-type fragment pseudo-colloids of Carbonate Mineral Class*
6. *Experimental determination and/or characterization of absence or presence of mineral-type fragment pseudo-colloids of Oxide and Hydroxide Mineral Class*

4 EXPERIMENTAL PROCESS DESCRIPTION

4.1 Overall Strategy and Process

The overall strategy is to determine the stability of mineral fragment colloids in WIPP relevant conditions. If mineral fragment colloids are shown to be stable in synthetic brines, then the highest concentration of colloids present will be determined. The colloid concentration will be determined by either applying experimental separation techniques that will not underestimate the total mass of particulate matter present (seen using filtration) or result in a different charge to mass ratio or agglomeration due to heat (seen using centrifugation) at reduced ionic strengths or utilize characterization techniques that do not require separation methods.

4.1.1 Experimental Procedure

Task 1. Characterize starting materials: Brines, Solids, etc.

All starting material minerals will be characterized prior to the start of experimental set-up. Materials purchased from all vendors will be analyzed before use in experiments (even that in pure form). Due to the fact that some of the minerals are from natural sources and cannot be purchased in the pure form, this step is critical in understanding the purity of the mineral. Starting solids will be characterized as needed, utilizing techniques described in section 4.5.3 (D).

Starting brine solutions will also be characterized as needed, utilizing instruments as described in section 4.5.3 (C).

Task 2-6. Experimental determination and/or characterization of absence or presence of mineral-type fragment pseudo-colloids of all mineral classes.

After characterization, the solid starting material will be ground into a fine powder and analyzed to determine chemical purity. All simulated brines will be prepared in accordance to

Activity/Project Specific Procedure (SP) 20-4 (Xiong 2008). The recipes will be developed to match as closely as possible to the compositions of equilibrated and non-equilibrated GWB and ERDA-6 brines according to SP 20-4. The compositions of the prepared simulated brines will be verified by chemical analyses. Hydrochloric acid, boric acid or sodium hydroxide may be used to adjust the pH and p_H (hydrogen ion concentration) of the brine, if necessary. Colloid-GWB and Colloid-ERDA-6 brine mixtures will be prepared for each colloid-GWB and colloid-ERDA-6 brine combination. In addition, if needed, mixtures will be prepared for dilutions of simplified, and equilibrated GWB and ERDA-6 brine and for a dilute NaCl solution. The dilute NaCl solution is needed to examine the potential colloid concentrations given conditions conducive to colloid stability. In order to do this, the pH for the colloid mixture will be monitored and adjusted if necessary. Colloid suspensions will be prepared by precipitation of chemical reagents or by mechanical disaggregation of representative material. The original size distributions of the mineral fragment colloids will ensure if possible expansion of porous particles occurred by a minimum addition of 1 mg of test mineral per liter of brine per size fraction. This addition will be ensured by weighing equipment and particle size distribution analysis of source mineral fragment material. After a determined period of time, supernatants of these mixtures will be analyzed for pH and stable colloid suspensions. Samples of the supernatants will be collected as needed; however, no less than two collections will be made.

Characterization techniques will first be attempted that allow the supernatant, aliquots, precipitates, etc. to be analyzed without separation techniques. If characterization cannot be accomplished without separation techniques, then either decantation, filtration, evaporation, distillation or centrifugation will be employed. Analytical characterization techniques can then be utilized to provide qualitative and quantitative data. For example, after removing the large, non-colloidal size particles using a 1000 nm filter, size fractions could be collected using at a minimum the following (or comparable) sequential filters: 450, 200, 50, 10 (if possible) and 2.5 nm (if possible). These suspensions will be characterized and monitored over time. The volumes of these mixtures will be large enough to provide a sufficient amount of supernatant for several replicate sets for characterization by multiple analytical techniques and to provide continued sampling over an extended time period for testing long term stability. At which point, the samples would be passed through filters of sequentially smaller pore size. Filters in the colloidal size range (< 1000 nm) will be examined to identify the mineralogy and to quantify the concentrations of observed particles in each size fraction.

Laboratory activities will be recorded in scientific notebooks, and they will be maintained as quality assurance (QA) records.

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 with a unique sample ID. 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 at room temperature and sealed in a secure area with associated documentation (e.g., SNL WIPP Activity/Project Specific Procedure (SP) Form SP 13-1-1, "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 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; and (4) maintaining calibration records. In addition, NP 13-1 and SNL Activity/Project Specific Procedure (SP) 13-1, "Chain of Custody", identify requirements and appropriate forms for documenting and tracking samples possession. Computer-based data handling will follow NP 9-1, "Analyses".

4.3.2 Data Acquisition System

Data collection procedures are specific to individual instruments. For details of the data acquisition for a particular instrument, see the Specific Procedures (SP) or User's 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.

4.4 Data Identification and Use

All calculations performed as part of the activities of TP 14-04 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.

4.5 Equipment and Techniques

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.

4.5.1 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 NBS-traceable weight sets, which, in turn, are calibrated by the SNL Calibration Laboratory. Calibration checks will be recorded in a Balance Calibration Records log book.

4.5.2 Liquid Measuring Equipment

Standard Laboratory Class A glassware (pipettes, volumetric flasks, etc.) will be used at all times. In addition, several adjustable pipettes are available for use in the laboratory. The calibration of pipettes will be checked periodically against a calibrated balance, and will be recorded in the scientific notebook. The accuracy of pipettes will be within 1%.

4.5.3 Other Analytical Equipment & Techniques

- A. Centrifuge—Floor Centrifuge.
- B. pH Meters and Autotitrators – Solution pH may be measured using pH meters and/or autotitrators. The pH probes and meters used will be recorded in the appropriate scientific notebook at the time of use. The range for all pH meters is 0.00 to 14.00. SP 12-14 will be followed for this procedure.
- C. Equipment for Chemical Analysis – The following instruments may be used for chemical analyses:
 - Inductively-Coupled Plasma Mass Spectrometer (ICP-MS)
 - Inductively-Coupled Plasma Atomic Emission Spectrometer (ICP-AES)
 - UV-Visible (UV-Vis) Spectrophotometer
 - CO₂ coulometer
 - Ion Chromatograph (IC)
 - Particle Size Analyzer
 - Refractive Index Analyzer

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

D. Experimental Techniques for Mineralogical, Stability, and Textural Characterization – The mineralogy and texture of samples may be characterized using the following methods:

- **Light Transmission-** a nondestructive, noninvasive method that allows for quantitative measurement of colloid distribution with unprecedented two-dimensional spatial and temporal resolution. This technique is well-suited to observing the effects of saturation transitions and physical heterogeneities on colloidal transport. This technique is explored by investigating the effect of particle size and size calculations.
- **Light Scattering-** (static and dynamic) a nondestructive and non-invasive technique for determination of molecular mass, hydrodynamic radius, polydispersity, kinetics of aggregation, and second virial coefficient in order to ascertain concentrations.
- **Single Particle Counting-** detects and counts pulses of scattered light from particles one at a time by either light scattering, light obscuration, or direct imaging. This technique is used for particle data, trend tracking, statistically valid sampling, and data normalization. It provides particle number, particle size and distribution.
- **Indirect Techniques-** Because many properties of colloidal suspensions depend on the state of aggregation of the suspended particles, various indirect techniques have also been used to monitor particle aggregation. While it can be difficult to obtain quantitative information on aggregation rates or cluster properties from such experiments, they can be valuable for practical applications.
 - **Settling Tests** - inspects a series of test tubes with suspensions prepared at different concentration of the flocculant, stable suspensions often remain dispersed, while the unstable ones settle. Automated instruments based on light scattering/transmittance to monitor suspension settling have been developed, and they can be used to probe particle aggregation.
 - **Filtration** - separation of materials of different chemical composition; filters particulates of different size
 - **Rheology** - characterizes the rheological properties of materials. Rheometers impose a specific stress field or deformation to the fluid, and monitor the resultant deformation or stress.
 - **Ultrasonic waves** - can identify the existence of flaws, measure their size, thickness and identify their location.
 - **Dielectric Properties** - (Electrical Impedance Spectroscopy) measures dielectric properties of medium as a function of frequency. Interaction of external fluid with electric dipole moment (permittivity). This can reveal reaction mechanisms of electrochemical process, identify rate limiting step, and microstructural characterization. The downside to this technique is that it is very dependent on temperature, pressure, and chemical surroundings.

5 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 13-1 – "Control of Samples and Standards"
- SP 13-1 – "Chain of Custody"
- NP 12-1 – "Control of Measuring and Test Equipment"
- NP 20-2 – "Scientific Notebooks"
- NP 17-1 – "Records"

6 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 (SOP). 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 PERMITTING/LICENSING

There is no special license or permit requirements for the activities described in this Test Plan.

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