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Second Milestone Report on Test Plan TP 14-04, "Experimental Investigation of Stability of Mineral Colloids under WIPP Conditions"

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DEFINITION OF ABBREVIATIONS, ACRONYMS AND INITIALISMS

Abbreviation or Acronym	Definition
ASAP	Accelerated Surface Area and Porosimetry System
DI	de-ionized
EBSD	Electron backscatter diffraction
EDS	Energy dispersive spectroscopy
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 Spectroscopy
ICP-MS	Inductively Coupled Plasma Mass Spectroscopy
m	molal (mol/kg H_2O)
M	molar (mol/L)
nm	Nano meter, i.e., 10^{-9} meter
QA	quality assurance
SEM	Scanning electron microscope
SNL	Sandia National Laboratories
Syn	Synthetic
TGA/DSC	thermogravimetric analysis/differential scanning calorimetry
TP	Test Plan
WIPP	Waste Isolation Pilot Plant
XRD	X-ray diffractometer

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1 INTRODUCTION

The purpose of this milestone report is to provide an update on the status of TP 14-04 (Kirkes et al. 2014). A brief introduction to the test plan is provided to orient the reader. Mineral fragment colloids, also known as mineral colloids, have been identified as potential vehicles of actinide mobility in the Waste Isolation Pilot Plant (WIPP) (Papenguth and Behl TP 96-01 1996). Mineral fragment colloids have the potential to bind to dissolved actinides, and as such they act to enhance actinide solubility. Colloidal particles are usually defined as 1 nm to 1μm in size. Mineral colloids can be present in natural groundwater as well as the surrounding geology of the WIPP. Mechanical disturbance of mineral surfaces in the area and drilling operations can also produce mineral colloids.

The purpose of TP 14-04 is to determine the aqueous stability of mineral fragment colloids in WIPP relevant conditions. Mineral colloids are kinetically stabilized and destabilized by electrostatic forces (Papenguth and Behl, 1996). In a kinetically stable colloidal dispersion, colloidal particles repel each other, and therefore stay suspended (Papenguth 1996). As the ionic strength of the matrix increases, repelling forces decrease. Weaker repulsion causes an increase in the potential for colloid agglomeration and gravimetric settling. Because WIPP groundwaters have high ionic strength, measurable concentrations of colloids may be unsustainable in WIPP conditions.

Colloidal dispersions will be made for each of the minerals listed in TP 14-04. The mineral fragment particles to be tested include colloid-size fragments of natural geologic minerals of water-insoluble minerals identified in the vicinity of the repository horizon (calcite, magnesite, strontianite, polyhalite, ankerite, dolomite, anhydrite, gypsum, quartz, illite and chlorite). These dispersions will be introduced into various brine concentrations (see Table 1). Each mineral fragment colloid will be tested in the matrices listed in Table 1. This range of matrices will span all expected ionic strengths of the WIPP and is expected to show decreasing colloidal stability with increasing ionic strength. Establishing such a trend is useful and can serve as a control if colloid concentrations are below detection limits at the higher ionic strengths of WIPP brines. GWB has been reported to have an ionic strength of 8.26 m and ERDA-6 is reported to have an ionic strength of 5.82 m (Xiong and Lord, 2008). These will be the maximum ionic strengths researched in TP 14-04.

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Table 1 Test Matrices

Test Matrices	
100% GWB	100% ERDA-6
GWB at 10X dilution	ERDA-6 at a 10X dilution
GWB at a 100X dilution	ERDA-6 at a 100X dilution
Simplified GWB	0.01M NaCl

The minerals are grouped in chemically similar mineral classes to facilitate simpler dissemination of information. These groups will stay consistent throughout the following sections. Table 2 shows the mineral classes and their respective task numbers.

Table 2 Mineral Classes for Analysis and Tasks

Task 1: Characterize Solid Starting Materials	
Task 2: Set-up and Characterize Colloid Concentrations for Experiments for Phyllosilicate Mineral Class	
Illite	$KAl_4[Si_7AlO_{20}](OH)_4$
Corrensite	$(Ca, Na, K)(Mg, Fe, Al)_9(Si, Al)_8O_{20}(OH)_{10}\cdot nH_2O$
Task 3: Set-up and Characterize Colloid Concentrations for Experiments for Tectosilicate Mineral Class	
Quartz	SiO_2
Task 4: : Set-up and Characterize Colloid Concentrations for Experiments for Sulfate Mineral Class	
Anhydrite	$CaSO_4$
Gypsum	$CaSO_4\cdot 2H_2O$
Polyhalite	$K_2Ca_2Mg(SO_4)_4\cdot 2H_2O$
Task 5: Set-up and Characterize Colloid Concentrations for Experiments for Carbonate Mineral Class	
Strontianite	$SrCO_3$
Calcite	$CaCO_3$

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Magnesite	$MgCO_3$
Ankerite	$Ca(Fe, Mg, Mn)(CO_3)_2$
Dolomite	$CaMg(CO_3)_2$

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2 MATERIALS AND METHODS

Necessary minerals for this test plan have been purchased where available. All of the purchased minerals have been characterized for purity using the X-ray diffractometer (XRD) and any minerals purchased in the future will be characterized as well. It may be necessary to chemically synthesize some of the minerals if the measured purity of the purchased minerals is not deemed acceptable. In many cases, both a natural and synthetic form of each mineral have been obtained and will be tested.

The purchased minerals have been verified for purity and the results are reported in this document. At this time, all the purchased minerals are being ground in an effort to create colloidal size particles (1nm-1μm) and the progress of this effort is documented in this report. This is being accomplished through various automatic and manual grinding techniques. Colloid size is being determined using a particle size analyzer (Malvern Mastersizer), filtering techniques and scanning electron microscope (SEM). All supporting solutions, will be prepared from reagent grade chemicals from Fisher Scientific or its associated vendors.

Further analysis of the starting mineral colloids may be desired or necessary by utilizing physisorption analysis or thermogravimetric analysis/differential scanning calorimetry (TGA/DSC).

Once the starting materials have been fully characterized the colloidal suspension solutions will be created at concentrations of 100mg/100mL. This concentration may need to be altered if the colloid concentration is not detectable by the analysis techniques. Colloidal suspensions will be analyzed by a particle size analysis if possible, and inductively coupled plasma mass spectroscopy (ICP-MS) and/or inductively coupled plasma atomic emission spectroscopy (ICP-AES), if necessary. In addition, the suspension will be analyzed on the scanning electron microscope/energy dispersive spectroscopy (SEM/EDS), to determine absence or presence of colloids and physical characteristics. UV/VIS techniques for concentration determination are also being explored and developed for this test plan.

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2.1 Ball Mill Grinding

The physical process of grinding the minerals is still ongoing for most of the minerals in this test plan. Achieving colloidal size particles (<1um, <1000nm) is a very difficult task for some types of minerals, due to their physical properties (e.g. Mohs hardness, conchoidal fracturing properties etc.). A Retsch Planetary Ball Mill PM100, with a Zirconia Oxide grinding jar and Zirconia Oxide beads are being used for the grinding process. Two sizes of Zirconia Oxide beads are being used: 1mm and 3mm. The grinding process varies slightly for each mineral and is documented in the associated scientific notebooks (WIPP-Colloids-1 and WIPP-Colloids-2). Wet grinding and Dry grinding have been employed for mineral processing. If not already in a powder form, the minerals were all ground in a mortor and pestal to render them in a powder form. XRD analysis was conducted on all the ground minerals. The general dry grinding procedure is outlined below.

General Dry Grinding Procedure:

1. Place ~1 gram of material in zirconia oxide grinding jar along with ~12 1mm zirconia oxide beads
2. Run Retsch program at 500rpm for 3 hours with 7 minute intervals and 2 minute rests (Intervals are set-up in the program so that the jar rotates in one direction for 7 minutes at 500 rpm, the vessel rests for 2 minutes (to allow the mineral and vessel to cool) and then the jar rotates in the inverse direction for 7 more minutes at 500 rpm.)

The program is designed in this way to prevent the mineral from over-heating and potentially changing phases. After the 3 hour grinding process the sample was analyzed for particle size distribution. If the desired size range was not achieved, the 3 hour process was repeated until the desired results were achieved or a point of diminishing return was reached and another grinding process was considered. If the desired particle size distribution results are not achieved with dry grinding, wet grinding may need to be employed. The general wet grinding procedure is outlined below.

General Wet Grinding Procedure:

1. Place ~1-2 grams of material in zirconia oxide jar along with ~15 3mm zirconia oxide beads.

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2. Fill grinding jar ~66% with Ethanol or other appropriate solvent
3. Run Retsch program at 400 rpm for 2 hours within pre-determined intervals and 5 minute rests (Intervals are set-up in the program so that the jar rotates in one direction for pre-determined minutes at 400 rpm, the vessel rests for 5 minutes (to allow the mineral and vessel to cool) and then the jar rotates in the inverse direction for the pre-determined minutes at 400 rpm.)

This process was repeated until the desired particle size was achieved or a limit of diminishing return was reached and then other processes were considered. All deviations to these procedures are outlined in the associated scientific notebooks (series WIPP-Colloids). The wet and dry general grinding procedures described here are examples of what has been performed. These procedures can be altered and customized for each mineral as needed and changes and processes are documented in the scientific notebooks. Wet grinding processes are considered with great care because certain issues may arise with this process, such as causing mineral chemical transformation. For example, ethanol was used in an attempt to achieve colloidal size particles with gypsum ($CaSO_4 \cdot 2H_2O$). The process was successful at producing a higher percentage of colloidal size particles, however after grinding with the ethanol process, the XRD analysis was repeated and some of the gypsum was transformed to anhydrite ($CaSO_4$), rendering the sample unusable for the project (WIPP-Colloids-1 Pgs. 76-77).

2.2 Particle Size Distribution Analysis

Throughout the grinding process for each mineral, a Malvern Mastersizer 3000 was used to determine the particle size distribution of each mineral at each grinding stage. The results of the grinding process and particle size distribution data can be seen in the Tables in sections 3.1.1 to 3.1.4. The instrument has two analysis modes; a dry analysis mode using an Aero S accessory and a wet analysis mode using the Hydro MV accessory. Due to agglomeration of colloidal size particles in fluid, it may be necessary to analyze some of the minerals for particle size in the dry mode to obtain accurate particle size data. In this report, only the wet mode has been utilized for particle size analysis, however all minerals will be analyzed using both methods. Once the dry mode analysis is completed for each mineral, the results will be documented in a scientific notebook and reported in the next version of this milestone report.

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In a typical particle size analysis report, the following parameters and a corresponding brief description are provided:

1. The **Residual**, is an indication on how well the analysis data was fitted to the measurement data. A good fit is indicated by a residual of under 1%.
2. The **Obscuration** helps set the concentration of the sample when it is added to the dispersant. It is a measure of the amount of laser light lost due to the introduction of the sample within the analyzer beam. An ideal range is between 0.10 and 25%.
3. **Dv (10)** is the size of the particle for which 10% of the sample is below this size.
4. **Dv (50)** is the size of the particle at which 50% of the sample is smaller and 50% is larger than this size. This value is also known as the Mass median diameter (MMD).
5. **Dv (90)** gives a size of particle for which 90% of the sample is below this size.
6. **D [4,3]** is the volume mean diameter.
7. **D [3,2]** is the surface area mean diameter. Also known as the Sauter mean.
8. **Span** is the measurement of the width of the distribution. The smaller the value the narrower the distribution. The width is calculated as:
    a. $(d(0.9)-d(0.1))/(d(0.5))$
9. **Concentration** is the volume concentration. It is calculated from the Beer-Lambert law and is expressed as a percentage.
10. **Uniformity** is a measure of the absolute deviation from the median.
11. **S.S.A (Specific Surface Area)** is defined as the total area of the particles divided by the total weight.

2.3 Solid Materials Analysis Techniques

Solids of interest were purchased where available, however some may need to be synthesized. Solutions will be prepared using appropriate reagent grade commercial salts. Purchased solid starting materials were analyzed using the Bruker D8 XRD and further analysis may be completed using the JEOL JSM 5900LV SEM wit Thermo NORAN System 7 EDS. In addition, solid starting materials may be measured for surface area and porosity, using the Micromeretics ASAP 2020. Once the solids were ground into colloid size particles, the size of

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the particles were verified by analysis on the Malvern Mastersizer. The experiments will not be started until the purity of the solid has been verified and the size of the solid particles has been assessed and deem acceptable. When grinding solids, it is desired that all particles be less than 1000 nm in size (1um). Achieving 100% of particles below this threshold may not be possible, however at least 60% of the material is desired to be in the colloidal size range. The most important aspect of this is that the stability of the present colloids can be determined. It may be possible to achieve this goal with much lower percentages of colloids present than the desired 60%.

2.4 Colloid Suspension and Sample Analysis Techniques

This work for TP 14-04 has not yet been started. Liquid colloidal suspensions will be made by mixing each starting solid colloid at 100mg/100mL. This suspension will be analyzed on various instruments (UV/VIS, Mastersizer, etc.), to verify the colloid dispersion concentration.

GWB brine and ERDA-6 brine will be made using reagent grade commercial salts as described in SP 20-4 (Xiong 2008). The Simplified GWB brine containing 1 M $MgCl_2$ and 3.6 M NaCl will also be made using reagent grade commercial salts. All starting brine solutions will be equilibrated with large particles of the colloid mineral for approximately 2 weeks. This will allow each solution to be saturated with the mineral prior to adding the colloidal solid to the solution. This will ensure that the colloid particles will not dissolve after they are added to the solution and give a false result. If necessary or desired, matrix elemental concentrations and colloid concentrations of interest will be determined by using the Perkin Elmer Optima DV 8300 ICP-AES and the NexIon 300D ICP-MS.

After the experiments have been set-up and the colloids added to the described solutions, the experiments will be sampled periodically to determine if the colloid can persist in the described conditions. A liquid sample will be extracted from the experiment. A portion of the extracted sample will be used for liquid analysis to determine whether the composition of solution has changed (e.g., pH) and other analysis such as the particle size analysis. Another portion of the sample will also be filtered through various size filters (nm sizes: 450, 200, 50, 10 (if possible) and 2.5 (if possible)) and then analyzed on the scanning electron

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microscope/energy dispersive spectroscopy (SEM/EDS) for colloid presence. The filter will be surveyed for colloid absence or presence and the concentration will be determined if possible.

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3 RESULTS

3.1 Task 1: Solid Characterization

All solid starting materials were characterized for purity before use in the experiments. Characterization of the solids was completed for the starting materials that have been obtained. Some of the minerals still need to be purchased, obtained or synthesized including: ankerite, chlorite, corrensite (if possible) and magnesite. The completed, XRD and particle size analysis for the obtained minerals, is summarized in the following sections. The International Centre for Diffraction Database PDF4+ software was utilized in the following XRD analyses to verify and match the mineral XRD patterns to existing verified mineral patterns in the ICDD database.

3.1.1 Phyllosilicate Mineral Class Solid Characterization

Phyllosilicates are a class of minerals with sheet like atomic structures. Clays make up a large part of this mineral class. The minerals in this study that fall within this class are: illite ($K_{0.6}(H_3O)_{0.4}Al_{1.3}Mg_{0.3}Fe^{2+}_{0.1}Si_{3.5}O_{10}(OH)_2 \cdot (H_2O)$) and corrensite ($(Mg_{0.2}(Fe^{3+}_{0.34}Mg_{7.32}Al_{1.10})(Al_{1.36}Si_{6.64})O_{20}(OH)_{10}$). Finding a pure source of this class of minerals is difficult due to the complex nature of the chemical formulas as well as the relative abundance of the chemicals in nature. A source of corrensite could not be found, however two sources of Illite were found and purchased. The Illite sources were from The Clay Mineral Society (Illite, Green Shale) and also Ward's Scientific (Illite IMt-2, Cambrinshale).

Purity Characterization

The XRD results from both illite sources are displayed in Figure 1 and Figure 2. Both illite sources were verified utilizing the ICDD database and matched with good agreement to verified illite XRD pattern PDF card 00-015-0603. The natural illite sample from the Clay Mineral Society showed a slightly more crystalline XRD pattern than the illite sample from Ward's Scientific (IMt-2) as seen in the figures below.

Figure 1 XRD Analysis of Natural Illite (Green Shale) Clay Mineral Society

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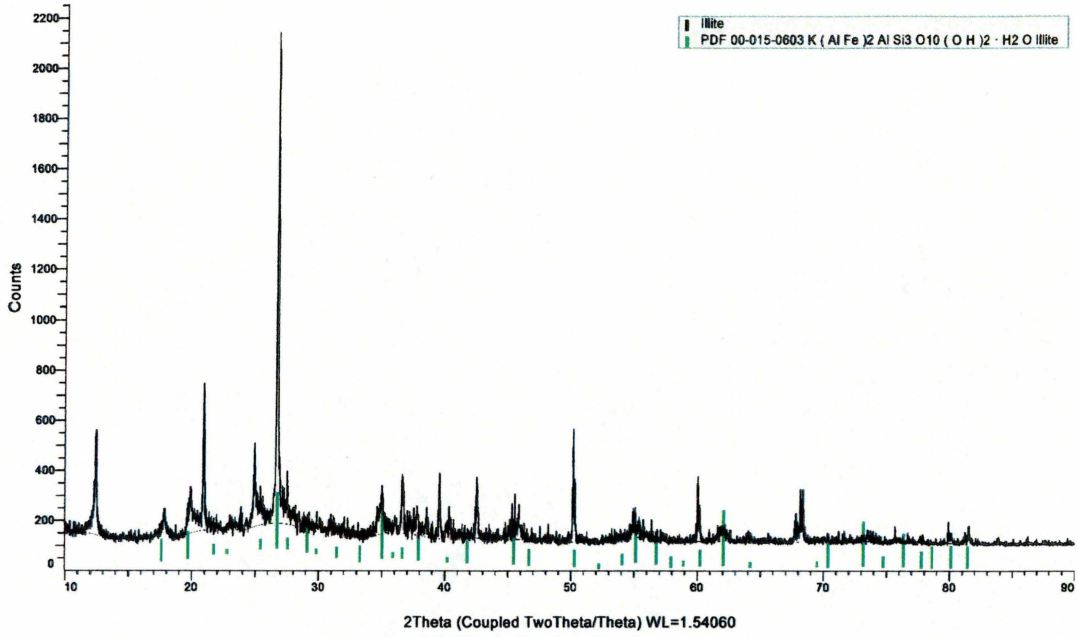
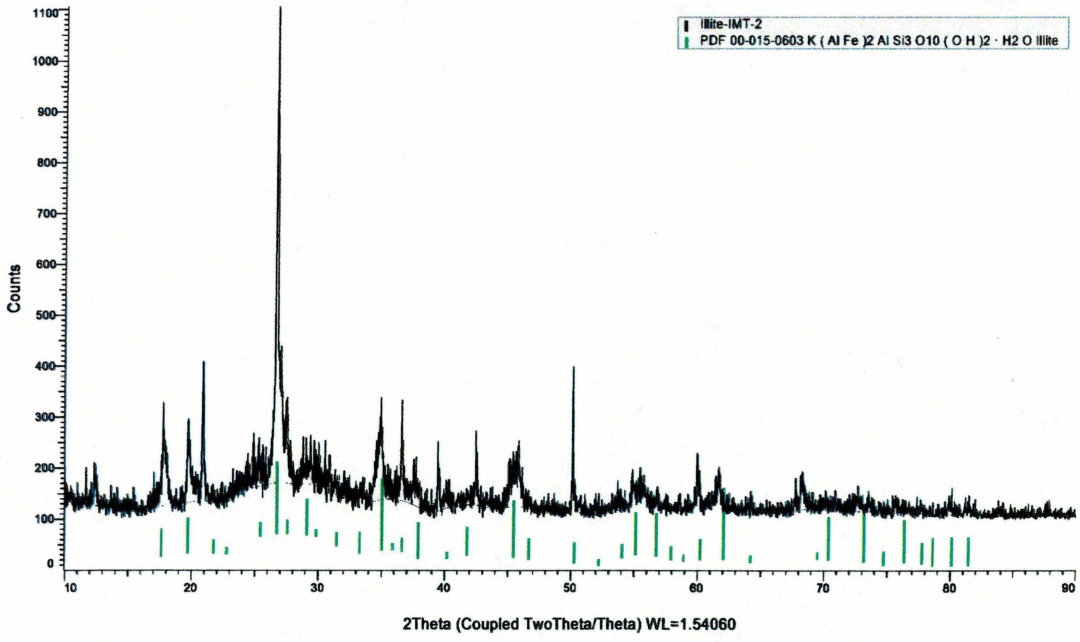


WIPP-Colloids-1 Pg. 33

Figure 2 XRD Analysis of Natural Illite-IMt-2 (Cambrinshale) Ward's Scientific



WIPP-Colloids-1 Pg. 42

Size Distribution Characterization

Size distribution characterization was performed throughout the grinding process for these minerals. The table below summarizes the results to date for particle size distribution for the

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phyllosilicate mineral class. A sufficient amount of colloidal product has not been generated for this mineral class. More grinding is necessary before experiments can begin.

Table 3 Particle Size Distribution Analysis for Phyllosilicate Mineral Class

ID	Grinding Process	*Mohs Hardness Scale	*Particle Refractive Index	D [4,3] (µm)	D [3,2] (µm)	Dv (10) (µm)	Dv (50) (µm)	Dv (90) (µm)	Reference
Illite	Hand-ground	1.5	1.590	103	10.7	3.45	40.7	299	WIPP-Colloids-1 Pg. 85
Illite-IMt-2	Hand-ground	1.5	1.590	54.5	11.4	3.90	32.3	140	WIPP-Colloids-1 Pg. 95
Illite-IMt-2	Wet-Post-Ethanol grind	1.5	1.590	22.2	6.52	2.61	10.9	57.5	WIPP-Colloids-1 Pg. 94
*Values are from Linde 2005.									

3.1.2 Tectosilicate Mineral Class Solid Characterization

Tectosilicates are silica containing minerals that have a three-dimensional framework type structure. This is a very large class of minerals and can be found abundantly in the Earth's crust. The mineral in this study that falls within this class is quartz (SiO_2). A source of Silicon (IV) Oxide was purchased from Fisher Scientific and a naturally occurring source of quartz was purchased from AGSCO. A local naturally occurring source of quartz is in the process of being located.

Purity Characterization

Both sources of quartz were analyzed for purity on the XRD as shown in

Figure 3 and Figure 4. The synthetic quartz shows a typical XRD pattern for SiO_2, with a largely amorphous pattern. Figure 3 shows the presence of an amorphous strong peak at Bragg angle 21°-22°. The natural quartz sample from AGSCO matched with good agreement to natural quartz and showed to be highly pure when compared to PDF card 00-005-0490 in the ICDD.

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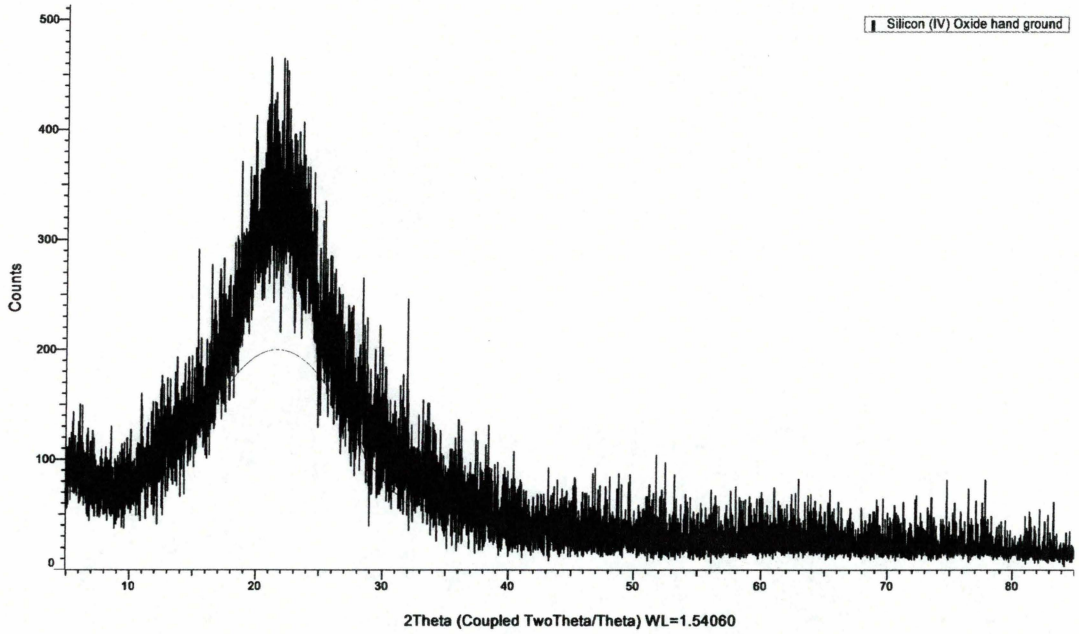
Figure 3 XRD Analysis for Synthetic Silicon (IV) Oxide, Fisher Scientific



WIPP-Colloids-1 Pg. 74

Figure 4 XRD Analysis for Natural Quartz, AGSCO

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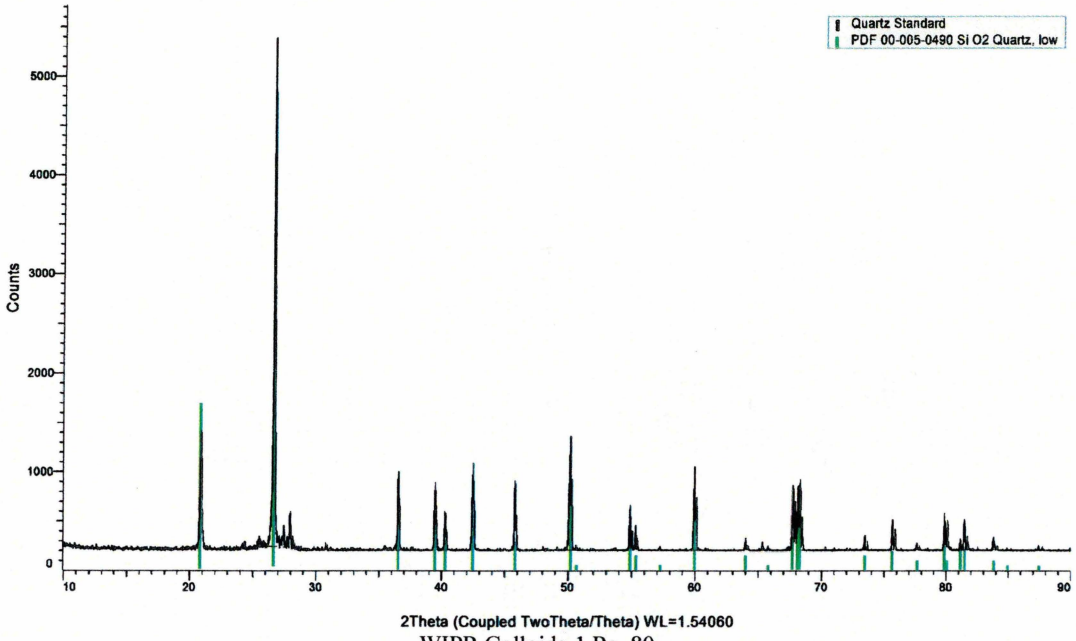

WIPP-Colloids-1 Pg. 80

Size Distribution Characterization

Size distribution characterization was performed throughout the grinding process for these mineral sources. Table 4 below summarizes the results to date. A sufficient number of colloidal particles have not yet been obtained for this mineral and more processing will be necessary before experiments can be started.

Table 4 Particle Size Distribution Analysis for Tectosilicate Mineral Class

ID	Grinding Process	*Mohs Hardness Scale	*Particle Refractive Index	D [4,3] (µm)	D [3,2] (µm)	Dv (10) (µm)	Dv (50) (µm)	Dv (90) (µm)	Reference
Quartz (Silicon IV Oxide)	Hand-ground	7.0	1.553	57.6	17.5	6.13	43.3	133	WIPP-Colloids-1 Pg. 73
Quartz (Silicon IV Oxide)	Dry for 3Hrs.	7.0	1.553	45.7	16.3	5.95	36.7	101	WIPP-Colloids-1 Pg. 75
Quartz (Silicon IV Oxide)	Dry for 6Hrs.	7.0	1.553	46.0	14.5	5.42	31.4	110	WIPP-Colloids-1 Pg. 83
Quartz (Silicon IV Oxide)	Dry for 9Hrs.	7.0	1.553	41.7	14.0	4.97	34.4	91.4	WIPP-Colloids-1 Pg. 78

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Quartz (AGSCO)	Not ground	7.0	1.553	115	59.6	61.0	114	179	WIPP-Colloids-1 Pg. 96
*Values are from Linde 2005.									

3.1.3 Sulfate Mineral Class Solid Characterization

The sulfate mineral class is a class of minerals containing the sulfate anion (SO_4^{2-}). These minerals are prevalent in certain rock formations. Many of these minerals are known to be prevalent in the geology surrounding the WIPP site. The minerals in this study that fall within this class are: anhydrite ($CaSO_4$), gypsum ($CaSO_4 \cdot 2\ H_2O$) and polyhalite ($K_2Ca_2Mg(SO_4)_4 \cdot 2H_2O$). Synthetic anhydrite and gypsum were purchased from Acros Organics. However, naturally occurring sources of these minerals are in the process of being located. A natural source of polyhalite was obtained from Mosaic Potash mine.

Purity Characterization

XRD analyses were conducted on the three minerals in this class. Figure 5 shows the XRD pattern for synthetic anhydrite purchased from Acros Organics. This pattern matches with good agreement to the Anhydrite PDF card 04-011-1764 in the ICDD. Figure 6 shows the XRD pattern for synthetic Gypsum purchased from Acros Organics. This source matches with good agreement to the Gypsum PDF card 00-033-0311 found in the ICDD. Figure 7 shows the XRD pattern for natural polyhalite from Mosaic Potash mine. This sample matches with good agreement with PDF cards 00-021-0982 and 00-010-0355 in the ICDD. Both PDF cards were used to match the polyhalite because the 00-021-0982 card has pattern data to only ~40 ° theta and therefore the second card 00-010-0355 was utilized to match the remaining pattern angles.

Figure 5 XRD Analysis for Synthetic Anhydrite, Acros Organics

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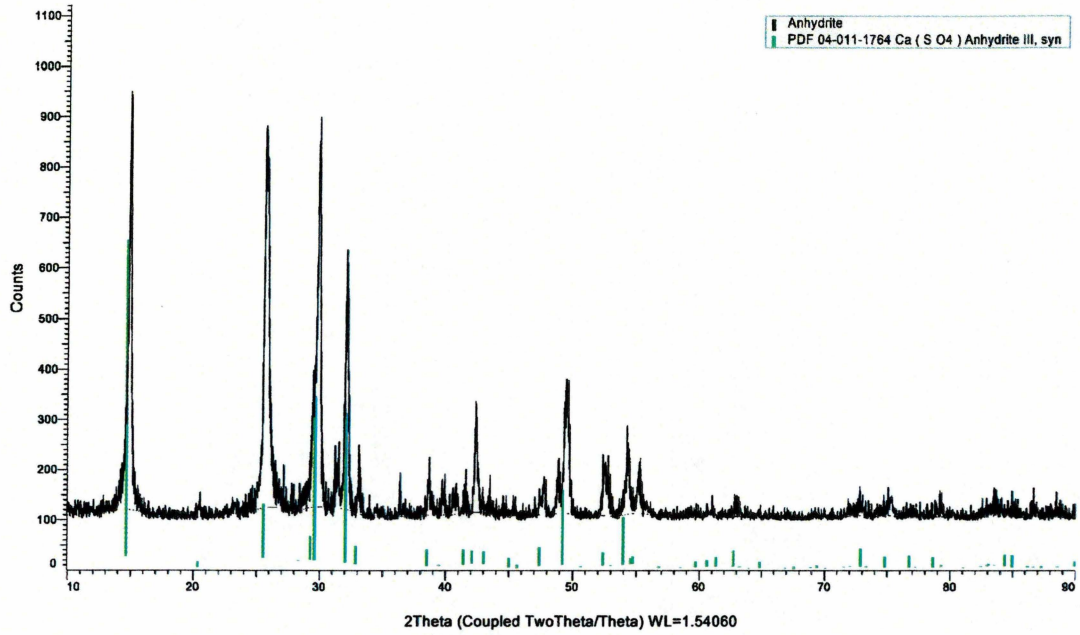
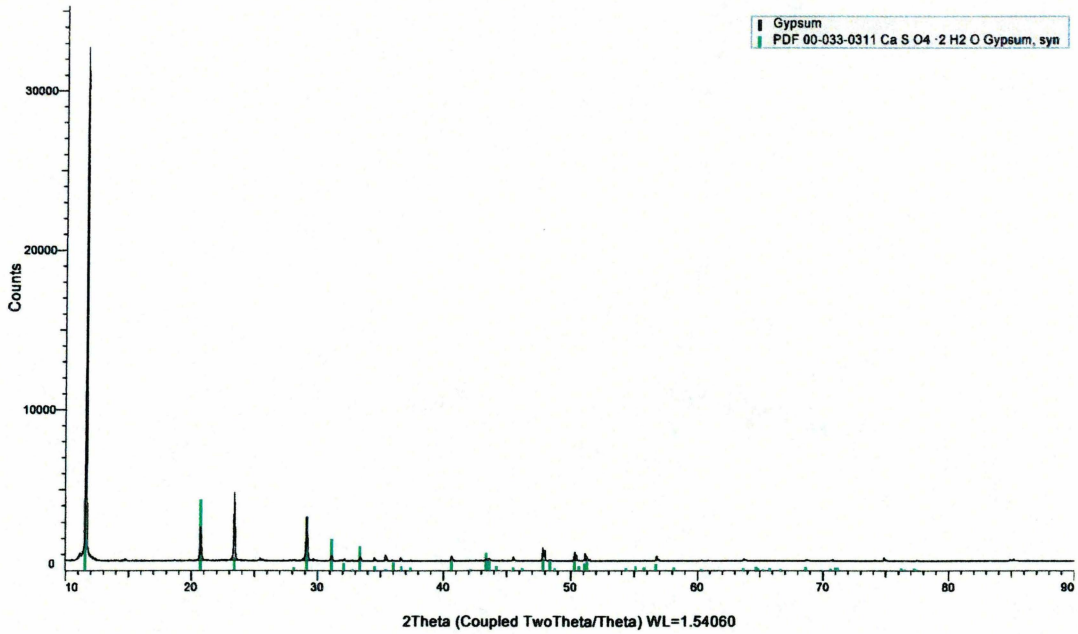


WIPP-Colloids-1 Pg. 41

Figure 6 XRD Analysis for Synthetic Gypsum, Acros Organics



WIPP-Colloids-1 Pg. 40

Figure 7 XRD Analysis for Natural Polyhalite, Mosaic Potash Mine

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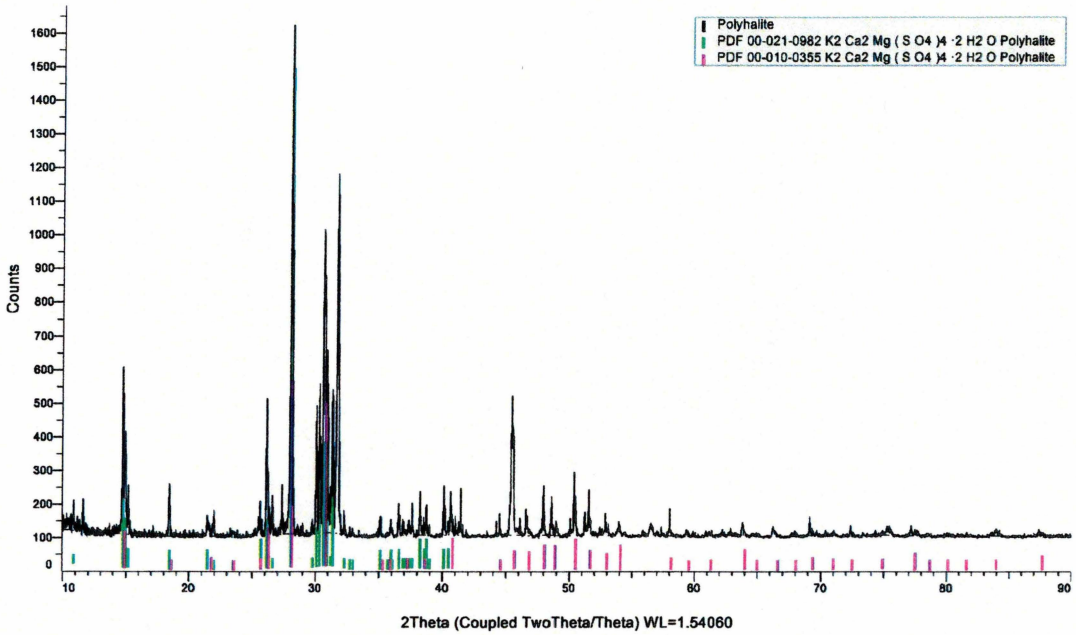

WIPP-Colloids-1 Pg. 37

Size Distribution Characterization

Size distribution characterization was performed throughout the grinding process for these minerals. Table 5 below summarizes the results to date. A sufficient amount of colloidal size mineral has not yet been produced for these minerals. More processing will be necessary before experiments can be initiated.

Table 5 Particle Size Distribution Analysis for Sulfate Mineral Class

ID	Grinding Process	*Mohs Hardness Scale	*Particle Refractive Index	D [4,3] (µm)	D [3,2] (µm)	Dv (10) (µm)	Dv (50) (µm)	Dv (90) (µm)	Reference
Anhydrite	Not Ground	3.5	1.575	126	44.7	30.5	98.2	264	WIPP-Colloids-1 Pg. 97
Anhydrite	Wet-Post Ethanol Grind	3.5	1.575	19.3	4.91	1.92	18.0	43.0	WIPP-Colloids-1 Pg. 90
Gypsum	Not Ground	2.0	1.525	101	48.2	24.3	76.4	210	WIPP-Colloids-1 Pg. 98

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Polyhalite	Hand Ground	2.5-3.5	1.548	162	43.1	16.0	148	333	WIPP-Colloids-1 Pg. 60
Polyhalite	Dry for 3Hr.	2.5-3.5	1.548	43.3	19.7	7.89	38.3	86.7	WIPP-Colloids-1 Pg. 61
Polyhalite	Dry for 6Hr.	2.5-3.5	1.548	38.8	18.2	7.73	33.3	79.0	WIPP-Colloids-1 Pg. 62
Polyhalite	Dry for 9Hr. + hand grind	2.5-3.5	1.548	38.7	17.7	7.16	33.5	79.5	WIPP-Colloids-1 Pg. 64
Polyhalite	Dry for 9Hr. + hand grind w/ethanol	2.5-3.5	1.548	23.6	11.9	5.35	18.5	50.1	WIPP-Colloids-1 Pg. 65
*Anhydrite and Gypsum values are from Linde 2005. Polyhalite values are from: Bladh, K.W. et al 2001.									

3.1.4 Carbonate Mineral Class Solid Characterization

Carbonate minerals are minerals that have the (CO_3^{2-}) anion present. These minerals tend to be very symmetrical in structure. The geology surrounding the WIPP site is known to have some carbonate type rock formations. The minerals in this study that fall within this class are: strontianite ($SrCO_3$), calcite ($CaCO_3$), magnesite ($MgCO_3$), ankerite ($Ca(Fe^{2+},Mg,Mn)(CO_3)_2$ and Dolomite ($CaMg(CO_3)_2$). Synthetic calcite (Fisher), strontianite (Alfa Aesar) and magnesite (Fisher and Acros Organics) have been purchased and have been analyzed for purity. The synthetic source of calcite and strontianite have proven to be pure, however two different sources of magnesite have been purchased (Fisher and Acros Organics) and both sources have proven to be hydromagnesite rather than magnesite. A natural and synthetic source of magnesite are in the process of being located. A source of ankerite is in the process of being located. Two natural sources of Dolomite have been obtained. The first source is from United Materials, a local crushing company, and the second source is a natural source from Alfa Aesar.

Purity Characterization

XRD analyses were completed on the minerals in this class as shown in the following figures. Figure 8 shows the XRD pattern of synthetic strontianite. This pattern is in good agreement with PDF card 00-005-0418 from the ICDD. Figure 9 shows the XRD pattern of synthetic calcite. This XRD pattern matches with good agreement to PDF card 00-005-0586 for Calcite in the

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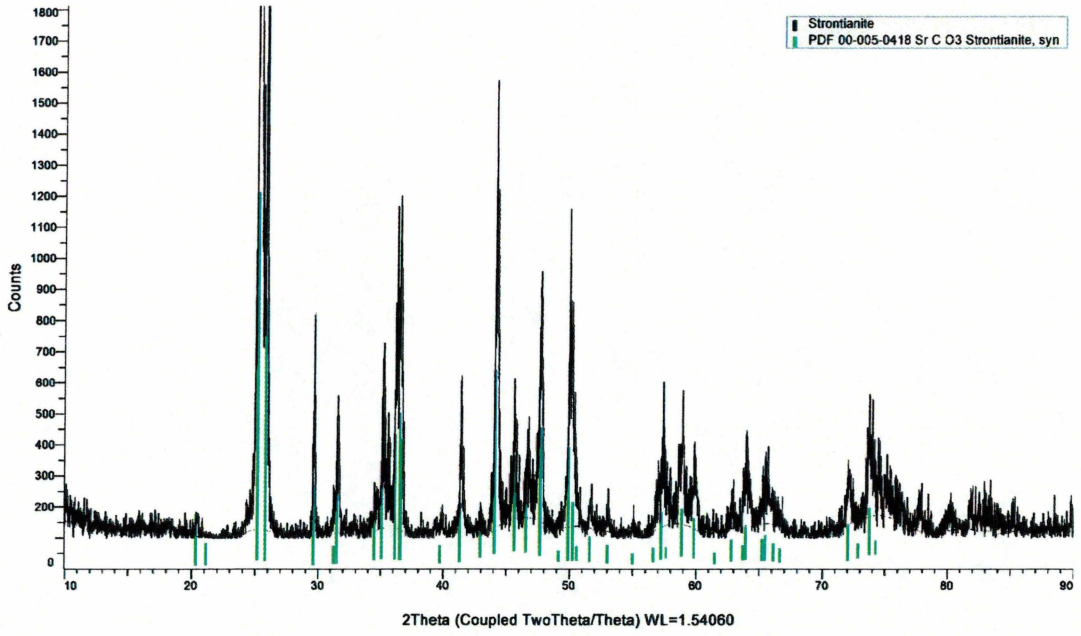
ICDD. Figure 10 shows the XRD patterns of both sources of synthetic magnesite. Neither of these XRD patterns could be matched to magnesite in the ICDD, however both patterns matched with good agreement to PDF card 04-013-7631 for hydromagnesite in the ICDD. Figures 11 and 12 show the XRD patterns of the natural dolomite sources. Both dolomite patterns match with good agreement to PDF card 04-008-0789 in the ICDD. However, the United Materials sample has higher peak intensity at various areas of the pattern, indicating a highly crystalline material. Both dolomite samples are good examples of the mineral.

Figure 8 XRD Analysis for Synthetic Strontianite, Alfa Aesar



WIPP-Colloids-1 Pg. 38

Figure 9 XRD Analysis for Synthetic Calcite, Fisher

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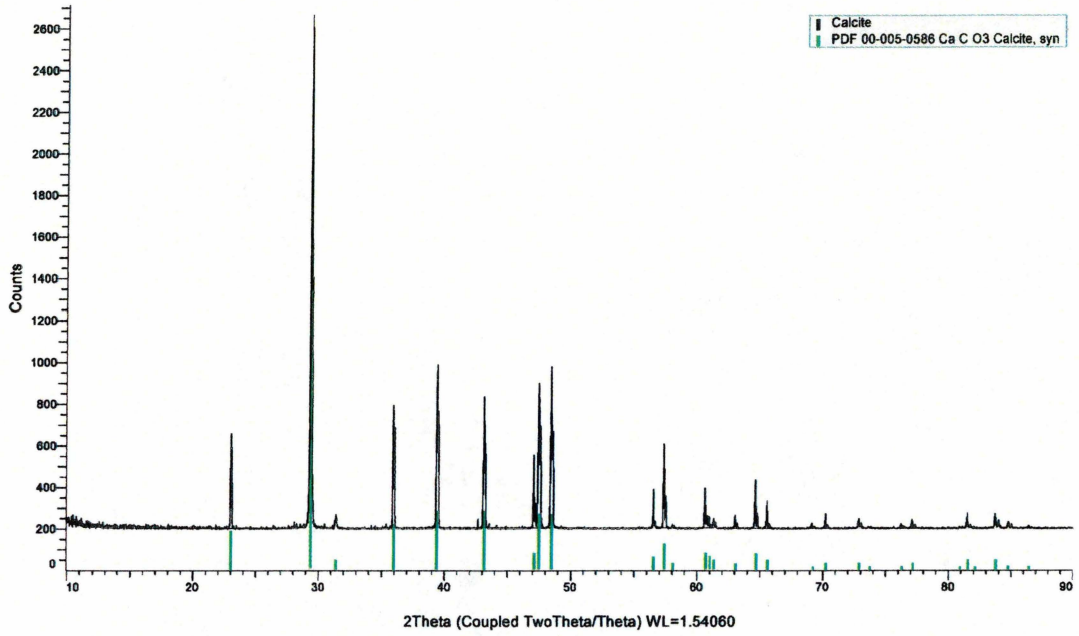
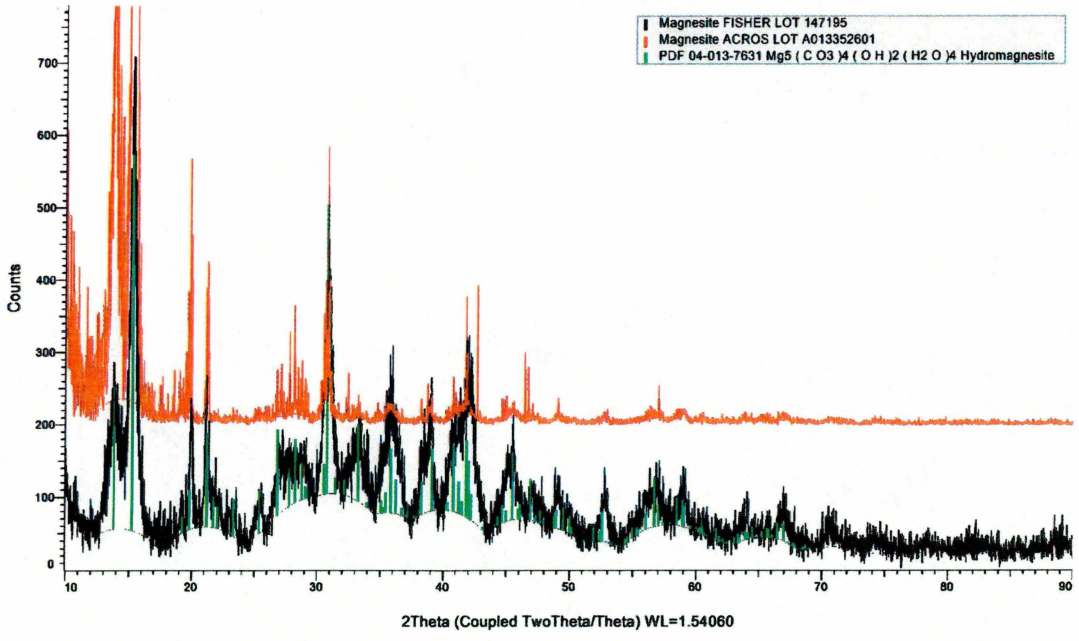


WIPP-Colloids-1 Pg. 35

Figure 10 XRD Analysis for Synthetic Magnesite, Fisher Scientific and Acros Organics



WIPP-Colloids-1 Pg. 79

Figure 11 XRD Analysis for Natural Dolomite, United Materials.

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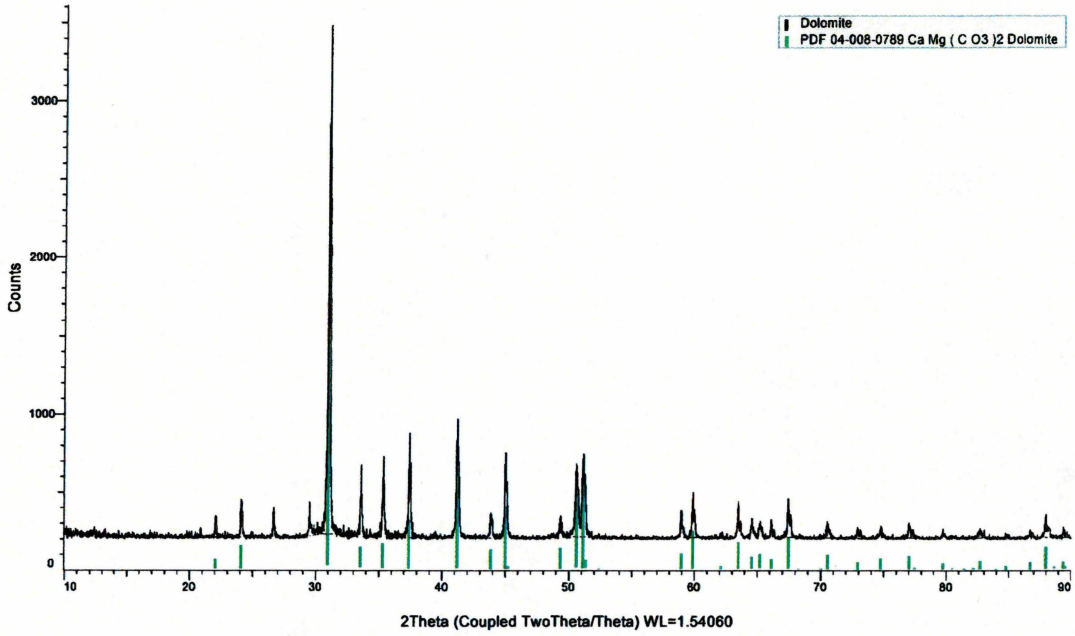
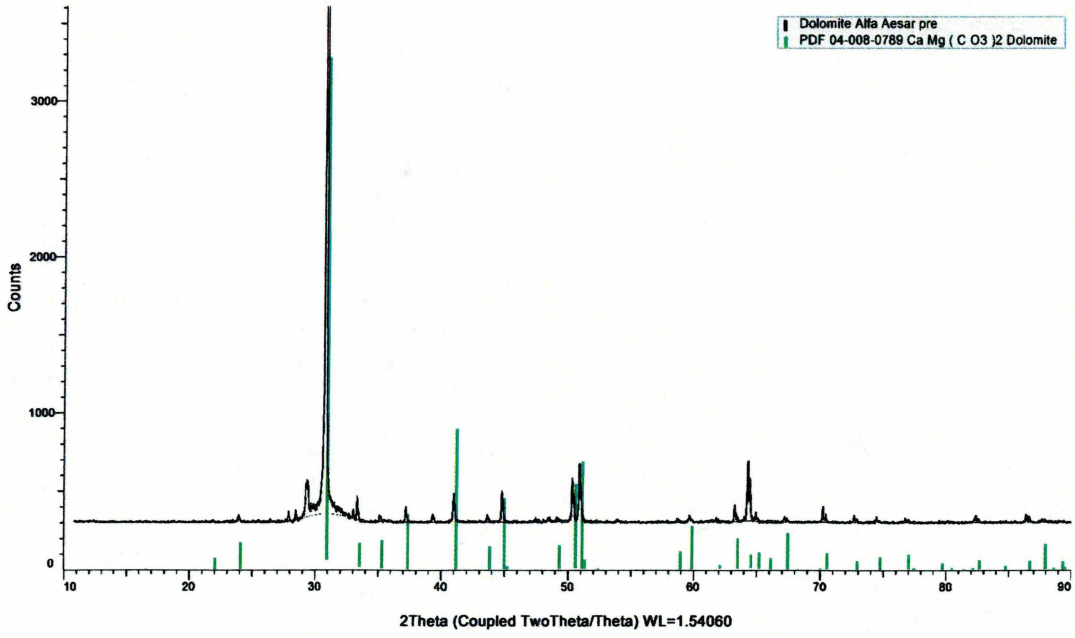


WIPP-Colloids-1 Pg. 72

Figure 12 XRD Analysis For Natural Dolomite, Alfa Aesar.



WIPP-Colloids-1 pg 71

Size Distribution Characterization

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Size distribution characterization was performed throughout the grinding process for these minerals. Results for magnesite are not reported because we have not found a pure source of this mineral to date. Table 6 below summarizes the results to date. Strontianite and Calcite have sufficient amounts of colloidal material for the experiments to be started. Dolomite will continue to be ground until a greater quantity of material is available and a sufficient amount of material is in the colloidal size range.

Table 6 Particle Size Distribution Analysis for Carbonate Mineral Class

ID	Grinding Process	*Mohs Hardness Scale	*Particle Refractive Index	D [4,3] (µm)	D [3,2] (µm)	Dv (10) (µm)	Dv (50) (µm)	Dv (90) (µm)	Reference
Strontianite, Syn	Not Ground	3.5	1.666	5.96	0.178	0.0553	2.59	12.2	WIPP-Colloids-1 Pg. 55
Strontianite, Syn	Dry for 3Hrs.	3.5	1.666	1.12	0.449	0.207	0.563	2.70	WIPP-Colloids-1 Pg. 56
Strontianite, Syn	Dry for 6Hrs.	3.5	1.666	1.88	0.571	0.252	0.735	3.55	WIPP-Colloids-1 Pg. 57
Strontianite, Syn	Dry for 9Hrs.	3.5	1.666	1.43	0.139	0.0538	0.322	2.67	WIPP-Colloids-1 Pg. 58
Calcite, Syn	Not Ground	3.0	1.658	7.15	4.29	2.18	5.72	12.2	WIPP-Colloids-1 Pg. 86
Calcite, Syn	Wet-Post Ethanol Grind	3.0	1.658	2.56	0.152	0.0450	1.61	4.28	WIPP-Colloids-1 Pg. 88
Dolomite, United Materials	Hand Ground	3.5	1.679	72.3	11.6	4.27	30.1	197	WIPP-Colloids-1 Pg. 84
Dolomite, Alfa Aesar	Hand Ground	3.5	1.679	87.6	16.3	5.93	55.8	218	WIPP-Colloids-1 Pg. 81
*Values are from Linde 2005.									

3.2 Task 2: Set-up and Analysis of experiments for Phyllosilicate Mineral Class

The set-up for these experiments has not been started. Solid starting materials are being characterized. Once task 1 of this test plan is complete, this task will be started.

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3.3 Task 3: Set-up and Analysis of experiments for Tectosilicate Mineral Class

The set-up for these experiments has not been started. Solid starting materials are being characterized. Once task 1 of this test plan is complete, this task will be started.

3.4 Task 4: Set-up and Analysis of experiments for Sulfate Mineral Class

The set-up for these experiments has not been started. Solid starting materials are being characterized. Once task 1 of this test plan is complete, this task will be started.

3.5 Task 5: Set-up and Analysis of experiments for Carbonate Mineral Class

The set-up for these experiments has not been started. Solid starting materials are being characterized. Once task 1 of this test plan is complete, this task will be started.

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4 ACKNOWLEDGEMENTS

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