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Milestone Report on Test Plan TP 08-02, "Iron, Lead, Sulfide, and EDTA Solubilities"

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DEFINITION OF ABREVIATIONS, ACRONYMS AND INITIALISMS

Abbreviation or Acronym	Definition
[X]	concentration of component X
A	pH correction factor
ACS	American Chemical Society
AP	Analysis Plan
aq	aqueous (phase identifier)
Cit^{-3}	citrate anion, $C_6H_5O_7^{-3}$, conjugate base of citric acid
$CaCit^{-}$	calcium citrate aqueous complex ion
Cl^{-}	chloride anion
CO_3^{2-}	carbonate anion
DI	de-ionized
DDI	deoxygenated de-ionized
$EDTA^{-4}$	ethylenediaminetetraacetate anion, $C_{10}H_{12}N_2O_8^{-4}$
ES&H	Environmental Safety and Health
Fe^{2+}	ferrous iron aqueous ion
Fe(II)	total ferrous iron
Fe(III)	total ferric iron
$FeCit^{-}$	ferrous iron citrate aqueous complex ion
$FeOH^{+}$	ferrous iron hydroxide aqueous complex ion
$Fe(OH)_3^{-}$	ferrous iron trihydroxide aqueous complex ion
$FeEDTA^{-2}$	ferrous iron EDTA aqueous complex ion
FeOx(aq)	ferrous iron oxalate aqueous complex ion
Fe_T	total iron, i.e., Fe(II) + Fe(III)
FeS	iron sulfide
g	gram, or gas (phase identifier)
H_2	hydrogen molecule

HCO_3^-	bicarbonate anion
$HEDTA^{3-}$	hydrogen EDTA aqueous complex ion
HS^-	hydrogen sulfide anion
HCl	hydrochloric acid
H_2S	hydrogen sulfide
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectroscopy
m	molal (mol/kg)
M	molar (mol/L)
Mg^{2+}	magnesium aqueous ion
Na^+	sodium aqueous species
Ox^{-2}	oxalate anion, $C_2O_4^{-2}$, conjugate base of oxalic acid
Pb_T	total lead
PbOx(aq)	lead oxalate aqueous complex ion
$PbEDTA^{2-}$	lead EDTA aqueous complex ion
$PbCl^+$	lead chloride aqueous complex ion
$PbCit^-$	lead citrate aqueous complex ion
pH	negative logarithm of hydrogen ion activity
PI	Principle Investigator
QA	Quality Assurance
s	solid (phase identifier)
SNL	Sandia National Laboratories
SNL/DWMP	Sandia National Laboratories Defense Waste Management Programs
SP	Specific Procedure
SO_4^{2-}	sulfate anion
TP	Test Plan
WIPP	Waste Isolation Pilot Plant
XRD	X-ray diffraction

1 INTRODUCTION

The purposes of this milestone report are to (1) summarize experimental data obtained to date under Test Plan TP 08-02, "Iron, Lead, Sulfide, and EDTA Solubilities" (Ismail et al., 2008), and (2) determine future activities needed to complete the work scope defined in Test Plan TP 08-02 by tabulating the results to date. The data provided in this report addresses the solubility of WIPP-relevant non-actinide solids and the Pitzer ion-interaction parameters for the aqueous species associated with those solids. An Analysis Plan (AP) for data in this report is under development. Test Plan TP 08-02 supports the Waste Isolation Pilot Plant's (WIPP's) geochemical model used to estimate the solubilities of major actinide species present in the repository.

In Test Plan TP 08-02, the systems containing Fe(II)- and Pb(II)-bearing species shown in Table 1-1 were identified for investigation in the initial phase of the test plan. The test plan allows for additional investigations as warranted by examining the results from the initial test phase.

Table 1-1 Ion-pairs identified for investigation in Test Plan TP 08-02[a].

1.	$FeOH^+$—$SO4^{2-}$	15.	$PbCl^+$—$CO3^{2-}$
2.	$FeOH^+$—$CO3^{2-}$	16.	$PbCl^+$—$SO4^{2-}$
3*.	$FeOH^+$—HS^-	17*.	$PbCl^+$—HS^-
4*.	Fe^{2+}—HS^-	18.	Na^+—$PbEDTA^{2-}$
5.	Na^+—$Fe(OH)_3^-$	19.	Mg^{2+}—$PbEDTA^{2-}$
6.	Mg^{2+}—$Fe(OH)_3^-$	20.	Na^+—$PbCit^-$
7.	FeOx(aq)—Na^+	21.	Mg^{2+}—$PbCit^-$
8.	FeOx(aq)—Mg^{2+}	22.	PbOx(aq)—Na^+
9.	FeOx(aq)—Cl^-	23.	PbOx(aq)—Mg^{2+}
10.	Na^+—$FeEDTA^{2-}$	24.	PbOx(aq)—Cl^-
11.	Mg^{2+}—$FeEDTA^{2-}$	25*.	Na^+—HS^-
12.	Na^+—$FeCit^-$	26*.	Mg^{2+}—HS^-
13.	Mg^{2+}—$FeCit^-$	27*.	Cl^-—HS^-
14.	$PbCl^+$—$HCO3^-$		

[a] Based on Table 2 of TP 08-02 (Ismail et al., 2008)

Table 1-1 identifies twenty-seven systems for investigation, which are currently underway, except those marked with asterisks. Many of the experiments have been running for

periods exceeding one year. Once a system has come to equilibrium, based on the stability of measured concentration(s) of selected aqueous component(s) over time, final measurements of the aqueous component(s) of interest can be made and the thermodynamic and Pitzer parameters of interest can be derived. Investigations of the systems marked with asterisks in Table 1-1 have not yet begun (See section 2.6).

Four additional systems have been identified for investigation based on the results from the initial test phase. These systems are listed in Table 1-2, and were selected due to their WIPP-relevance and lack of data in the literature.

Table 1-2 Ion-pairs and/or triplets added for investigation in the second phase of Test Plan TP 08-02.

28.	$FeOx(aq)—Na^{+}—Mg^{2+}$	30.	$Na^{+}—HEDTA^{3-}$
29.	$Na^{+}—CaCit^{-}$	31.	$Mg^{2+}—HEDTA^{3-}$

2 MATERIALS AND METHODS

Solubility-controlling solids of interest were either chemicals that are certified as ACS reagent grade or better, or synthesized and characterized in the laboratory using XRD when ACS reagents are not available. Solutions were prepared using appropriate reagent grade commercial salts.

2.1 Anoxic Conditions

Experiments involving Fe(II) were strictly performed inside anoxic gloveboxes. Samples containing dissolved Fe(II) were preserved in dilute HCl inside the gloveboxes prior to further handling under normal air to limit the oxidation rate of Fe(II).

2.2 Measurement of Dissolved Fe(II) and Dissolved Total Fe

Ferrozine assay was used to measure the concentrations of dissolved Fe(II) ($[Fe(II)]_{diss}$) and dissolved total Fe ($[Fe_T]_{diss} = [Fe(II)]_{diss} + [Fe(III)]_{diss}$). Details on ferrozine assay are in WIPP-Solubility-4 Supplemental Binder-1, WIPP-Solubility-11 Supplemental Binder-1, and page 77 of WIPP-Solubility-9. UV-Visible spectrophotomer was used to read the absorbance at 562 nm as per SP 12-25. ICP-AES was sometimes used to measure $[Fe_T]_{diss}$, when it was desirable (See section 2.4 for cation analysis using ICP-AES).

2.3 Anion Measurements

IC was used for anion analysis per SP 12-22.

2.4 Cation Measurements

ICP-AES was used to measure the concentrations of cations, including $[Fe_T]_{diss}$ and $[Pb_T]_{diss}$ per SP 12-9.

2.5 pH Measurements

Solution pH was measured using commercial combination electrodes and meters per SP 12-14.

2.6 Experiments involving H_2S

Items 3, 4, 17, and 25-27 in Table 1-1, which are solubility experiments involving the hydrogen sulfide ion (HS^-) in conjunction with Fe(II) and Pb(II), have not been initiated yet. Because of Environmental Safety and Health (ES&H) concerns regarding the use of sulfide-containing compounds, investigation of the systems requiring the use of HS^- was delayed until all ES&H concerns could be addressed.

Recently, the ES&H requirements for safe handling of hydrogen sulfide were fulfilled. The ES&H requirements include: (1) the use of a dedicated glovebox with its own ventilation, (2) the use of appropriate personal protective equipment, and (3) the use of appropriate atmospheric monitors. A "Readiness Review" was performed as a last step in the ES&H process. Procedures and requirements for the safe handling of hydrogen sulfide within the SNL/DWMP laboratory have been documented in: National Environmental Policy Act (NEPA) documents (SNA10-0048), Primary Hazard Screening (PHS) document (SNL09A00320-001), and a Job Safety Analysis (JSA). A Technical Work Document (TWD) was prepared and presented at the time of the "Readiness Review".

A type of iron sulfide (presumably "FeS") is under preparation in the dedicated glovebox. The required duration for these experiments is uncertain. Because of the low solubility of sulfide-bearing minerals in general, the sulfide experiments may require substantially more time to reach equilibrium than many of the other experiments listed in Table 1-1. Careful monitoring of system component(s), such as, pH and concentration of selected metal(s), may be required to establish that equilibrium conditions exist.

3 RESULTS

Following sections describe the preparation of the experimental set-ups and results to date for ion-pairs listed in Table 1-1 and Table 1-2 in the order therein. Each section has two types of tables; one to describe the experimental set-ups and the other to tabulate results to date. When single table frame does not provide proper summary of experimental set-ups and/or results to date, multiple tables were used under same table identifier followed by "(continued)". Refer to Table 3-1 and Table 3-2 in section 3.1 for an example. For those experiments which were not initiated, a short description of the experiments and two empty tables were provided; one for description of experimental set-ups, the other for measurement of all components involved in the experimental set-ups. One or two of the listed components will be monitored over time to see if the system approaches a steady-state so that we can assume equilibrium. Though all components are listed, it is each PI's decision whether to proceed with all the measurements or to select a few relevant components. For example, to prepare NaCl solutions, the mass of NaCl is weighed, and the amount of DI water is determined either gravimetrically (for molality) or volumetrically (for molarity). This procedure determines the concentrations of sodium and chloride which are significantly more accurate than common laboratory instruments can provide, such as ICP-AES or IC. It might be wiser to analyze a few, not every single, NaCl solution as check samples. However, due to the hygroscopic nature of commercial salt $MgCl_2 \cdot 6H_2O$, it is recommended that the PIs frequently check the Mg concentrations in $MgCl_2$ solutions when the solutions are prepared using aged $MgCl_2 \cdot 6H_2O$.

3.1 Solubility of $Fe_2(OH)_3Cl(s)$ and $Fe(OH)_2(s)$ in mixed NaCl and Na_2SO_4 solutions (the "$FeOH^+$—SO_4^{2-}" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $Fe_2(OH)_3Cl(s)$ (iron hibbingite) and $Fe(OH)_2(s)$ (ferrous iron hydroxide) in mixed NaCl (sodium chloride) and Na_2SO_4 (sodium sulfate) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "$FeOH^+$—SO_4^{2-}" (Item 1, Table 1-1).

The experimental set-ups including initial brine molalities and mass of solid materials are shown below in Table 3-1. The solid phases, $Fe_2(OH)_3Cl(s)$ and $Fe(OH)_2(s)$, were synthesized from $FeCl_2 \cdot 4H_2O$ (Fisher) and KOH (Fisher). Brines were prepared from DI water, NaCl (Fisher), and Na_2SO_4 (Fisher).

Over a period of several months, the solid phase in the first six experimental set-ups defined in Table 3-1 reduced water in the brine and formed green rust sulfate. Thus, new experimental set-ups (later six set-ups) were prepared with an $Fe(OH)_2$ solid phase and a brine containing NaCl in addition to Na_2SO_4. The new formulation seems to have prevented (or greatly reduced) the rate of water reduction.

The experimental set-ups are periodically monitored for pH and dissolved Fe(II) concentrations($[Fe(II)]_{diss}$). Once stable pH readings are achieved, concentrations of other dissolved components (sodium, sulfate, and chloride) are to be determined. Experimental results gathered to date are shown in Table 3-2.

Table 3-1 Preparation of experimental set-ups for the solubility of $Fe_2(OH)_3Cl(s)$ in Na_2SO_4 solutions, and the solubility of $Fe(OH)_2(s)$ in mixed Na_2SO_4 + NaCl solutions (the "$FeOH^+$—SO_4^{2-}" experiment).

Set-up ID	$Fe_2(OH)_3Cl(s)$ (g)	$Fe(OH)_2$ (g)	Na_2SO_4 (m)	NaCl (m)	References
0.05-SO4-Fe(OH)2-(1,2)	0.7	-	0.05	-	WIPP-Solubility-1, pg. 15-19, 96-97
0.38-SO4-Fe(OH)2-(1,2)	0.7	-	0.38	-	
0.76-SO4-Fe(OH)2-(1,2)	0.7	-	0.76	-	
1.14-SO4-Fe(OH)2-(1,2)	0.7	-	1.19	-	
1.52-SO4-Fe(OH)2-(1,2)	0.7	-	1.52	-	
1.9-SO4-Fe(OH)2-(1,2)	0.7	-	1.9	-	
0.01m Na2SO4+0.15NaCl-GR	-	2.05	0.01	0.15	WIPP-Solubility-1, pg. 91
0.1m Na2SO4+0.15NaCl-GR	-	2.02	0.1	0.15	
0.5m Na2SO4+0.15NaCl-GR	-	2.04	0.5	0.15	
1m Na2SO4+0.15NaCl-GR	-	2.02	1.0	0.15	
1.5m Na2SO4+0.15NaCl-GR	-	2.02	1.5	0.15	
1.8m Na2SO4+0.15NaCl-GR	-	2.03	1.8	0.15	

Table 3-2 Measured data for the solubility of $Fe_2(OH)_3Cl(s)$ in mixed Na_2SO_4 + NaCl solutions (the "$FeOH^+$—SO_4^{2-}" experiment).

Set-up ID	pH[a] (84 days)	pH[b1,b2] (133 days)	$[Fe_T]$ (mol/L)[c] (90 days)	$[Fe_T]$ (mol/L)[d] (133 days)	[Fe(II)] (mol/L)[e] (206 days)	$[SO_4^{2-}]$ (mol/L)[f] (311 days)
0.05-SO4-Fe(OH)2-1	8.126	-	6.27E-03	-	-	-
0.38-SO4-Fe(OH)2-1	8.307	-	8.58E-03	-	-	-
0.76-SO4-Fe(OH)2-1	8.361	-	8.90E-03	-	-	-
1.14-SO4-Fe(OH)2-1	8.507	-	6.21E-03	-	-	-
1.52-SO4-Fe(OH)2-1	8.400	-	8.31E-03	-	-	-
1.9-SO4-Fe(OH)2-1	8.521	-	5.36E-03	-	-	-
0.05-SO4-Fe(OH)2-2	-	7.908[b1]	-	9.88E-03	1.16E-02	4.05E-02
0.38-SO4-Fe(OH)2-2	-	8.158[b1]	-	1.16E-02	1.25E-02	3.99E-01[fl]
0.76-SO4-Fe(OH)2-2	-	8.265[b1]	-	1.13E-02	1.34E-02	8.02E-01
1.14-SO4-Fe(OH)2-2	-	8.357[b1]	-	9.61E-03	1.10E-02	1.82E+00
1.52-SO4-Fe(OH)2-2	-	8.372[b2]	-	1.18E-02	1.39E-02	1.79E+00
1.90-SO4-Fe(OH)2-2	-	8.471[b2]	-	1.02E-02	1.46E-02	2.05E+00

[a] Measured with pH electrode and meter; WIPP-Solubility-1, pg. 66
[b1,b2] Measured with pH electrode and meter; WIPP-Solubility-4, pg. 38 (133 days)[b1], and pg.40 (134 days)[b2]
[c] measured using ICP; WIPP-Solubility-1, pg.71-72, and 7/21/08 tab of WIPP-Solubility-3 Binder A
[d] measured using ICP; WIPP-Solubility-4, pg. 34-46 and 9/8/08 tab of WIPP-Solubility-3 Binder A
[e] measured using the Ferrozine method; WIPP-Solubility-6, pg.14-15, and 1/9/09 tab of WIPP-Solubility-6 Binder 1
[f] measured using IC; WIPP-Solubility-6, p.62-63; WIPP-Solubility-6 Supplemental Binder 1, Tab 3/4/09
[fl] measured using IC; WIPP-Solubility-6, p.64; WIPP-Solubility-6 Supplemental Binder 1, Tab 3/9/09

Table 3-2(continued) Measured data for the solubility of $Fe(OH)_2(s)$ in mixed Na_2SO_4 + NaCl solutions (the "$FeOH^+$—SO_4^{2-}" experiment).

Set-up ID	pH (55 days)[a]	pH (86 days)[b]	[Fe(II)] (mol/L) (55 days)[c]	[Fe(II)] (mol/L) (85 days)[d]
0.01m Na2SO4 + 0.15 NaCl –GR	8.110	7.943	1.57E-03	1.81E-03
0.1m Na2SO4 + 0.15 NaCl –GR	8.354	8.369	1.18E-03	1.41E-03
0.5m Na2SO4 + 0.15 NaCl –GR	8.603	8.634	9.80E-04	1.22E-03
1 m Na2SO4 + 0.15 NaCl –GR	8.730	8.767	9.17E-04	1.01E-03
1.5 m Na2SO4 + 0.15 NaCl –GR	8.810	8.834	7.14E-04	9.99E-04
1.8 m Na2SO4 + 0.15 NaCl –GR	8.773	8.817	6.72E-04	7.84E-04

[a] Measured with pH electrode and meter; WIPP-Solubility-6, pg. 50

[b] Measured with pH electrode and meter; WIPP-Solubility-6, pg. 66

[c] Measured using the Ferrozine method; WIPP-Solubility-6, pg. 51-55, and 2/18/09 tab of WIPP-Solubility-6 Binder 1

[d] Measured using the Ferrozine method; WIPP-Solubility-6, pg. 70, and 4/10/09 tab of WIPP-Solubility-6 Binder I

3.2 Solubility of $FeCO_3(s)$ in mixed Na_2CO_3 and NaCl solutions (the "$FeOH^+$—CO_3^{2-}" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $FeCO_3(s)$ (siderite) in mixed Na_2CO_3 (sodium carbonate) and NaCl (sodium chloride) solutions, and (2) determine the Pitzer ion-interaction parameters for the ion pair "$FeOH^+$—CO_3^{2-}" (Item 2, Table 1-1). Experimental set-ups are listed in Table 3-3, and they will be periodically monitored for one or more component(s) among those listed in Table 3-4. Once stable readings are achieved for the selected component(s), concentrations of other components will be determined.

Table 3-3 Preparation of experimental set-ups for the solubility of $FeCO_3(s)$ in mixed Na_2CO_3 and NaCl solutions (the "$FeOH^+$—CO_3^{2-}" experiment).

Set-up ID	$FeCO_3(s)$ (g)	NaCl (m)	Na_2CO_3 (m)	Reference
FeCO3-0.01CO3-I	0.5[#]	0.15	0.01	CAR W-09-05, and notebooks cited therein[*]
FeCO3-0.1CO3-1	0.5[#]	0.15	0.1	CAR W-09-05, and notebooks cited therein[*]
FeCO3-0.5CO3-1	0.5[#]	0.15	0.5	CAR W-09-05, and notebooks cited therein[*]
FeCO3-1.0CO3-I	0.5[#]	0.15	1.0	CAR W-09-05, and notebooks cited therein[*]
FeCO3-1.5CO3-1	0.5[#]	0.15	1.5	CAR W-09-05, and notebooks cited therein[*]
FeCO3-2.0CO3-1	0.5[#]	0.15	2.0	CAR W-09-05, and notebooks cited therein[*]

[#] Approximately 0.5g based on visual appearance. Exact solid mass is not critical in solubility experiments.
[*] Those pages are p.31 and p.97 of WIPP-Solubility-1 for $FeCO_3(s)$ preparation and XRD, p.21-22 of WIPP-Solubility-3 for solution preparation. On p.70-77 of WIPP-Solubility-4, one of the PIs mentioned Fe(II) measurement for the samples above by the Ferrozine method, but data are not traceable in the associated binder therein (WIPP-Solubility-4 supplemental binder-1 tab 11/11/08).

Table 3-4 Measured data for the solubility of $FeCO_3(s)$ in mixed Na_2CO_3 and NaCl solutions (the "$FeOH^+$—CO_3^{2-}" experiment).

Set-up ID	$[Fe(II)]_{diss}$	$[CO_3^{2-}{}_T]_{diss}$	[Na(I)]diss	[Cl(-I)]diss	pH
Reference	No Data to Date				

3.3 Solubility of an iron sulfide in mixed NaHS and Na_2S solutions (the "$FeOH^+$—HS^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of an iron sulfide in mixed NaHS (sodium bisulfide) and Na_2S (sodium sulfide) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "$FeOH^+$—HS^-" (Item 3, Table 1-1). The solid phase is under preparation in an anoxic glovebox dedicated for sulfide experiments. The solubility experiments will start after characterization is completed for the iron sulfide being prepared. Experimental set-ups will be listed in Table 3-5, and they will be periodically monitored for one or more component(s) among those listed in Table 3-6. Once stable readings are achieved for the selected component(s), concentrations of other components will be determined.

Table 3-5 Preparation of experimental set-ups for the solubility of an iron sulfide in mixed NaHS and Na_2S solutions (the "$FeOH^+$—HS^-" experiment).

Set-up ID	Iron sulfide (g)	NaHS (m)	Na_2S (m)	Reference
No Data to Date				

Table 3-6 Measured data for the solubility of an iron sulfide in mixed NaHS and Na_2S solutions (the "$FeOH^+$—HS^-" experiment).

Set-up ID	$[Fe(II)]_{diss}$	$[S(-II)]_{diss}$	[Na(I)]diss	pH
Reference	No Data to Date			

3.4 Solubility of an iron sulfide in Na_2S solutions (the "Fe^{2+}—HS^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of an iron sulfide in Na_2S (sodium sulfide) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "Fe^{2+}—HS^-" (Item 4, Table 1-1). This experiment will be performed at lower pH values than the experiments described in section 3.3. Experimental set-ups will be listed in Table 3-7, and they will be periodically monitored for one or more component(s) among those listed in Table 3-8. Once stable readings are achieved for the selected component(s), concentrations of other components will be determined.

Table 3-7 Preparation of experimental set-ups for the solubility of an iron sulfide in Na_2S solutions (the "Fe^{2+}—HS^-" experiment).

Set-up ID	Iron sulfide (g)	Na_2S (m)	Reference
No Data to Date			

Table 3-8 Measured data for the solubility of an iron sulfide in Na_2S solutions (the "Fe^{2+}—HS^-" experiment).

Set-up ID	[Fe(II)]	[S(-II)]	[Na(I)]	pH
Reference	No Data to Date			

3.5 Solubility of $Fe_2(OH)_3Cl(s)$ and $Fe(OH)_2(s)$ in NaCl solutions (the "Na^+—$Fe(OH)_3^-$" experiment).

The objectives of this set of experiments are to (1) determine the solubility of $Fe_2(OH)_3Cl(s)$ (iron hibbingite), $FeCl_2{\cdot}4H_2O$(ferrous chloride tetrahydrate) and $Fe(OH)_2(s)$ (ferrous iron hydroxide) in NaCl (sodium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "Na^+—$Fe(OH)_3^-$" (Item 5, Table 1-1).

The experimental set-ups are shown in Table 3-9. These have been periodically monitored for pH and dissolved Fe(II) concentrations ($[Fe(II)]_{diss}$). Once stable pH readings are achieved, concentrations of other dissolved components (sodium and chloride) are to be determined. Experimental results gathered to date are shown in Table 3-10.

Table 3-9 Preparation of experimental set-ups for the solubility of $Fe_2(OH)_3Cl(s)$ and $Fe(OH)_2(s)$ in approximately 50 ml of NaCl solutions (the "Na^+—$Fe(OH)_3^-$" experiment).

Set-up ID	$Fe_2(OH)_3Cl(s)$ (g)	NaCl (m)	NaOH (m)	References
0.1-Cl-Fe(OH)2-(1,2)	0.7	0.1	-	WIPP-Solubility-1 pg.15-17
1-Cl-Fe(OH)2-(1,2)	0.7	1.0	-	
2-Cl-Fe(OH)2-(1,2)	0.7	2.0	-	
3-Cl-Fe(OH)2-(1,2)	0.7	3.0	-	
4-Cl-Fe(OH)2-(1,2)	0.7	4.0	-	
5-Cl-Fe(OH)2-(1,2)	0.7	5.0	-	
Set-up ID	$FeCl_2{\cdot}4H_2O(s)$ (g)	NaCl (m)	NaOH (m)	References
1m-Cl-Fe(OH)2-O-1	0.7207	1.0	1.0[a]	WIPP-Solubility-1 pg.82-86
1m-Cl-Fe(OH)2-O-2	0.5104	1.0	1.0[b]	
5m-Cl-Fe(OH)2-O-1	0.5629	5.0	1.0[c]	
5m-Cl-Fe(OH)2-O-2	0.5486	5.0	1.0[d]	
Set-up ID	$Fe(OH)_2(s)$ (m)	NaCl (m)	NaOH (m)	References
SQDLTN+0	0.2176	0.0363	-	WIPP-Solubility-5, pg.73
SQDLTN+1	0.2176	0.152	-	
SQDLTN+2	0.2176	0.504	-	
SQDLTN+3	0.2176	1.22	-	
SQDLTN+4	0.2176	3.03	-	
SQDLTN+5	0.2176	5.97	-	

[a] 1m NaOH was added too much, and pH reached nearly 11
[b] 1m NaOH was added until pH reached 10.6
[c] 1m NaOH was added until pH reached 9.3
[d] 1m NaOH was added until pH reached 10.7

Table 3-10 Measured data for the solubility of $Fe_2(OH)_3Cl(s)$ in NaCl solutions (the "Na^+—$Fe(OH)_3^-$" experiment).

Set-up ID	pH[a0] (139 days)	pH[a] (273 days)	pH[b] (382 days)	pH[c] ([a,b,c] days)	[Fe(II)] (mol/L)[d] (277 days)	[Fe(II)] (mol/L)[e] (371 days)	[Fe(II)] (mol/L)[f] (439 days)
0.1-Cl-Fe(OH)2-1	-	-	8.24	8.29[c1]	-	1.5E-3	1.4E-3
1-Cl-Fe(OH)2-1	-	-	8.61	8.55[c1]	-	2.4E-4	2.5E-4
2-Cl-Fe(OH)2-1	-	-	8.59	8.51[c1]	-	1.5E-4	1.6E-4
3-Cl-Fe(OH)2-1	-	-	8.55	8.43[c2]	-	1.45E-4	1.1E-4
4-Cl-Fe(OH)2-1	-	-	8.48	8.35[c2]	-	6.4E-5	7.2E-5
5-Cl-Fe(OH)2-1	-	-	8.28	8.16[c3]	-	8.0E-5	1.0E-4
0.1-Cl-Fe(OH)2-2	8.40	8.26	-	8.21[c1]	1.3E-3	-	1.9E-3
1-Cl-Fe(OH)2-2	8.51	8.55	-	8.53[c1]	2.6E-4	-	2.5E-4
2-Cl-Fe(OH)2-2	8.52	8.54	-	8.51[c1]	1.4E-4	-	1.6E-4
3-Cl-Fe(OH)2-2	8.46	8.43	-	8.34[c2]	1.1E-4	-	1.3E-4
4-Cl-Fe(OH)2-2	8.39	8.34	-	8.29[c2]	8.2E-5	-	1.0E-4
5-Cl-Fe(OH)2-2	8.31	8.22	-	8.17[c3]	7.1E-5	-	8.2E-5

[a0] Measured with pH electrode and meter; WIPP-Solubility-4 pg. 38-42
[a] Measured with pH electrode and meter; WIPP-Solubility-6 pg. 24-27
[b] Measured with pH electrode and meter; WIPP-Solubility-8 pg.15-16
[c] Measured with pH electrode and meter; WIPP-Solubility-8 pg. 28-31
[c1] 428 days; [c2] 432 days; [c3] 435 days
[d] Measured using the Ferrozine method; WIPP-Solubility-6 pg. 30-32; 1/19/09 tab of WIPP-Solubility-6 binder A
[e] Measured using the Ferrozine method; WIPP-Solubility-6 pg. 87-90; 4/24/09 tab of WIPP-Solubility-6 binder A
[f] Measured using the Ferrozine method; WIPP-Solubility-8 pg. 37-38; 6/30/09 tab of WIPP-Solubility-8 binder A

Table 3-10(continued) Measured data for the solubility of $Fe_2(OH)_3Cl(s)$ in NaCl solutions (the "Na^+—$Fe(OH)_3^-$" experiment).

Set-up ID	pH* (40 days)	pH* (191 days)	pH* (243 days)	pH* (590 days)	[Fe(II)]# (mol/L) (75 days)	[Fe(II)]# (mol/L) (243 days)	[Fe_T]# (mol/L) (75 days)	[Fe_T]# (mol/L) (176 days)	[Fe_T]# (mol/L) (243 days)
1m-Cl-Fe(OH)2-O-1		10.59	10.46**	8.90					
1m-Cl-Fe(OH)2-O-2	8.67	8.58	8.53		1.30E-4	2.15E-4	1.33E-4		2.38E-4
5m-Cl-Fe(OH)2-O-1	8.89	8.97	8.96	8.90	7.81E-6		8.57E-6	5.92E-6	6.28E-6
5m-Cl-Fe(OH)2-O-2	8.80	8.93	8.97	8.94	6.71E-6		7.43E-6	6.94E-6	7.42E-6
Reference	WIPP-Solubility-4, p.89-91	WIPP-Solubility-8, p.24-25	WIPP-Solubility-8, p.30-31	WIPP-Solubility-11, p.24	WIPP-Solubility-6, p. 18-24	WIPP-Solubility-8, p. 32-33,54-55	WIPP-Solubility-6, p. 18-24	WIPP-Solubility-11, p.31	WIPP-Solubility-8, p. 32-33,54-55

* Measured with pH electrode and meter.
** 240 days
# Measured using the Ferrozine method.

Table 3-10(continued) Measured data for the solubility of $Fe_2(OH)_3Cl(s)$ in NaCl solutions (the "$Fe(OH)_3^-Na^+$" experiment).

Set-up ID	pH* (1 days)	pH* (10 days)	pH* (21 days)	pH* (28 days)	pH* (57 days)	pH* (94 days)	pH* (112 days)	pH* (168 days)	[Fe(II)] (mol/L) (28 days)	[Fe(II)] (mol/L) (94 days)	[Fe(II)] (mol/L) (112 days)	[Cl(-I)] (m) (28, 94, 112 days)[a]
SQDLTN+0	8.105	8.039	7.973	8.004	8.076	8.017	8.021	8.011	1.48E-3	1.59E-3	1.60E-3	0.0363
SQDLTN+1	8.144	7.929	8.031	8.096	8.109	8.101	8.090	7.982	1.17E-3	1.28E-3	1.28E-3	0.152
SQDLTN+2	8.181	8.017	8.084	8.121	8.090	8.172	8.150	8.178	1.02E-3	1.14E-3	1.15E-3	0.504
SQDLTN+3	8.195	8.008	8.112	8.151	8.101	8.207	8.188	8.232	8.59E-4	9.13E-4	9.15E-4	1.22
SQDLTN+4	8.110	7.862	8.036	8.077	7.956	8.145	8.137	8.278	6.26E-4	6.39E-4	6.10E-4	3.03
SQDLTN+5	7.943	7.949	8.043	8.153	8.725	8.830	8.841	8.957	1.19E-4	6.14E-6	3.90E-6	5.97
Reference	WIPP-Solubility-5, p.73, 95								WIPP-Solubility-5, p.94	WIPP-Solubility-7, p.56	WIPP-Solubility-7, p.70	WIPP-Solubility-5, p.73

* Measured with pH electrode and meter; [a] gravimetric determination.

3.6 Solubility of $Fe_2(OH)_3Cl(s)$ in $CaCl_2$ solutions, where Ca^{2+} is analogue for Mg^{2+} (the "Mg^{2+}—$Fe(OH)^{3-}$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $Fe_2(OH)_3Cl(s)$ (iron hibbingite) in $MgCl_2$ (magnesium chloride) solutions, and (2) determine the Pitzer ion-interaction parameters for the ion pair "Mg^{2+}—$Fe(OH)_3^-$" (Item 6, Table 1-1). Preparation and available data are in tables below.

The experimental set-ups including initial brine molalities and mass of solid materials are shown below in Table 3-11. The solid phase, $Fe_2(OH)_3Cl(s)$, was synthesized from $FeCl_2 \cdot 4H_2O$ (Fisher) and NaOH (Fisher). Brines were prepared from DI water and $CaCl_2$ (Fisher). Because a high pH is needed to obtain a strong interaction between Mg^{2+} and $Fe(OH)_3^-$, we decided to use Ca^{2+} as an analogue. The solid phase, $Ca(OH)_2$ (Fisher), was added to each experimental set-up to maintain a high pH.

The experimental set-ups have been monitored one time to date for pH or dissolved Fe(II) concentrations($[Fe(II)]_{diss}$). Monitoring will continue, and once stable pH readings and values of $[Fe(II)]_{diss}$ are achieved, concentrations of other dissolved components ($[Ca(II)]_{diss}$, $[Cl(-I)]_{diss}$) are to be determined. Experimental results gathered to date are shown in Table 3-12.

Table 3-11 Preparation of experimental set-ups for the solubility of $Fe(OH)_2(s)$ in $CaCl_2$ solutions, where Ca^{2+} is analogue for Mg^{2+} (the "Mg^{2+}—$Fe(OH)^{3-}$" experiment).

Set-up ID*	$Fe(OH)_2(s)$ (g)	$CaCl_2$ (m)	Ca(OH)2(s) (g)	Reference
Fe(OH)2/Cl -0.01A	1	0.01	n.d.#	WIPP-Solubility-1, pg. 37-44
Fe(OH)2/Cl -0.1A	1	0.1	n.d.#	WIPP-Solubility-1, pg. 37-44
Fe(OH)2/Cl -1A	1	1.0	n.d.#	WIPP-Solubility-1, pg. 37-44
Fe(OH)2/Cl -1.5A	1	1.5	n.d.#	WIPP-Solubility-1, pg. 37-44
Fe(OH)2/Cl -2A	1	2.0	n.d.#	WIPP-Solubility-1, pg. 37-44
Fe(OH)2/Cl -2.5A	1	2.5	n.d.#	WIPP-Solubility-1, pg. 37-44

* These set-ups were prepared over a period of time spanning from 6/5/2008 to 8/11/2008. The last date of preparation was considered completion date of preparation to determine the number of days of equilibration in the table below.

Not Determined. Added in excess (~0.3gram) (Refer to email from Martin Nemer on 3/3/11). Exact mass of $Ca(OH)_2$ is not critical in this experiment, and no record in the above reference.

Table 3-12 Measured data for the solubility of $Fe_2(OH)_3Cl(s)$ in $CaCl_2$ solutions, where Ca^{2+} is analogue for Mg^{2+} (the "Mg^{2+}—$Fe(OH)_3^-$" experiment)

Set-up ID	pH[a] (483 days)	Fe_T (mol/L)[b] (343 days)
Fe(OH)2/Cl-0.01A	7.754[a1]	6.53E-03
Fe(OH)2/Cl -0.1A	12.074	1.50E-05[c]
Fe(OH)2/Cl -1A	11.599	1.32E-05[c]
Fe(OH)2/Cl -1.5A	-	1.52E-05[c]
Fe(OH)2/Cl -2A	10.682	2.11E-05[c]
Fe(OH)2/Cl -2.5A	10.985	3.41E-03
Reference	pg.56, 57, and 59 of WIPP-Solubility-8	pg. 75-76 of WIPP-Solubility-1 and 7-24-08 tab of WIPP-Solubility-3 Binder A

[a] Measured with Thermo Scientific Ross Sure-flow semi-micro electrode and Orion 720A pH meter; [a1] 485 days; [b] Measured using ICP-AES ; [c]Results below ICP-AES calibration curve

3.7 Solubility of $FeC_2O_4 \cdot 2H_2O$(s) in NaCl solutions (the "FeOx(aq)—Na^+" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $FeC_2O_4 \cdot 2H_2O$(s) (ferrous oxalate dihydrate) in NaCl (sodium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "FeOx(aq)—Na^+" (Item 7, Table 1-1).

The experimental set-ups including initial brine molalities and mass of solid materials are shown below in Table 3-13. Ferrous oxalate dihydrate ($FeC_2O_4 \cdot 2H_2O$) was purchased from Alfa Aesar (Cat A13479) and sodium chloride (NaCl) from Fisher. A known mass of $FeC_2O_4 \cdot 2H_2O$ was added to 50 mL of NaCl solutions of incremental concentrations (Table 3-13). In series -3 and -4 listed below, the ferrous oxalate dihydrate was first washed with sodium hydroxide to remove excess oxalic acid from the starting material.

The experimental set-ups are periodically monitored for pH and dissolved Fe(II) concentrations($[Fe(II)]_{diss}$). Once stable pH readings are achieved, concentrations of other dissolved components (sodium, oxalate, and chloride) are to be determined. Experimental results gathered to date are shown in Table 3-14.

Table 3-13 Preparation of experimental set-ups for the solubility of $FeC_2O_4 \cdot 2H_2O$ in NaCl solutions (the "FeOx(aq)—Na^+" experiment).

Set-up ID	$FeC_2O_4 \cdot 2H_2O(s)$ (g)	NaCl	Reference
0.1-FeOx-3	1.2[a]	0.1 M	WIPP-Solubility-4, pg. 24-25
1M-FeOx-3	1.2[a]	1.0 M	WIPP-Solubility-4, pg. 24-25
2M-FeOx-3	1.2[a]	2.0 M	WIPP-Solubility-4, pg. 24-25
3M-FeOx-3	1.2[a]	3.0 M	WIPP-Solubility-4, pg. 24-25
4M-FeOx-3	1.2[a]	4.0 M	WIPP-Solubility-4, pg. 24-25
5M-FeOx-3*	1.2[a]	5.0 m	WIPP-Solubility-4, pg. 24-25
0.1-FeOx-4	1.2[a]	0.1 M	WIPP-Solubility-4, pg. 24-25
1M-FeOx-4	1.2[a]	1.0 M	WIPP-Solubility-4, pg. 24-25
2M-FeOx-4	1.2[a]	2.0 M	WIPP-Solubility-4, pg. 24-25
3M-FeOx-4	1.2[a]	3.0 M	WIPP-Solubility-4, pg. 24-25
4M-FeOx-4	1.2[a]	4.0 M	WIPP-Solubility-4, pg. 24-25
5M-FeOx-4*	1.2[a]	5.0 m	WIPP-Solubility-4, pg. 24-25
FeOx-0.1A	2	0.1 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-1A	2	1.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-2A	2	2.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-3A	2	3.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-4A	2	4.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-5A	2	5.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-0.1B	2	0.1 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-1B	2	1.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-2B	2	2.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-3B	2	3.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-4B	2	4.0 m[#]	WIPP-Solubility-1, p. 44, 80
FeOx-5B	2	5.0 m[#]	WIPP-Solubility-1, p. 44, 80

* Unlike their IDs, these samples were made in 5m NaCl solution.
[a] No record in reference of WIPP-Solubility-4, p.24-25. Martin Nemer approximately estimated.
[#] Preparation of NaCl solution was described in p.11 of WIPP-Solubility-2.

Table 3-14 Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in NaCl solutions (the "FeOx(aq)—Na^+" experiment)

Set-up ID	pH[a] (65 day)	pH[a] (71 days)	pH[a] (78 days)	pH[a] (92 days)	pH[a] (114 days)	pH[a] (141 days)	pH[a] (160 days)	pH[a] (241 days)	pH[a] (244 days)	pH[a] (366 days)
0.1-FeOx-3	5.231	5.264	5.130	5.045	5.225	5.104	5.096	5.188	-	-
1M-FeOx-3	5.498	5.481	5.381	5.318	5.445	5.282	5.228	5.321	-	-
2M-FeOx-3	5.232	5.223	5.032	5.213	5.210	5.065	5.029	5.171	-	-
3M-FeOx-3	5.159	5.150	5.139	5.123	5.114	5.006	4.858	5.036	-	-
4M-FeOx-3	5.131	5.113	5.083	5.066	5.095	4.980	4.985	4.977	-	-
5M-FeOx-3*	6.347	6.493	6.384	6.263	6.493	5.879	5.883	5.587	-	-
0.1-FeOx-4	-	-	-	-	-	5.150	5.098	-	5.144	5.199
1M-FeOx-4	-	-	-	-	-	5.226	5.204	-	5.263	5.327
2M-FeOx-4	-	-	-	-	-	5.063	5.036	-	5.098	5.177
3M-FeOx-4	-	-	-	-	-	5.011	4.955	-	5.005	5.112
4M-FeOx-4	-	-	-	-	-	4.989	4.947	-	4.904	4.878
5M-FeOx-4*	-	-	-	-	-	5.933	6.031	-	5.629	5.259
Reference	WIPP-Solubility-5, p.12	WIPP-Solubility-5, p.16	WIPP-Solubility-5, p.24	WIPP-Solubility-5, p.38	WIPP-Solubility-5, p.43	WIPP-Solubility-5, p.57	WIPP-Solubility-5, p.70	WIPP-Solubility-5, p.72	WIPP-Solubility-5, p.72	WIPP-Solubility-5, p.72

* Unlike their IDs, these samples were made in 5m NaCl solution.

[a] Measured with Accumet pH electrode and VWR sympHony pH meter.

Table 3-14(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in NaCl solutions (the "FeOx(aq)—Na^+" experiment)

	($[Fe(II)]_{diss}$, mol/L[b]						
Set-up ID	(65 days)	(71 days)	(78 days)	(92 days)	(115 days)	(244 days)	(367 days)
0.1-FeOx-3	3.11E-04	3.46E-04	3.27E-04	3.31E-04	3.30E-04	3.11E-04	-
1M-FeOx-3	6.32E-04	6.21E-04	6.37E-04	6.19E-04	6.28E-04	6.26E-04	-
2M-FeOx-3	7.90E-04	7.50E-04	7.67E-04	7.33E-04	7.48E-04	7.49E-04	-
3M-FeOx-3	8.11E-04	7.70E-04	7.73E-04	7.56E-04	7.63E-04	7.56E-04	-
4M-FeOx-3	8.43E-04	7.81E-04	7.88E-04	7.76E-04	7.74E-04	7.54E-04	-
5M-FeOx-3	7.75E-04	7.72E-04	7.70E-04	7.49E-04	7.54E-04	7.35E-04	-
0.1-FeOx-4	-	-	-	-	-	3.13E-04	3.25E-04
1M-FeOx-4	-	-	-	-	-	6.21E-04	5.98E-04
2M-FeOx-4	-	-	-	-	-	7.43E-04	7.33E-04
3M-FeOx-4	-	-	-	-	-	7.70E-04	7.63E-04
4M-FeOx-4	-	-	-	-	-	7.48E-04	7.46E-04
5M-FeOx-4	-	-	-	-	-	7.20E-04	7.05E-04
Reference	WIPP-Solubility-5, p.13	WIPP-Solubility-5, p.17	WIPP-Solubility-5, p.25	WIPP-Solubility-5, p.39	WIPP-Solubility-5, p.45	WIPP-Solubility-7, p.50	WIPP-Solubility-7, p.78

[b] Measured using the Ferrozine method

Table 3-14(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in NaCl solutions (the "FeOx(aq)—Na^+" experiment)

Set-up ID	Fe_T (mol/L) ICP (43 days)	pH* (511 days)	Set-upID	pH* (511 days)
FeOx-0.1A	2.14E-04	3.975	FeOx-0.1B	4.030
FeOx-1A	7.53E-04	3.354	FeOx-1B	3.421
FeOx-2A	9.26E-04	3.011	FeOx-2B	2.903
FeOx-3A	1.32E-03	2.274	FeOx-3B	2.246#
FeOx-4A	1.42E-03	1.923	FeOx-4B	1.923#
FeOx-5A	1.62E-03	1.680	FeOx-5B	1.668#
Reference	pg. 75-76 of WIPP-Solubility-1 and 7-24-08 tab of WIPP-Solubility-3 Binder A	pg.70-71 of WIPP-Solubility-8		pg.70-71 of WIPP-Solubility-8

* Measured with pH electrode and meter.
# 510 days

3.8 Solubility of $FeC_2O_4 \cdot 2H_2O$(s) in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $FeC_2O_4 \cdot 2H_2O$(s) (ferrous oxalate dihydrate) in $MgCl_2$ (magnesium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "FeOx(aq)—Mg^{+2}" (Item 8, Table 1-1).

The experimental set-ups including initial brine molalities and mass of solid materials are shown below in Table 3-15. $FeC_2O_4 \cdot 2H_2O$(s) was purchased from Alfa Aesar (puratronic, 99.999 % metal basis), and magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) was purchased from Fisher. $FeC_2O_4 \cdot 2H_2O$(s) was added to $MgCl_2 \cdot 6H_2O$ solutions of incremental concentrations, ranging from 0.01 to 2.5 m $MgCl_2$. Since solubility should be independent of the solid mass and liquid volume as long as the solid phase persists when the equilibrium is reached, the mass of $FeC_2O_4 \cdot 2H_2O$ and the volume of the $MgCl_2$ solutions were not measured for 30 of the experimental set-ups (FeOxMg-(1~6)-(1~5), Table 3-15 below). However, as these set-ups progressed toward equilibrium, some phenomena were observed that might indicate the solid-to-liquid ratio in the set-ups has an effect. Thus, 8 additional set-ups (FeOxMg-Ratio-(no identifier or DI)-(1~4), Table 3-15 below) were prepared to test the hypothesis regarding the effect of the solid-to-liquid ratio.

The concentration of dissolved ferrous iron ($[Fe(II)]_{diss}$) and the pH are monitored over time to see if the experimental set-ups are approaching equilibrium. After observing stable pH and/or Fe(II) concentrations, dissolved concentrations for the other components in the system (magnesium, oxalate, chloride) are to be measured. Measured values to date for this investigation are documented in Table 3-16.

Table 3-15 Experimental set-ups for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

Set-up ID	$MgCl_2$ (m)	$FeC_2O_4 \cdot 2H_2O$(s) (g)	Volume of solution (mL)	Reference
FeOxMg-1-(1,2,3)	0.010	n.d.*	n.d.	WIPP-Solubility-5, p.4
FeOxMg-2-(1,2,3)	0.10	n.d.	n.d.	WIPP-Solubility-5, p.4
FeOxMg-3-(1,2,3)	0.48	n.d.	n.d.	WIPP-Solubility-5, p.4
FeOxMg-4-(1,2,3)	0.93	n.d.	n.d.	WIPP-Solubility-5, p.4
FeOxMg-5-(1,2,3)	1.3	n.d.	n.d.	WIPP-Solubility-5, p.4
FeOxMg-6-(1,2,3)	2.0	n.d.	n.d.	WIPP-Solubility-5, p.4
FeOxMg-1-(4,5)	0.010	n.d.	n.d.	WIPP-Solubility-5, p.71
FeOxMg-2-(4,5)	0.10	n.d.	n.d.	WIPP-Solubility-5, p.71
FeOxMg-3-(4,5)	0.49	n.d.	n.d.	WIPP-Solubility-5, p.71
FeOxMg-4-(4,5)	0.92	n.d.	n.d.	WIPP-Solubility-5, p.71
FeOxMg-5-(4,5)	1.3	n.d.	n.d.	WIPP-Solubility-5, p.71
FeOxMg-6-(4,5)	2.0	n.d.	n.d.	WIPP-Solubility-5, p.71
FeOxMg-Ratio-1	2.5	0.1205	56.4#	WIPP-Solubility-9, p.23
FeOxMg-Ratio-2	2.5	1.0864	48.7#	WIPP-Solubility-9, p.23
FeOxMg-Ratio-3	2.5	4.8462	41.2#	WIPP-Solubility-9, p.23
FeOxMg-Ratio-4	2.5	10.2721	36.2#	WIPP-Solubility-9, p.23
FeOxMg-Ratio-DI-1	0	0.1005	47.0#	WIPP-Solubility-9, p.36
FeOxMg-Ratio-DI-2	0	0.4593	48.2#	WIPP-Solubility-9, p.36
FeOxMg-Ratio-DI-3	0	2.4009	43.7#	WIPP-Solubility-9, p.36
FeOxMg-Ratio-DI-4	0	10.0479	41.7#	WIPP-Solubility-9, p.36

Either 2.5 m $MgCl_2$ or DDI water.
* Not determined.

Table 3-16 Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

Set-up ID	pH[a] (7 days)	pH[a] (13 days)	pH[a] (21 days)	pH[a] (34 days)	pH[a] (57 days)	pH[a] (83 days)	pH[a] (104 days)	pH[a] (156 days)	pH[a] (171 days)	pH[a] (287 days)
FeOxMg-1-1	5.692	5.654	5.305	5.347	5.692	5.725	5.567	5.748	5.652	6.104
FeOxMg-2-1	5.272	5.282	5.305	5.110	5.302	5.402	5.345	5.356	5.319	5.730
FeOxMg-3-1	5.159	5.174	4.973	5.003	5.288	5.148	5.196	5.183	5.363	5.679
FeOxMg-4-1	5.146	5.114	4.916	4.948	5.275	5.048	5.036	4.998	5.221	5.414
FeOxMg-5-1	5.012	5.052	4.793	4.804	5.222	4.961	4.853	4.797	5.159	5.346
FeOxMg-6-1	4.585	4.571	4.565	4.364	4.807	4.385	3.677	4.117	4.236	4.509
Reference	WIPP-Solubility-5, p.10	WIPP-Solubility-5, p.14	WIPP-Solubility-5, p.22	WIPP-Solubility-5, p.34	WIPP-Solubility-5, p.41	WIPP-Solubility-5, p.55	WIPP-Solubility-5, p.70	WIPP-Solubility-5, p.96	WIPP-Solubility-5, p.96	WIPP-Solubility-5, p.96

[a] Measured with pH electrode and meter.

Table 3-16(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

Set-up ID	pH[a] (83 days)	pH[a] (104 days)	pH[a] (156 days)	pH[a] (177 days)	pH[a] (181 days)	pH[a] (295 days)	pH[a] (307 days)	pH[a] (534 days)
FeOxMg-1-2	5.710	5.697	5.663	5.716	-	6.126	-	-
FeOxMg-2-2	5.416	5.374	5.458	5.540	-	5.913	-	-
FeOxMg-3-2	5.244	5.204	5.282	5.392	-	5.731	-	-
FeOxMg-4-2	4.968	4.896	5.181	5.429	-	5.794	-	-
FeOxMg-5-2	4.864	4.794	4.750	4.942	-	4.961	-	-
FeOxMg-6-2	3.825*	3.782	4.153	4.069	-	4.310	-	-
FeOxMg-1-3	4.567	4.562	4.589	-	4.636	-	4.663	4.695
FeOxMg-2-3	4.411	4.338	4.346	-	4.380	-	4.402	4.397
FeOxMg-3-3	4.163	4.111	4.134	-	4.158	-	4.190	4.192
FeOxMg-4-3	3.997*	3.952	3.941	-	4.005	-	4.040	4.042
FeOxMg-5-3	3.938*	3.864	3.911	-	3.958	-	3.994	4.006
FeOxMg-6-3	3.674*	3.553	3.649	-	3.767	-	3.827	3.864
Reference	WIPP-Solubility-5, p.55, 56	WIPP-Solubility-5, p.70	WIPP-Solubility-5, p.96	WIPP-Solubility-5, p.96	WIPP-Solubility-5, p.96	WIPP-Solubility-5, p.96	WIPP-Solubility-5, p.96	WIPP-Solubility-5, p.96

* Measured on day 84 (WIPP-Solubility-5, p.56). [a] Measured with pH electrode and meter.

Table 3-16(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

Set-up ID	pH[a] (6 days)	pH[a] (7 days)	pH[a] (42 days)	pH[a] (42 days)	pH[a] (51 days)	pH[a] (79 days)	pH[a] (105 days)	pH[a] (203 days)	pH[a] (204 days)
FeOxMg-1-4	4.799	-	-	-	4.628	4.679	-	4.684	-
FeOxMg-2-4	4.646	-	-	-	4.477	4.516	-	4.543	-
FeOxMg-3-4	4.409	-	-	-	4.279	4.344	-	4.406	-
FeOxMg-4-4	4.114	-	-	-	3.992	4.048	-	4.103	-
FeOxMg-5-4	3.837	-	-	-	3.683	3.743	-	3.792	-
FeOxMg-6-4	3.579	-	-	-	3.463	3.556	-	3.600	-
FeOxMg-1-5	4.854	-	-	-	4.614	4.686	-	-	4.649
FeOxMg-2-5	4.706	-	-	-	4.508	4.548	-	-	4.532
FeOxMg-3-5	4.424	-	-	-	4.273	4.302	-	-	4.288
FeOxMg-4-5	4.153	-	-	-	4.030	4.060	-	-	4.063
FeOxMg-5-5	3.951	-	-	-	3.797	3.863	-	-	3.864
FeOxMg-6-5	3.557	-	-	-	3.480	3.571	-	-	3.581
FeOxMg-Ratio-1	-	-	5.671	-	-	-	5.548	-	-
FeOxMg-Ratio-2	-	-	3.705	-	-	-	3.570	-	-
FeOxMg-Ratio-3	-	-	2.688	-	-	-	2.635	-	-
FeOxMg-Ratio-4	-		2.435	-	-	-	2.396	-	-
FeOxMg-Ratio-DI-1	-	5.898	-	6.202	-	-	-	-	-
FeOxMg-Ratio-DI-2	-	5.224	-	5.125	-	-	-	-	-
FeOxMg-Ratio-DI-3	-	4.886	-	4.609	-	-	-	-	-
FeOxMg-Ratio-DI-4	-	4.498	-	4.197	-	-	-	-	-
Reference	WIPP-Solubility-5, p.72	WIPP-Solubility-9, p.37	WIPP-Solubility-9, p.32	WIPP-Solubility-9, p.37	WIPP-Solubility-5, p.72	WIPP-Solubility-5, p.72	WIPP-Solubility-9, p.32	WIPP-Solubility-5, p.72	WIPP-Solubility-5, p.72

[a] Measured with pH electrode and meter.

Table 3-16(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

	$Fe[(II)]_{diss}$, mol/L[b]								
Set-up ID	(7 days)	(13 days)	(21 days)	(34 days)	(57 days)	(171 days)	(177 days)	(288 days)	(295 days)
FeOxMg-1-1	5.03E-04	5.17E-04	5.20E-04	4.99E-04	5.17E-04	5.08E-04	-	5.10E-04	-
FeOxMg-2-1	1.48E-03	1.47E-03	1.47E-03	1.51E-03	1.49E-03	1.54E-03	-	1.51E-03	-
FeOxMg-3-1	2.76E-03	3.50E-03	3.50E-03	3.58E-03	3.68E-03	3.81E-03	-	3.98E-03	-
FeOxMg-4-1	2.62E-03	4.15E-03	4.78E-03	5.14E-03	5.39E-03	6.69E-03	-	7.62E-03	-
FeOxMg-5-1	2.68E-03	4.53E-03	5.30E-03	6.02E-03	6.67E-03	8.70E-03	-	8.65E-03	-
FeOxMg-6-1	2.79E-03	5.14E-03	6.26E-03	7.42E-03	8.46E-03	8.95E-03	-	8.64E-03	-
FeOxMg-1-2	-	-	-	-	-	-	5.18E-04	-	5.01E-04
FeOxMg-2-2	-	-	-	-	-	-	1.53E-03	-	1.56E-03
FeOxMg-3-2	-	-	-	-	-	-	3.74E-03	-	4.00E-03
FeOxMg-4-2	-	-	-	-	-	-	6.40E-03	-	7.51E-03
FeOxMg-5-2	-	-	-	-	-	-	8.41E-03	-	8.72E-03
FeOxMg-6-2	-	-	-	-	-	-	8.99E-03	-	9.33E-03
Reference	WIPP-Solubility-5, p.11	WIPP-Solubility-5, p.15	WIPP-Solubility-5, p.23	WIPP-Solubility-5, p.35	WIPP-Solubility-5, p.42	WIPP-Solubility-7, p.37	WIPP-Solubility-7, p.44	WIPP-Solubility-7, p.76	WIPP-Solubility-7, p.76

[b] Measured using the Ferrozine method

Table 3-16(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

	$Fe[(II)]_{diss}$, mol/L[a]								
Set-up ID	(79 days)	(80 days)	(80 days)	(103 days)	(181 days)	(203 days)	(204 days)	(307 days)	(535 days)
FeOxMg-1-3	-	-	-	-	5.09E-04	-	-	5.08E-04	5.07E-04
FeOxMg-2-3	-	-	-	-	1.55E-03	-	-	1.57E-03	1.60E-03
FeOxMg-3-3	-	-	-	-	4.18E-03	-	-	4.42E-03	4.93E-03
FeOxMg-4-3	-	-	-	-	7.55E-03	-	-	8.49E-03	1.01E-02
FeOxMg-5-3	-	-	-	-	9.15E-03	-	-	9.95E-03	1.00E-02
FeOxMg-6-3	-	-	-	-	9.80E-03	-	-	9.65E-03	9.92E-03
FeOxMg-1-4	5.27E-04	-	-	-	-	5.25E-04	-	-	-
FeOxMg-2-4	1.60E-03	-	-	-	-	1.61E-03	-	-	-
FeOxMg-3-4	4.63E-03	-	-	-	-	5.20E-03	-	-	-
FeOxMg-4-4	8.00E-03	-	-	-	-	1.01E-02	-	-	-
FeOxMg-5-4	1.23E-02	-	-	-	-	1.41E-02	-	-	-
FeOxMg-6-4	1.79E-02	-	-	-	-	2.24E-02	-	-	-
FeOxMg-1-5	-	5.11E-04	-	-	-	-	5.04E-04	-	-
FeOxMg-2-5	-	1.57E-03	-	-	-	-	1.58E-03	-	-
FeOxMg-3-5	-	4.54E-03	-	-	-	-	5.16E-03	-	-
FeOxMg-4-5	-	7.93E-03	-	-	-	-	9.37E-03	-	-
FeOxMg-5-5	-	1.12E-02	-	-	-	-	1.41E-02	-	-
FeOxMg-6-5	-	1.78E-02	-	-	-	-	2.23E-02	-	-
FeOxMg-Ratio-1	-	-	-	1.09E-02	-	-	-	-	-
FeOxMg-Ratio-2	-	-	-	3.20E-02	-	-	-	-	-
FeOxMg-Ratio-3	-	-	-	4.69E-02	-	-	-	-	-
FeOxMg-Ratio-4	-	-	-	5.34E-02	-	-	-	-	-
FeOxMg-Ratio-DI-1	-	-	1.82E-04	-	-	-	-	-	-
FeOxMg-Ratio-DI-2	-	-	1.86E-04	-	-	-	-	-	-
FeOxMg-Ratio-DI-3	-	-	2.09E-04	-	-	-	-	-	-
FeOxMg-Ratio-DI-4	-	-	3.30E-04	-	-	-	-	-	-
Reference	WIPP-Solubility-7, p.45	WIPP-Solubility-7, p.46	WIPP-Solubility-9, p.59	WIPP-Solubility-9, p.60	WIPP-Solubility-7, p.45	WIPP-Solubility-7, p.77	WIPP-Solubility-7, p.78	WIPP-Solubility-7, p.77	WIPP-Solubility-9, p.64

[a] Measured using the Ferrozine method

Table 3-16(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

	$Cl[(-I)]_{diss}$, mol/L[a]		$Mg[(II)]_{diss}$, mol/L[b]		
Set-up ID	(103 days)	(535 days)	(103 days)	(535 days)	(541 days)
FeOxMg-1-1	-	-	-	-	1.02E-02
FeOxMg-2-1	-	-	-	-	9.99E-02
FeOxMg-3-1	-	-	-	-	5.03E-01
FeOxMg-4-1	-	-	-	-	9.56E-01
FeOxMg-5-1	-	-	-	-	1.30E-00
FeOxMg-6-1	-	-	-	-	1.77E-00
FeOxMg-1-2	-	-	-	-	1.01E-02
FeOxMg-2-2	-	-	-	-	1.00E-01
FeOxMg-3-2	-	-	-	-	4.98E-01
FeOxMg-4-2	-	-	-	-	9.39E-01
FeOxMg-5-2	-	-	-	-	1.30E-00
FeOxMg-6-2	-	-	-	-	1.76E-00
FeOxMg-1-3	-	1.83E-02	-	1.26E-02	-
FeOxMg-2-3	-	1.89E-01	-	1.29E-01	-
FeOxMg-3-3	-	8.77E-01	-	5.45E-01	-
FeOxMg-4-3	-	1.69E-00	-	1.06E-00	-
FeOxMg-5-3	-	2.42E-00	-	1.38E-00	-
FeOxMg-6-3	-	3.46E-00	-	1.87E-00	-
FeOxMg-Ratio-1	4.47E-00	-	2.29E-00	-	-
FeOxMg-Ratio-2	4.28E-00	-	2.23E-00	-	-
FeOxMg-Ratio-3	4.47E-00	-	2.22E-00	-	-
FeOxMg-Ratio-4	4.45E-00	-	2.17E-00	-	-
Reference	WIPP-Solubility-9, p.99	WIPP-Solubility-9, p.66	WIPP-Solubility-9, p.67	WIPP-Solubility-9, p.65	WIPP-Solubility-9, p.71

[a] $[Cl(-I)]_{diss}$ measured by IC; [b] $[Mg(II)]_{diss}$ measured by ICP-AES.

Table 3-16(continued) Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O$ in $MgCl_2$ solutions (the "FeOx(aq)—Mg^{2+}" experiment).

	$[Ox^{-2}]_{T, diss}$, mol/L[a]				
Set-up ID	(49 days)	(103 days)	(455 days)	(535 days)	(541 days)
FeOxMg-1-1	-	-	-	-	3.20×10^{-4}
FeOxMg-2-1	-	-	-	-	1.47×10^{-3}
FeOxMg-3-1	-	-	-	-	3.10×10^{-3}
FeOxMg-4-1	-	-	-	-	3.80×10^{-3}
FeOxMg-5-1	-	-	-	-	1.60×10^{-3}
FeOxMg-6-1	-	-	-	-	1.95×10^{-3}
FeOxMg-1-2	-	-	-	-	4.80×10^{-4}
FeOxMg-2-2	-	-	-	-	1.34×10^{-3}
FeOxMg-3-2	-	-	-	-	3.08×10^{-3}
FeOxMg-4-2	-	-	-	-	3.59×10^{-3}
FeOxMg-5-2	-	-	-	-	1.21×10^{-3}
FeOxMg-6-2	-	-	-	-	1.36×10^{-3}
FeOxMg-1-3	-	-	-	5.74×10^{-4}	-
FeOxMg-2-3	-	-	-	1.61×10^{-3}	-
FeOxMg-3-3	-	-	-	2.85×10^{-3}	-
FeOxMg-4-3	-	-	-	5.98×10^{-4}	-
FeOxMg-5-3	-	-	-	8.80×10^{-4}	-
FeOxMg-6-3	-	-	-	1.16×10^{-3}	-
FeOxMg-1-4	-	-	4.95×10^{-4}	-	-
FeOxMg-2-4	-	-	1.07×10^{-3}	-	-
FeOxMg-3-4	-	-	2.15×10^{-3}	-	-
FeOxMg-4-4	-	-	2.63×10^{-3}	-	-
FeOxMg-5-4	-	-	3.64×10^{-3}	-	-
FeOxMg-6-4	-	-	4.40×10^{-3}	-	-
FeOxMg-1-5	-	-	4.46×10^{-4}	-	-
FeOxMg-2-5	-	-	1.58×10^{-3}	-	-
FeOxMg-3-5	-	-	3.73×10^{-3}	-	-
FeOxMg-4-5	-	-	2.91×10^{-3}	-	-
FeOxMg-5-5	-	-	3.43×10^{-3}	-	-
FeOxMg-6-5	-	-	4.48×10^{-3}	-	-
FeOxMg-Ratio-1	-	2.54×10^{-3}	-	-	-
FeOxMg-Ratio-2	-	2.46×10^{-3}	-	-	-
FeOxMg-Ratio-3	-	2.33×10^{-3}	-	-	-
FeOxMg-Ratio-4	-	2.35×10^{-3}	-	-	-
FeOxMg-Ratio-DI-1	1.66×10^{-4}	-	-	-	-
FeOxMg-Ratio-DI-2	1.75×10^{-4}	-	-	-	-
FeOxMg-Ratio-DI-3	2.22×10^{-4}	-	-	-	-
FeOxMg-Ratio-DI-4	4.14×10^{-4}	-	-	-	-
Reference	WIPP-Solubility-9, p.99	WIPP-Solubility-9, p.97	WIPP-Solubility-9, p.97	WIPP-Solubility-9, p.97	WIPP-Solubility-9, p.97

[a] Measured with IC

3.9 Solubility of $FeC_2O_4 \cdot 2H_2O$(s) in NaCl solutions; Solubility of $FeC_2O_4 \cdot 2H_2O$(s) in $MgCl_2$ solutions (the "FeOx(aq)—Cl^-" experiment)

The objective of this section is to determine the Pitzer ion-interaction parameters for the ion pair "FeOx(aq)-Cl^-" (Item 9, Table 1-1). The experimental results from sections 3.7 and 3.8 will be combined to determine the parameters for the ion pair "FeOx(aq)-Cl^-". Refer to sections 3.7 and 3.8 for the data.

3.10 Solubility of $Fe(OH)_2$(s) in H_2Na_2EDTA solutions (the "Na^+—$FeEDTA^{2-}$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $Fe(OH)_2$(s) (ferrous iron hydroxide) in H_2Na_2EDTA (disodium dihydrogen EDTA) solutions, and (2) determine the Pitzer ion-interaction parameters for the ion pair "Na^+—$FeEDTA^{2-}$" (Item 10, Table 1-1).

The experimental set-ups are listed in Table 3-17. Solid was synthesized by mixing 13.353 g NaOH and 33.1869 g $FeCl_2 \cdot 4H_2O$ (WIPP-Solubility-1, p.48). The experimental set-ups have been periodically monitored for pH and dissolved total Fe concentrations ($[Fe_T]_{diss}$). Once stable pH readings are achieved, concentrations of other dissolved components (sodium, EDTA, and chloride) are to be determined. Experimental results gathered to date are shown in Table 3-18.

Table 3-17 Preparation of experimental set-ups for the solubility of $Fe(OH)_2$ in H_2Na_2EDTA solutions (the "Na^+—$FeEDTA^{2-}$" experiment).

Set-up ID	$Fe(OH)_2$(s) (g)	NaCl (m)	Reference
Fe(OH)2-Na4EDTA-0.01A	0.9	0.01	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-0.1A	0.9	0.1	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-1A	0.9	1.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-2A	0.9	2.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-3A	0.9	3.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-4A	0.9	4.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-5A	0.9	5.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-0.01B	0.9	0.01	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-0.1B	0.9	0.1	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-1B	0.9	1.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-2B	0.9	2.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-3B	0.9	3.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-4B	0.9	4.0	WIPP-Solubility-1, pg. 51-55
Fe(OH)2-Na4EDTA-5B	0.9	5.0	WIPP-Solubility-1, pg. 51-55

Table 3-18 Measured data for the solubility of $Fe(OH)_2$ in H_2Na_2EDTA solutions (the "Na^+—$FeEDTA^{2-}$" experiment).

Set-up ID	pH[a] (464 days)	$[Fe_T]$ (mol/L)[b] by ICP-AES (58 days)	$[Fe_T]$ (mol/L)[c] by ICP-AES (519 days)
Fe(OH)2-Na4EDTA-0.01A	7.820	4.71E-02	5.43E-02
Fe(OH)2-Na4EDTA-0.1A	7.986	4.28E-02	5.13E-02
Fe(OH)2-Na4EDTA-1A	8.343	3.82E-02	4.45E-02
Fe(OH)2-Na4EDTA-2A	8.655	3.75E-02	4.70E-02
Fe(OH)2-Na4EDTA-3A	8.836	3.62E-02	4.84E-02
Fe(OH)2-Na4EDTA-4A	8.817	3.69E-02	4.79E-02
Fe(OH)2-Na4EDTA-5A	8.998	3.66E-02	4.84E-02
Fe(OH)2-Na4EDTA-0.1B	7.953	-	5.15E-02
Fe(OH)2-Na4EDTA-1B	8.370	-	4.47E-02
Fe(OH)2-Na4EDTA-2B	8.503	-	4.63E-02
Fe(OH)2-Na4EDTA-3B	8.772	-	4.80E-02
Fe(OH)2-Na4EDTA-4B	8.904	-	4.77E-02
Fe(OH)2-Na4EDTA-5B	9.001	-	4.90E-02

[a] Measured with pH electrode and meter; pg.59, 61 of WIPP-Solubility-8

[b] pg. 26-31 of WIPP-Solubility-4 and 8-29-09 tab of WIPP-Solubility-3 Binder A

[c] pg.92-95 of WIPP-Solubility-8, pg.9 of WIPP-Solubility-10, 12/3/09 tab of WIPP-Solubility-10 Binder 1

3.11 Solubility of $Fe(OH)_2(s)$ in $C_{10}H_{12}MgN_2Na_2O_8 \cdot 4H_2O$ ($MgNa_2EDTA \cdot 4H_2O$) solutions (the "Mg^{2+}—$FeEDTA^{2-}$" experiment).

The objectives of this set of experiments are to (1) determine the solubility of $Fe(OH)_2(s)$ (ferrous iron hydroxide) in $C_{10}H_{12}MgN_2Na_2O_8 \cdot 4H_2O$ ($MgNa_2EDTA \cdot 4H_2O$) (magnesium disodium EDTA) solutions, and (2) determine the Pitzer ion-interaction parameters for ion pair "Mg^{2+}—$FeEDTA^{2-}$" (Item 11, Table 1-1).

Ferrous iron hydroxide, $Fe(OH)_2(s)$, was synthesized; 0.256 mol of $FeCl_2 \cdot 4H_2O$ (Fisher) was dissolved and mixed with 98 mL of 5.211 M NaOH (Fisher) (WIPP-Solubility-5, page 52). XRD is given in WIPP-Solubility-5, page 77. $Fe(OH)_2(s)$ was added to magnesium disodium EDTA (J.T. Baker) solutions of incremental concentrations, ranging from 0.0107 to 0.769 m magnesium disodium EDTA (See Table 3-19). Since solubility is independent of the solid mass and experimental set-up volume as long as solid remains upon equilibrium, the mass of $Fe(OH)_2(s)$ and the volume of $MgNa_2EDTA$ solutions were not determined for all experimental set-ups.

The experimental set-ups were periodically monitored for pH. Once stable pH readings were observed, the concentrations of other dissolved components (dissolved total Fe, magnesium, sodium, and EDTA) were or are to be determined. Experimental results gathered to date are shown in Table 3-20

Table 3-19 Preparation of experimental set-ups for the solubility of $Fe(OH)_2(s)$* in $MgNa_2EDTA \cdot 4H_2O$ solutions (the "Mg^{2+}—$FeEDTA^{2-}$" experiment).

Set-up ID	$MgNa_2EDTA$ (m)	Reference
FeMgNa2EDTA-1-(1,2,3)	0.0107	WIPP-Solubility-7, p.41
FeMgNa2EDTA-2-(1,2,3)	0.108	WIPP-Solubility-7, p.41
FeMgNa2EDTA-3-(1,2,3)	0.216	WIPP-Solubility-7, p.41
FeMgNa2EDTA-4-(1,2,3)	0.396	WIPP-Solubility-7, p.41
FeMgNa2EDTA-5-(1,2,3)	0.597	WIPP-Solubility-7, p.41
FeMgNa2EDTA-6-(1,2,3)	0.769	WIPP-Solubility-7, p.41

* Mass of solid not determined. See second paragraph above for reason.

Table 3-20 Measured data for the solubility of $Fe(OH)_2$ in $MgNa_2EDTA{\cdot}4H_2O$ solutions (the "Mg^{2+}—$FeEDTA^{2-}$" experiment).

Set-up ID	pH* (1 day)	pH* (32 days)	pH* (35 days)	pH* (93 days)	pH* (94 days)	pH* (206 days)	pH* (212 days)
FeMgNa2EDTA-1-1	10.597	10.528	-	10.381	-	10.359	-
FeMgNa2EDTA-1-2	10.583	10.549	-	10.397	-	10.382	-
FeMgNa2EDTA-1-3	10.593	10.546	-	10.407	-	10.388	-
Average	**10.59**	**10.54**	**-**	**10.40**	**-**	**10.38**	**-**
2*σ	**0.01**	**0.02**	-	**0.03**	-	**0.03**	-
FeMgNa2EDTA-2-1	10.705	10.613	-	10.516	-	10.472	-
FeMgNa2EDTA-2-2	10.705	10.647	-	10.539	-	10.491	-
FeMgNa2EDTA-2-3	10.717	10.635	-	10.527	-	10.473	-
Average	**10.71**	**10.63**	-	**10.53**	-	**10.48**	-
2*σ	**0.01**	**0.03**	-	**0.02**	-	**0.02**	-
FeMgNa2EDTA-3-1	10.680	-	10.676	10.527	-	-	10.507
FeMgNa2EDTA-3-2	10.719	-	10.688	10.534	-	-	10.510
FeMgNa2EDTA-3-3	10.705	-	10.699	10.539	-	-	10.533
Average	**10.70**	**-**	**10.69**	**10.53**	**-**	**-**	**10.52**
2*σ	**0.04**	-	**0.02**	**0.01**	-	-	**0.03**
FeMgNa2EDTA-4-1	10.673	-	10.685	10.530	-	-	10.524
FeMgNa2EDTA-4-2	10.649	-	10.701	10.550	-	-	10.545
FeMgNa2EDTA-4-3	10.603	-	10.694	10.539	-	-	10.529
Average	**10.64**	**-**	**10.69**	**10.54**	-	**-**	**10.53**
2*σ	**0.07**	-	**0.02**	**0.02**	-	-	**0.02**
FeMgNa2EDTA-5-1	10.640	-	10.715	-	10.552	-	10.560
FeMgNa2EDTA-5-2	10.513	-	10.708	-	10.551	-	10.553
FeMgNa2EDTA-5-3	10.544	-	10.702	-	10.549	-	10.540
Average	**10.57**	**-**	**10.71**	**-**	**10.55**	**-**	**10.55**
2*σ	**0.13**	-	**0.01**	-	**0.00**	-	**0.02**
FeMgNa2EDTA-6-1	10.362	-	10.678	-	10.565	-	10.562
FeMgNa2EDTA-6-2	10.495	-	10.708	-	10.560	-	10.555
FeMgNa2EDTA-6-3	10.397	-	10.702	-	10.567	-	10.564
Average	**10.42**	**-**	**10.70**	**-**	**10.56**	**-**	**10.56**
2*σ	**0.14**	-	**0.03**	-	**0.01**	-	**0.01**
Reference	WIPP-Solubility-7, p.42	WIPP-Solubility-7, p.42	WIPP-Solubility-7, p.42	WIPP-Solubility-7, p.42	WIPP-Solubility-7, p.42	WIPP-Solubility-7, p.42	WIPP-Solubility-7, p.42

* Measured with pH electrode and meter

Table 3-20(continued) Measured data for the solubility of $Fe(OH)_2$ in $MgNa_2EDTA{\cdot}4H_2O$ solutions (the "Mg^{2+}—$FeEDTA^{2-}$" experiment).

Set-up ID	$[Fe_T]_{diss}$ (mol/L)	Reference	$[Mg(II)]_{diss}$ (mol/L)	Reference	$[Na(I)]_{diss}$ (mol/L)	Reference
FeMgNa2EDTA-1-1	1.08E-02	WIPP-Solubility-9, p.42 (295 days)	1.51E-04	WIPP-Solubility-9, p.42 (295 days)	1.79E-02	WIPP-Solubility-9, p.52 (295 days)
FeMgNa2EDTA-1-2	1.07E-02		1.39E-04		1.74E-02	
FeMgNa2EDTA-1-3	1.08E-02		1.12E-04		1.66E-02	
Average	**1.08E-02**		**1.34E-04**		**1.73E-02**	
2*σ	**1.15E-04**		**3.99E-05**		**1.26E-03**	
FeMgNa2EDTA-2-1	7.61E-02	WIPP-Solubility-9, p.43 (295 days)	5.90E-02	WIPP-Solubility-9, p.43 (295 days)	1.80E-01	WIPP-Solubility-9, p.52 (295 days)
FeMgNa2EDTA-2-2	6.01E-02		8.72E-02		1.82E-01	
FeMgNa2EDTA-2-3	7.49E-02		6.62E-02		1.80E-01	
Average	**7.04E-02**		**7.08E-02**		**1.80E-01**	
2*σ	**1.78E-02**		**2.93E-02**		**1.79E-03**	
FeMgNa2EDTA-3-1	8.36E-02	WIPP-Solubility-9, p.34 (237 days)	2.63E-01	WIPP-Solubility-9, p.40 (237 days)	4.52E-01	WIPP-Solubility-9, p.45 (237 days)
FeMgNa2EDTA-3-2	7.90E-02		2.55E-01		4.23E-01	
FeMgNa2EDTA-3-3	6.65E-02		2.85E-01		4.43E-01	
Average	**7.64E-02**		**2.68E-01**		**4.39E-01**	
2*σ	**1.77E-02**		**3.11E-02**		**2.97E-02**	
FeMgNa2EDTA-4-1	9.98E-02	WIPP-Solubility-9, p.34 (237 days)	4.86E-01	WIPP-Solubility-9, p.34 (237 days)	8.72E-01	WIPP-Solubility-9, p.45 (237 days)
FeMgNa2EDTA-4-2	7.60E-02		5.08E-01		8.01E-01	
FeMgNa2EDTA-4-3	8.67E-02		5.02E-01		8.20E-01	
Average	**8.75E-02**		**4.99E-01**		**8.31E-01**	
2*σ	**2.38E-02**		**2.27E-02**		**7.35E-02**	
FeMgNa2EDTA-5-1	6.30E-02	WIPP-Solubility-9, p.41 (295 days)	7.90E-01	WIPP-Solubility-9, p.41 (295 days)	1.03E+00	WIPP-Solubility-9, p.52 (295 days)
FeMgNa2EDTA-5-2	6.47E-02		7.78E-01		9.99E-01	
FeMgNa2EDTA-5-3	7.72E-02		7.70E-01		1.02E+00	
Average	**6.83E-02**		**7.79E-01**		**1.02E+00**	
2*σ	**1.55E-02**		**2.01E-02**		**3.41E-02**	
FeMgNa2EDTA-6-1	6.92E-02	WIPP-Solubility-9, p.41 (295 days)	9.79E-01	WIPP-Solubility-9, p.41 (295 days)	1.32E+00	WIPP-Solubility-9, p.52 (295 days)
FeMgNa2EDTA-6-2	7.27E-02		9.79E-01		1.33E+00	
FeMgNa2EDTA-6-3	6.32E-02		9.71E-01		1.27E+00	
Average	**6.84E-02**		**9.76E-01**		**1.31E+00**	
2*σ	**9.61E-03**		**9.24E-03**		**5.44E-02**	

3.12 Solubility of $Fe(OH)_2(s)$ in $Na_3C_6H_5O_7 \cdot 2H_2O$ ($Na_3Citrate \cdot 2H_2O$) solutions (the "Na^+—$FeCit^-$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $Fe(OH)_2(s)$ (ferrous iron hydroxide) in $Na_3C_6H_5O_7 \cdot 2H_2O$ ($Na_3Citrate \cdot 2H_2O$, trisodium citrate) solutions, and (2) determine the Pitzer ion-interaction parameters for ion pair, "Na^+—$FeCit^-$(aq)" (Item 12, Table 1-1).

Ferrous iron hydroxide, $Fe(OH)_2(s)$, was synthesized; 0.256 mol of $FeCl_2 \cdot 4H_2O$ (Fisher) was dissolved and mixed with 98 mL of 5.2 M NaOH (Fisher) (WIPP-Solubility-5, page 52). XRD is given in WIPP-Solubility-5, page 77. $Fe(OH)_2(s)$ was added to trisodium citrate dihydrate (Fisher) solutions of incremental concentrations, ranging from 0.01 to 1.87 m of trisodium citrate (Table 3-21). Since solubility is independent of the solid mass and experimental set-up volume as long as solid remains upon equilibrium, the mass of $Fe(OH)_2$ and the volume of trisodium citrate solutions were not determined for all experimental set-ups.

The experimental set-ups were periodically monitored for pH and dissolved ferrous iron concentration. Once stable readings of pH and dissolved ferrous iron concentrations are achieved, the concentrations of other dissolved components (sodium, citrate) were to be measured. Experimental results gathered to date are shown in Table 3-22.

Table 3-21 Preparation of experimental set-ups for the solubility of $Fe(OH)_2(s)^*$ in $Na_3C_6H_5O_7 \cdot 2H_2O$ solutions (the "Na^+—$FeCit^-$" experiment).

Set-up ID	$Na_3C_6H_5O_7$ (m)	Reference
FeCitNa-1-(1,2,3)	0.0100	WIPP-Solubility-5, p.60-61
FeCitNa-2-(1,2,3)	0.0991	WIPP-Solubility-5, p.60-61
FeCitNa-3-(1,2,3)	0.493	WIPP-Solubility-5, p.60-61
FeCitNa-4-(1,2,3)	0.968	WIPP-Solubility-5, p.60-61
FeCitNa-5-(1,2,3)	1.43	WIPP-Solubility-5, p.60-61
FeCitNa-6-(1,2,3)	1.87	WIPP-Solubility-5, p.60-61

* Mass of solid not determined. See second paragraph above for reason.

Table 3-22 Measured data for the solubility of $Fe(OH)_2(s)$ in $Na_3C_6H_5O_7 \cdot 2H_2O$ solutions (the "Na^+—$FeCit^-$" experiment).

Set-up ID	pH* (1 day)	pH* (8 days)	pH* (26 days)	pH* (68 days)	pH* (97 days)	pH* (223 days)
FeCitNa-1-1	10.300	10.505	10.519	10.561	10.544	10.564
FeCitNa-1-2	10.327	10.512	10.519	10.552	10.540	10.553
FeCitNa-1-3	10.273	10.509	10.516	10.552	10.553	10.562
Average	**10.30**	**10.51**	**10.52**	**10.56**	**10.55**	**10.56**
2*σ	**0.05**	**0.01**	**0.00**	**0.01**	**0.01**	**0.01**
FeCitNa-2-1	10.774	11.213	11.401	11.442	11.451	11.461
FeCitNa-2-2	10.566	11.119	11.346	11.409	11.421	11.444
FeCitNa-2-3	10.829	11.247	11.401	11.451	11.455	11.461
Average	**10.72**	**11.19**	**11.38**	**11.43**	**11.44**	**11.46**
2*σ	**0.28**	**0.13**	**0.06**	**0.04**	**0.04**	**0.02**
FeCitNa-3-1	10.694	11.394	11.648	11.762	11.801[a]	11.892[c]
FeCitNa-3-2	10.799	11.432	11.674	11.783	11.819[a]	11.892[c]
FeCitNa-3-3	10.873	11.506	11.755	11.858	11.886[a]	11.940[c]
Average	**10.79**	**11.44**	**11.69**	**11.80**	**11.84**	**11.91**
2*σ	**0.18**	**0.11**	**0.11**	**0.10**	**0.09**	**0.06**
FeCitNa-4-1	10.669	11.532	11.838	11.961	12.011[a]	12.121[c]
FeCitNa-4-2	9.474	11.345	11.667	11.796	11.842[a]	11.950[c]
FeCitNa-4-3	10.879	11.610	11.888	12.023	12.065[a]	12.151[c]
Average	**10.34**	**11.50**	**11.80**	**11.93**	**11.97**	**12.07**
2*σ	**1.52**	**0.27**	**0.23**	**0.23**	**0.23**	**0.22**
FeCitNa-5-1	9.176	11.438	11.869	11.985	12.004[b]	12.156[d]
FeCitNa-5-2	10.497	11.660	12.006	12.128	12.153[b]	12.286[d]
FeCitNa-5-3	8.833	11.477	11.847	11.983	12.027[b]	12.168[d]
Average	**9.50**	**11.53**	**11.91**	**12.03**	**12.06**	**12.20**
2*σ	**1.76**	**0.24**	**0.17**	**0.17**	**0.16**	**0.14**
FeCitNa-6-1	8.938	11.615	12.004	12.136	12.163[b]	12.319[d]
FeCitNa-6-2	8.658	11.622	11.993	12.143	12.169[b]	12.354[d]
FeCitNa-6-3	8.859	11.691	12.046	12.194	12.220[b]	12.373[d]
Average	**8.82**	**11.64**	**12.01**	**12.16**	**12.18**	**12.35**
2*σ	**0.29**	**0.08**	**0.06**	**0.06**	**0.06**	**0.05**
Reference	WIPP-Solubility-5, p.61	WIPP-Solubility-5, p.67	WIPP-Solubility-5, p.67	WIPP-Solubility-5, p.67	WIPP-Solubility-5, p.67	WIPP-Solubility-5, p.67

* Measured with pH electrode and meter; [a] 104 days; [b] 105 days; [c] 230 days; [d] 236 days.

Table 3-22(continued) Measured data for the solubility of $Fe(OH)_2(s)$ in $Na_3C_6H_5O_7 \cdot 2H_2O$ solutions (the "Na^+—$FeCit^-$" experiment).

	$[Fe(II)]_{diss}$, mol/L					
Set-up ID	(97 day)	(104 days)	(105 days)	(223 days)	(231 days)	(237 days)
FeCitNa-1-1	6.25E-04	-	-	6.58E-04	-	-
FeCitNa-1-2	6.97E-04	-	-	7.33E-04	-	-
FeCitNa-1-3	5.94E-04	-	-	6.13E-04	-	-
Average	**6.39E-04**	-	-	**6.68E-04**	-	-
2*σ	**1.06E-04**	-	-	**1.21E-04**	-	-
FeCitNa-2-1	4.26E-03	-	-	4.36E-03	-	-
FeCitNa-2-2	3.99E-03	-	-	4.15E-03	-	-
FeCitNa-2-3	4.27E-03	-	-	4.39E-03	-	-
Average	**4.17E-03**	-	-	**4.30E-03**	-	-
2*σ	**3.14E-04**	-	-	**2.65E-04**	-	-
FeCitNa-3-1	-	1.06E-02	-	-	1.22E-02	-
FeCitNa-3-2	-	1.08E-02	-	-	1.24E-02	-
FeCitNa-3-3	-	1.24E-02	-	-	1.37E-02	-
Average	-	**1.13E-02**	-	-	**1.28E-02**	-
2*σ	-	**1.97E-03**	-	-	**1.55E-03**	-
FeCitNa-4-1	-	1.53E-02	-	-	1.80E-02	-
FeCitNa-4-2	-	1.07E-02	-	-	1.25E-02	-
FeCitNa-4-3	-	1.71E-02	-	-	1.94E-02	-
Average	-	**1.44E-02**	-	-	**1.66E-02**	-
2*σ	-	**6.64E-03**	-	-	**7.28E-03**	-
FeCitNa-5-1	-	-	1.45E-02	-	-	1.69E-02
FeCitNa-5-2	-	-	1.92E-02	-	-	2.29E-02
FeCitNa-5-3	-	-	1.45E-02	-	-	1.76E-02
Average	-	-	**1.60E-02**	-	-	**1.92E-02**
2*σ	-	-	**5.40E-03**	-	-	**6.59E-03**
FeCitNa-6-1	-	-	1.72E-02	-	-	2.01E-02
FeCitNa-6-2	-	-	1.91E-02	-	-	2.04E-02
FeCitNa-6-3	-	-	2.08E-02	-	-	2.19E-02
Average	-	-	**1.91E-02**	-	-	**2.08E-02**
2*σ	-	-	**3.59E-03**	-	-	**1.85E-03**
Reference	WIPP-Solubility-7, p.51	WIPP-Solubility-7, p.54	WIPP-Solubility-7, p.55	WIPP-Solubility-7, p.79	WIPP-Solubility-7, p.79	WIPP-Solubility-7, p.80

3.13 Solubility of $Fe(OH)_2(s)$ in mixed MgHCitrate and NaCl solutions (the "Mg^{2+}—$FeCit^-$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $Fe(OH)_2(s)$ (ferrous iron hydroxide) in mixed MgHCitrate (magnesium hydrogen citrate) and NaCl (sodium chloride) solutions, and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Mg^{2+}—$FeCit^-$" (Item 13, Table 1-1).

These experiments are under preparation. The experimental set-ups will be listed in Table 3-23, and they will be periodically monitored for one or more component(s) among those listed in Table 3-24. Once stable readings are achieved for the selected component(s), concentrations of other components are to be determined.

Table 3-23 Preparation of experimental set-ups for the solubility of $Fe(OH)_2(s)$ in mixed MgHCitrate and NaCl solutions (the "Mg^{2+}—$FeCit^-$" experiment).

Set-up ID	$Fe(OH)_2(s)$ (g)	MgHCitrate (M)	Reference
No Data to Date			

Table 3-24 Measured data for the solubility of $Fe(OH)_2(s)$ in mixed MgHCitrate and NaCl solutions (the "Mg^{2+}—$FeCit^-$" experiment).

Set-up ID	pH (x days)	Mg(II) (x days)	citrate (x days)	Na(I) (x days)	Cl(-I) (x days)
Reference	No Data to Date				

3.14 Solubility of $PbCO_3$(s) in $NaHCO_3$ solutions (the "$PbCl^+$—HCO_3^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $PbCO_3$(s) (lead carbonate) in $NaHCO_3$ (sodium bicarbonate) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "$PbCl^+$—HCO_3^-" (Item 14, Table 1-1).

A known mass of $PbCO_3$ from Alfa Aesar was added to 100 mL of mixed $NaHCO_3$ and NaCl solutions as shown in Table 3-25 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, chloride, and carbonate) are to be determined. Experimental results will be shown in Table 3-26.

Table 3-25 Preparation of experimental set-ups for the solubility of $Pb(CO)_3$(s) in $NaHCO_3$ + NaCl solutions (the "$PbCl^+$—HCO_3^-" experiment).

Set-up ID	$PbCO_3$(s) (g)	$NaHCO_3$ (m)	NaCl (m)	Reference
$PbCO_3$-0.01/0.15-1	2.0029	0.0086	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-0.05/0.15-1	2.0027	0.043	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-0.5/0.15-1	2.0022	0.43	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-1.0/0.15-1	2.0032	0.86	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-1.0/0.3-1	2.0026	0.86	0.30	WIPP-Solubility-3, p. 17
$PbCO_3$-0.01/0.15-2	1.9998	0.0086	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-0.05/0.15-2	2.0037	0.043	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-0.5/0.15-2	2.0013	0.43	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-1.0/0.15-2	1.9995	0.86	0.15	WIPP-Solubility-3, p. 17
$PbCO_3$-1.0/0.3-2	2.0029	0.86	0.30	WIPP-Solubility-3, p. 17

Table 3-26 Measured data for the solubility of $Pb(CO)_3$(s) in $NaHCO_3$ + NaCl solutions (the "$PbCl^+$—HCO_3^-" experiment).

Set-up ID	pH	[Pb(II)]	[Na(I)]	$[CO_3^{2-}]_T$	[Cl(-I)]
Reference	No Data to Date				

3.15 Solubility of $PbCO_3$(s) in Na_2CO_3 solutions (the "$PbCl^+$—CO_3^{2-}" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $PbCO_3$(s) (lead carbonate) in Na_2CO_3 (sodium carbonate) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "$PbCl^+$—CO_3^{2-}" (Item 15, Table 1-1).

A known mass of $PbCO_3$ from Alfa Aesar was added to 100 mL of mixed Na_2CO_3 and NaCl solutions as shown in Table 3-27 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, chloride, and carbonate) are to be determined. Experimental results gathered to date will be shown in Table 3-28.

Table 3-27 Preparation of experimental set-ups for the solubility of $PbCO_3$(s) in Na_2CO_3 + NaCl solutions (the "$PbCl^+$—CO_3^{2-}" experiment).

Set-up ID	$PbCO_3$(s) (g)	Na_2CO_3 (m)	NaCl (m)	Reference
$PbCO_3$-0.01CO_3-1	2.0079	0.010	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-0.1CO_3-1	2.0063	0.10	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-0.5CO_3-1	2.0057	0.50	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-1.0CO_3-1	2.0057	1.0	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-1.5CO_3-1	2.0031	1.5	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-2.0CO_3-1	2.0066	2.0	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-0.01CO_3-2	2.0077	0.010	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-0.1CO_3-2	2.0074	0.10	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-0.5CO_3-2	2.0003	0.50	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-1.0CO_3-2	2.0043	1.0	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-1.5CO_3-2	2.0020	1.5	0.15	WIPP-Solubility-3, p. 24
$PbCO_3$-2.0CO_3-2	2.0033	2.0	0.15	WIPP-Solubility-3, p. 24

Table 3-28 Measured data for the solubility of $PbCO_3$(s) in Na_2CO_3 + NaCl solutions (the "$PbCl^+$—CO_3^{2-}" experiment).

Set-up ID	pH	[Pb(II)]	[Na(I)]	$[CO_3^{2-}]_T$	[Cl(-I)]
Reference	No Data to Date				

3.16 Solubility of $PbSO_4$(s) in Na_2SO_4 solutions (the "$PbCl^+$—SO_4^{2-}" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $PbSO_4$(s) (lead sulfate) in mixed Na_2SO_4 (sodium sulfate) and NaCl (sodium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "$PbCl^+$—SO_4^{2-}" (Item 16, Table 1-1).

A known mass of $PbSO_4$ from ACROS Organics was added to 100 mL of mixed Na_2SO_4 and NaCl solutions as shown in Table 3-29 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, chloride, and sulfate) are to be determined. Experimental results gathered to date will be shown in Table 3-30.

Table 3-29 Preparation of experimental set-ups for the solubility of $PbSO_4$(s) in Na_2SO_4 + NaCl solutions (the "$PbCl^+$—SO_4^{2-}" experiment).

Set-up ID	$PbSO_4$(s) (g)	Na_2SO_4 (m)	NaCl (m)	Reference
$PbSO_4$-0.01-1	2.0038	0.010	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-0.1-1	2.0029	0.10	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-0.5-1	2.0032	0.50	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-1.0-1	2.0031	1.0	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-1.5-1	2.0056	1.5	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-1.8-1	2.0035	1.8	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-0.01-2	2.0020	0.010	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-0.1-2	2.0078	0.10	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-0.5-2	2.0064	0.50	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-1.0-2	2.0048	1.0	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-1.5-2	2.0007	1.5	0.15	WIPP-Solubility-3, p. 20
$PbSO_4$-1.8-2	2.0032	1.8	0.15	WIPP-Solubility-3, p. 20

Table 3-30 Measured data for the solubility of $PbSO_4$(s) in Na_2SO_4 + NaCl solutions (the "$PbCl^+$—SO_4^{2-}" experiment).

Set-up ID	pH	[Na(1)]	$[SO_4^{2-}]_T$	[Cl(-I)]
Reference	No Data to Date			

3.17 Solubility of PbS(s) in NaHS solutions (the "$PbCl^+$—HS^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of PbS(s) (lead sulfide) in NaHS (sodium bisulfide) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "$PbCl^+$—HS^-" (Item 17, Table 1-1).

This set of experiments has not been started yet. It is planned to use PbS as a starting material. The supporting solutions will be the mixtures of NaHS and NaCl. A known mass of PbS will be added to 100 mL of mixed NaHS and NaCl solutions as shown in Table 3-31 below.

The experimental set-ups will be periodically monitored for one or more component(s) among those listed in Table 3-32. Once stable readings are achieved for the selected component(s), concentrations of other components are to be determined.

Table 3-31 Preparation of experimental set-ups for the solubility PbS(s) in mixed NaHS + NaCl solutions (the "$PbCl^+$—HS^-" experiment).

Set-up ID	PbC_2O_4(s) (g)	NaCl (m)	Reference
No Data to Date			

Table 3-32 Measured data for experiments the solubility PbS(s) in mixed NaHS + NaCl solutions (the "$PbCl^+$—HS^-" experiment).

Set-up ID	pH	[Pb(II)]	[S(-II)]	[Na(I)]	[Cl(-I)]
Reference	No Data to Date				

3.18 Solubility of PbO(s) in mixed NaCl and Mg_2EDTA solutions (the "Na^+—$PbEDTA^{2-}$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of PbO(s) (lead oxide) in mixed NaCl (sodium chloride) and Mg_2EDTA (magnesium EDTA) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Na^+—$PbEDTA^{2-}$" (Item 18, Table 1-1).

A known mass of PbO from MP Biomedicals was added to 100 mL of mixed NaCl and Mg_2EDTA solutions as shown in Table 3-33 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, chloride, magnesium, and EDTA) are to be determined. Experimental results gathered to date will be shown in Table 3-34.

Table 3-33 Preparation of experimental set-ups for the solubility of PbO(s) in mixed NaCl + Mg_2EDTA solutions (the "Na^+—$PbEDTA^{2-}$" experiment).

Set-up ID	PbO(s) (g)	NaCl (m)	Mg_2EDTA (m)	Reference
Mg_2EDTA-0.01-1	2.5251	0.010	0.20	WIPP-Solubility-3, p. 38
Mg_2EDTA-0.1-1	2.6232	0.10	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-1.0-1	2.4546	1.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-2.0-1	2.7850	2.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-3.0-1	2.5003	3.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-4.0-1	2.5380	4.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-5.0-1	2.6222	5.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-0.01-2	2.7556	0.010	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-0.1-2	2.4638	0.10	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-1.0-2	2.6651	1.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-2.0-2	2.6525	2.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-3.0-2	2.4043	3.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-4.0-2	2.4662	4.0	0.16	WIPP-Solubility-3, p. 38
Mg_2EDTA-5.0-2	2.5535	5.0	0.16	WIPP-Solubility-3, p. 38

Table 3-34 Measured data for the solubility of PbO(s) in H_2Na_2EDTA solutions (the "Na^+—$PbEDTA^{2-}$" experiment).

Set-up ID	pH	[Pb(II)]	[Cl(-I)]	[Na(I)]	[Mg(II)]	$[EDTA^{4-}]_T$
Reference	No Data to Date					

3.19 Solubility of PbO(s) in mixed $MgCl_2$ and Na_2H_2EDTA solutions (the "Mg^{2+}—$PbEDTA^{2-}$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of PbO(s) (lead oxide) in mixed $MgCl_2$ (magnesium chloride) and Na_2H_2EDTA (di-sodium di-hydrogen EDTA) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Mg^{2+}—$PbEDTA^{2-}$" (Item 19, Table 1-1).

A known mass of PbO from MP Biomedicals was added to 100 mL of mixed $MgCl_2$ and Na_2H_2EDTA solutions as shown in Table 3-35 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, magnesium, chloride, and EDTA) are to be determined. Experimental results gathered to date will be shown in Table 3-36.

Table 3-35 Preparation of experimental set-ups for the solubility of PbO(s) in mixed $MgCl_2$ and Na_2H_2EDTA solutions (the "Mg^{2+}—$PbEDTA^{2-}$" experiment).

Set-up ID	PbO(s) (g)	$MgCl_2$ (m)	Na_2H_2EDTA (m)	Reference
PbO-0.01ED-1	2.0054	0.0080	0.042	WIPP-Solubility-3, p. 35
PbO-0.1ED-1	2.0040	0.080	0.042	WIPP-Solubility-3, p. 35
PbO-1.0ED-1	2.0041	0.80	0.042	WIPP-Solubility-3, p. 35
PbO-1.5ED-1	2.0049	1.2	0.042	WIPP-Solubility-3, p. 35
PbO-2.0ED-1	2.0033	1.6	0.042	WIPP-Solubility-3, p. 35
PbO-2.5ED-1	2.0016	2.0	0.042	WIPP-Solubility-3, p. 35
PbO-0.01ED-2	2.0004	0.0080	0.042	WIPP-Solubility-3, p. 35
PbO-0.1ED-2	2.0043	0.080	0.042	WIPP-Solubility-3, p. 35
PbO-1.0ED-2	2.0056	0.80	0.042	WIPP-Solubility-3, p. 35
PbO-1.5ED-2	2.0005	1.2	0.042	WIPP-Solubility-3, p. 35
PbO-2.0ED-2	2.0069	1.6	0.042	WIPP-Solubility-3, p. 35
PbO-2.5ED-1	2.0036	2.0	0.042	WIPP-Solubility-3, p. 35

Table 3-36 Measured data for the solubility of PbO(s) in mixed $MgCl_2$ and Mg_2EDTA solutions (the "Mg^{2+}—$PbEDTA^{2-}$" experiment).

Set-up ID	pH	[Pb(II)]	[Mg(II)]	[Cl(-I)]	$[EDTA^{4-}]_T$
Reference	No Data to Date				

3.20 Solubility of PbO(s) in mixed NaCl and MgHCitrate solutions (the "Na^+—$PbCit^-$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of PbO(s) (lead oxide) in mixed NaCl (sodium chloride) and MgHCit (magnesium hydrogen citrate) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Na^+—$PbCit^-$" (Item 20, Table 1-1).

A known mass of PbO from MP Biomedicals was added to 100 mL of mixed NaCl and MgHCit solutions as shown in Table 3-37 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, magnesium, chloride, and citrate) are to be determined. Experimental results gathered to date will be shown in Table 3-38.

Table 3-37 Preparation of experimental set-ups for the solubility of PbO(s) in mixed NaCl and MgHCitrate solutions (the "Na^+—$PbCit^-$" experiment).

Set-up ID	PbO(s) (g)	NaCl (m)	MgHCit (m)	Reference
PbO-0.1-1	2.0043	0.050	0.12	WIPP-Solubility-3, p. 26
PbO-1.0-1	2.0089	0.50	0.12	WIPP-Solubility-3, p. 26
PbO-2.0-1	2.0080	1.05	0.12	WIPP-Solubility-3, p. 26
PbO-3.0-1	1.9999	1.6	0.12	WIPP-Solubility-3, p. 26
PbO-4.0-1	2.0105	2.2	0.12	WIPP-Solubility-3, p. 26
PbO-5.0-1	2.0066	2.5	0.12	WIPP-Solubility-3, p. 26

Table 3-38 Measured data for the solubility of PbO(s) in mixed NaCl and MgHCitrate solutions (the "Na^+—$PbCit^-$" experiment).

Set-up ID	pH	[Na(I)]	[Mg(II)]	[Cl(-I)]	$[citrate^{3-}]_T$
Reference	No Data to Date				

3.21 Solubility of PbO(s) in mixed $MgCl_2$ and MgHCitrate solutions (the "Mg^{2+}—$PbCit^-$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of PbO(s) (lead oxide) in mixed $MgCl_2$ (magnesium chloride) and MgHCitrate (magnesium hydrogen citrate) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Mg^{2+}—$PbCit^-$" (Item 21, Table 1-1).

A known mass of PbO from MP Biomedicals was added to 100 mL of mixed $MgCl_2$ and MgHCit solutions as shown in Table 3-39 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (magnesium, chloride, and citrate) are to be determined. Experimental results gathered to date will be shown in Table 3-40.

Table 3-39 Preparation of experimental set-ups for the solubility of PbO(s) in mixed $MgCl_2$ and MgHCitrate solutions (the "Mg^{2+}—$PbCit^-$" experiment).

Set-up ID	PbO(s) (g)	$MgCl_2$ (m)	MgHCit (m)	Reference
Pb-0.01Mg-1	2.0053	0.0050	0.12	WIPP-Solubility-3, p. 26
Pb-0.1Mg-1	2.0051	0.050	0.12	WIPP-Solubility-3, p. 26
Pb-1.0Mg-1	2.0063	0.50	0.12	WIPP-Solubility-3, p. 26
Pb-1.5Mg-1	2.0082	0.75	0.12	WIPP-Solubility-3, p. 26
Pb-2.0Mg-1	2.0078	1.0	0.12	WIPP-Solubility-3, p. 26
Pb-2.5Mg-1	2.0001	1.25	0.12	WIPP-Solubility-3, p. 26

Table 3-40 Measured data for the solubility of PbO(s) in mixed $MgCl_2$ and MgHCitrate solutions (the "Mg^{2+}—$PbCit^-$" experiment).

Set-up ID	pH	[Mg(II)]	[Cl(-I)]	$[citrate^{3-}]_T$
Reference	No Data to Date			

3.22 Solubility of $PbC_2O_4(s)$ in NaCl solutions (the "PbOx(aq)—Na^+" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $PbC_2O_4(s)$ (lead oxalate) in NaCl (sodium chloride) solutions and (2) determine Pitzer ion-interaction parameters for the ion pair, "PbOx(aq)—Na^+" (Item 22, Table 1-1).

In this set of experiments, a known mass of $PbC_2O4(s)$ was added to NaCl solutions ranging from 0.010 m to 5.0 m (see Table 3-41). These solutions were prepared with ACS reagent grade NaCl and deionized water. The solubility-controlling phase is lead oxalate (PbC_2O_4) with a purity of 99.999%, which is from Alfar Aesar.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (mainly, oxalate) are to be determined. Experimental results gathered to date are shown in Table 3-42.

Table 3-41 Preparation of experimental set-ups for the solubility of $PbC_2O_4(s)$ in NaCl solutions (the "PbOx(aq)—Na^+" experiment).

Set-up ID	$PbC_2O_4(s)$ (g)	NaCl (m)	Reference
PbOx-0.01-1	0.5080	0.010	WIPP-Solubility-3, p. 8
PbOx-0.1-1	0.5016	0.10	WIPP-Solubility-3, p. 8
PbOx-1.0-1	0.5111	1.0	WIPP-Solubility-3, p. 8
PbOx-2.0-1	0.5122	2.1	WIPP-Solubility-3, p. 8
PbOx-3.0-1	0.5059	3.2	WIPP-Solubility-3, p. 8
PbOx-4.0-1	0.4999	4.4	WIPP-Solubility-3, p. 8
PbOx-5.0-1	0.5091	5.0	WIPP-Solubility-3, p. 8
PbOx-0.01-2	0.5028	0.010	WIPP-Solubility-3, p. 8
PbOx-0.1-2	0.5114	0.10	WIPP-Solubility-3, p. 8
PbOx-1.0-2	0.5078	1.0	WIPP-Solubility-3, p. 8
PbOx-2.0-2	0.5079	2.1	WIPP-Solubility-3, p. 8
PbOx-3.0-2	0.5055	3.2	WIPP-Solubility-3, p. 8
PbOx-4.0-2	0.5060	4.4	WIPP-Solubility-3, p. 8
PbOx-5.0-2	0.5037	5.0	WIPP-Solubility-3, p. 8

Table 3-42 Measured data for the solubility of $PbC_2O_4(s)$ in NaCl solutions (the "PbOx(aq)—Na^+" experiment).

Set-up ID	pH [a] (109 days)	Pb, m [b] (109 days)	Oxalate, m [c] (109 days)
PbOx-0.01-1	6.40	2.15E-04	NA
PbOx-0.1-1	6.03	3.28E-04	NA
PbOx-1.0-1	6.55	1.15E-03	1.08E-04
		1.13E-03 [d]	
PbOx-2.0-1	6.11	2.52E-03	1.48E-03
		2.48E-03 [d]	
PbOx-3.0-1	6.14	5.87E-03	3.55E-03
		5.83E-03 [d]	
PbOx-4.0-1	6.56	1.31E-02	2.75E-03
		1.33E-02 [d]	
PbOx-5.0-1	7.12	2.41E-02	2.25E-03
		2.47E-02 [d]	
PbOx-0.01-2	6.51	2.10E-04	NA
PbOx-0.1-2	5.88	3.25E-04	NA
PbOx-1.0-2	6.53	1.16E-03	1.34E-04
		1.17E-03 [d]	
PbOx-2.0-2	6.19	3.07E-03	1.76E-03
		3.00E-03 [d]	
PbOx-3.0-2	6.23	5.91E-03	3.39E-03
		5.84E-03 [d]	
PbOx-4.0-2	6.62	1.32E-02	2.72E-03
		1.34E-02 [d]	
PbOx-5.0-2	7.15	2.39E-02	2.19E-03
		2.44E-02 [d]	
Reference	WIPP-Solubility-3 p. 28	WIPP-Solubility-3 Binder A, ICP-AES Analysis Dates 08/27/08 and 08/29/08	WIPP-Solubility-10 Supplement Binder 1, IC Analysis Dates 07/12/10 and 07/13/10

[a] Measured with pH electrode and pH meter; [b] measured with ICP-AES; [c] measured with IC; [d] denotes a replicate analysis.

3.23 Solubility of $PbC_2O_4(s)$ in $MgCl_2$ solutions (the "PbOx(aq)—Mg^{2+}" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $PbC_2O_4(s)$ (lead oxalate) in $MgCl_2$ (magnesium chloride) solutions and (2) determine Pitzer ion-interaction parameters for the ion pair, "PbOx(aq)—Mg^{2+}" (Item 23, Table 1-1).

In this set of experiments, a known mass of $PbC_2O_4(s)$ was added to $MgCl_2$ solutions ranging from 0.01 m to 2.5 m (Table 3-43). These solutions were prepared by using ACS reagent grade $MgCl_2 \cdot 6H_2O$ and deionized water. The solubility-controlling phase is lead oxalate (PbC_2O_4) with a purity of 99.999%, which is from Alfar Aesar. The pmH's can be obtained based on pH readings by applying the correction factors for $MgCl_2$ solutions determined by Hansen (2001), and they will be reported elsewhere. The total dissolved lead concentrations were determined using the ICP-AES. The oxalate concentrations were determined using the IC.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (magnesium, chloride, and oxalate) are to be determined. Experimental results gathered to date are shown in Table 3-44.

Table 3-43 Preparation of experimental set-ups for the solubility of $PbC_2O_4(s)$ in $MgCl_2$ solutions (the "PbOx(aq)—Mg^{2+}" experiment).

Set-up ID	$PbC_2O_4(s)$ (g)	$MgCl_2$, m	Reference
PbOx-0.01MG-1	0.5129	0.01	WIPP-Solubility-3, p. 9
PbOx-0.1MG-1	0.4945	0.1	WIPP-Solubility-3, p. 9
PbOx-1.0MG-1	0.5128	1.0	WIPP-Solubility-3, p. 9
PbOx-1.5MG-1	0.4999	1.5	WIPP-Solubility-3, p. 9
PbOx-2.0MG-1	0.5002	2.0	WIPP-Solubility-3, p. 9
PbOx-2.5MG-1	0.5095	2.5	WIPP-Solubility-3, p. 9
PbOx-0.01MG-2	0.5111	0.01	WIPP-Solubility-3, p. 9
PbOx-0.1MG-2	0.4988	0.1	WIPP-Solubility-3, p. 9
PbOx-1.0MG-2	0.5082	1.0	WIPP-Solubility-3, p. 9
PbOx-1.5MG-2	0.5042	1.5	WIPP-Solubility-3, p. 9
PbOx-2.0MG-2	0.5193	2.0	WIPP-Solubility-3, p. 9
PbOx-2.5MG-2	0.5093	2.5	WIPP-Solubility-3, p. 9

Table 3-44 Measured data for solubility of $PbC_2O_4(s)$ in $MgCl_2$ solutions (the "PbOx(aq)—Mg^{2+}" experiment).

Set-up ID	pH [a]			Pb, m [b]			Oxalate, m [c]		
	113 days	254 days	553 days	113 days	254 days	553 days	113 days	254 days	553 days
PbOx-0.01MG-1	6.57	6.38	6.01	4.37E-04 4.43E-04 [d]	4.27E-04 3.27E-04 [d] 3.20E-04 [d]	3.47E-04 3.37E-04	NA	NA	1.74E-03
PbOx-0.1MG-1	5.86	5.86	5.88	1.65E-03 1.69E-03 [d]	1.46E-03 1.34E-03 [d] 1.31E-03 [d]	1.42E-03 1.37E-03	NA	NA	1.85E-03
PbOx-1.0MG-1	5.36	5.22	5.04	7.05E-03 7.18E-03 [d]	7.69E-03 6.77E-03 [d] 6.77E-03 [d]	6.94E-03 6.76E-03	4.58E-03	4.34E-03	4.58E-03
PbOx-1.5MG-1	5.01	4.92	4.82	2.64E-02 2.68E-02 [d]	2.62E-02 2.38E-02 [d] 2.37E-02 [d]	2.59E-02 2.61E-02	5.10E-03	4.66E-03	4.97E-03
PbOx-2.0MG-1	4.64	4.68	4.65	9.22E-02 9.45E-02 [d]	8.50E-02 7.84E-02 [d] 7.77E-02 [d]	8.20E-02 8.18E-02	5.74E-03	4.74E-03	5.31E-03
PbOx-2.5MG-1	4.35	4.44	4.39	1.29E-01 1.21E-01 [d]	1.34E-01 1.24E-01 [d] 1.22E-01 [d]	1.27E-01 1.27E-01	6.37E-03	5.12E-03	4.17E-03
PbOx-0.01MG-2	6.08	6.19	6.22	3.63E-04 3.68E-04 [d]	3.73E-04 2.81E-04 [d] 2.71E-04 [d]	3.54E-04 3.28E-04	NA	NA	2.36E-03
PbOx-0.1MG-2	5.86	5.87	5.87	1.64E-03 1.68E-03 [d]	1.45E-03 1.06E-03 [d] 1.05E-03 [d]	1.42E-03 1.39E-03	NA	NA	2.04E-03
PbOx-1.0MG-2	5.37	5.30	5.13	7.58E-03 7.63E-03 [d]	7.52E-03 6.78E-03 [d] 6.69E-03 [d]	7.00E-03 7.07E-03	4.73E-03	4.51E-03	4.71E-03
PbOx-1.5MG-2	5.00	4.93	4.80	2.64E-02 2.69E-02 [d]	2.52E-02 2.29E-02 [d] 2.26E-02 [d]	2.42E-02 2.39E-02	5.15E-03	4.62E-03	4.95E-03

PbOx-2.0MG-2	4.62	4.67	4.65	9.16E-02 9.20E-02 [d]	8.45E-02 7.92E-02 [d] 7.84E-02 [d]	8.17E-02 8.09E-02	6.11E-03	4.36E-03	5.13E-03
PbOx-2.5MG-2	4.33	4.35	4.32	1.23E-01 1.35E-01 [d]	1.27E-01 1.19E-01 [d] 1.17E-01 [d]	1.24E-01 1.27E-01	6.21E-03	4.88E-03	5.80E-03
Reference	WIPP-Solubility-3, p. 28	WIPP-Solubility-3, p. 28 and 43	WIPP-Solubility-3, p. 83	WIPP-Solubility-3 Binder A, ICP-AES Analysis Dates 09/08/08 and 09/10/08	WIPP-Solubility-3 Binder A, ICP-AES Analysis Date 02/18/09	WIPP-Solubility-3 Binder B, ICP-AES Analysis Date 11/17/09	WIPP-Solubility-10 Supplement Binder 1, IC Analysis Dates 06/30/10 and 07/12/10	WIPP-Solubility-10 Supplement Binder 1, IC Analysis Dates 06/28/10 and 06/30/10	WIPP-Solubility-10 Supplement Binder 1, IC Analysis Dates 03/30/10

[a] Measured with pH electrode and pH meter; [b] measured with ICP-AES; [c] measured with IC; [d] denotes a replicate analysis.

3.24 Solubility of $PbC_2O_4(s)$ in mixed NaCl and $MgCl_2$ solutions (the "PbOx(aq)—Cl^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $PbC_2O_4(s)$ (lead oxalate, Alfa Aesar, puratronic, 99.999%) in mixed NaCl (sodium chloride) and $MgCl_2$ (magnesium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "PbOx(aq)—Cl^-" (Item 24, Table 1-1).

A known mass of $PbC_2O_4(s)$ was added to 100 mL of mixed NaCl and $MgCl_2$ solutions ranging from as shown in Table 3-45 below.

The experimental set-ups are periodically monitored for pH and dissolved lead. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, magnesium, chloride, and oxalate) are to be determined. Experimental results gathered to date will be shown in Table 3-46.

Table 3-45 Preparation of experimental set-ups for the solubility of $PbC_2O_4(s)$ in mixed NaCl and $MgCl_2$ solutions (the "PbOx(aq)—Cl^-" experiment).

Set-up ID	$PbC_2O_4(s)$ (g)	NaCl (m)	$MgCl_2$ (m)	Reference
PbOx-$MgCl_2$-A-1	0.9331	5.0	0.50	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-B-1	1.1460	3.5	0.80	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-C-1	1.1885	3.0	1.25	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-D-1	1.2549	2.0	1.5	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-E-1	0.7271	1.5	1.75	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-F-1	0.6420	0.50	2.0	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-A-2	1.3690	5.0	0.50	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-B-2	1.2838	3.5	0.80	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-C-2	1.2200	3.0	1.25	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-D-2	1.4222	2.0	1.5	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-E-2	0.5815	1.5	1.75	WIPP-Solubility-3, p. 40
PbOx-$MgCl_2$-F-2	0.6298	0.50	2.0	WIPP-Solubility-3, p. 40

Table 3-46 Measured data for the solubility of $PbC_2O_4(s)$ in mixed NaCl and $MgCl_2$ solutions (the "PbOx(aq)—Cl^-" experiment).

Set-up ID	pH	[Na(I)]	[Mg(II)]	[Cl(-I)]	$[Ox^{2-}]_T$
Reference	No Data to Date				

3.25 Solubility of $Na_2B_4O_7 \cdot 10H_2O(s)$ in NaHS solutions (the "Na^+—HS^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $Na_2(B_4O_7)(s)$ (sodium tetraborate) in NaHS (sodium bisulfide) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Na^+—HS^-" (Item 25, Table 1-1).

This set of experiments has not been started yet. It is planned to use $Na_2B_4O_7 \cdot 10H_2O$ as a starting material. The supporting solutions will be NaHS solution. A known mass of $Na_2B_4O_7 \cdot 10H_2O$ will be added to 100 mL of NaHS solutions as to be shown in Table 3-47 below.

The experimental set-ups are periodically monitored for pH and dissolved sodium. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, borate, and sulfide) are to be determined. Experimental results gathered to date will be shown in Table 3-48.

Table 3-47 Preparation of experimental set-ups for the solubility of $Na_2(B_4O_7)(s)$ in NaHS solutions (the "Na^+—HS^-" experiment).

Set-up ID	$Na_2(B_4O_7)(s)$ (g)	NaHS(m)	Reference
No Data to Date			

Table 3-48 Measured data for the solubility of $Na_2(B_4O_7)(s)$ in NaHS solutions (the "Na^+—HS^-" experiment).

Set-up ID	pH	[Na(I)]	[B(IV)]	[S(-II)]
Reference	No Data to Date			

3.26 Solubility of $Mg(OH)_2(s)$ in $NaHS/Na_2S$ solutions (the "Mg^{2+}–HS^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of $Mg(OH)_2$ (magnesium hydroxide) in $NaHS/Na_2S$ solutions, and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Mg^{2+}–HS^-" (Item 26, Table 1-1). Experimental set-ups will be listed in Table 3-49, and they will be periodically monitored for one or more component(s) among those listed in Table 3-50. Once stable readings are achieved for the selected component(s), concentrations of other components are to be determined.

Table 3-49 Preparation of experimental set-ups for the solubility of $Mg(OH)_2(s)$ in $NaHS/Na_2S$ solutions (the "Mg^{2+}–HS^-" experiment).

Set-up ID	$Mg(OH)_2(s)$ (g)	$NaHS/Na_2S$ (m)	Reference
No Data to Date			

Table 3-50 Measured data for the solubility of $Mg(OH)_2(s)$ in $NaHS/Na_2S$ solutions (the "Mg^{2+}–HS^-" experiment).

Set-up ID	pH	[Mg(II)]	[S(-II)]	[Na(I)]
Reference	No Data to Date			

3.27 Solubility of PbS(s) in NaCl solutions (the "Cl^-—HS^-" experiment)

The objectives of this set of experiments are to (1) determine the solubility of PbS(s) (lead sulfide) in NaCl (sodium chloride) solutions, and (2) determine the Pitzer ion-interaction parameters for the ion pair, "Cl^-—HS^-" (Item 27, Table 1-1). This investigation is under preparation. The preparation of experimental set-ups will be shown in Table 3-51, and measured data will be listed in Table 3-52.

Table 3-51 Preparation of experimental set-ups for the solubility of PbS(s) in NaCl solutions (the "Cl^-—HS^-" experiment).

Set-up ID	PbS(s) (g)	HCl (m)	Reference
No Data to Date			

Table 3-52 Measured data for the solubility of PbS(s) in NaCl solutions (the "Cl^-—HS^-" experiment).

Set-up ID	pH	[Pb(II)]	[S(-II)]	[Cl(-I)]
Reference	No Data to Date			

3.28 Solubility of $FeC_2O_4 \cdot 2H_2O$(s) in mixed $MgCl_2$ and NaCl solutions (the "FeOx(aq)—Mg^{2+}—Na^+" experiment).

The objectives of this set of experiments are to (1) measure solubility of $FeC_2O_4 \cdot 2H_2O$(s) (ferrous iron oxalate) in mixed $MgCl_2$ (magnesium chloride) and NaCl (sodium chloride) solutions, and (2) to determine the Pitzer ion-interaction parameters for the ion triplet "FeOx(aq)–Mg^{+2}–Na^+". This experiment is an addition to TP-08-02 and this triplet is not listed in Table 1-1. This triplet is listed in Table 1-2 (Item 28, Table 1-2).

Ferrous oxalate dihydrate ($FeC_2O_4 \cdot 2H_2O$) was purchased from Alfa Aesar (puratronic, 99.999 % metal basis), and magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) and sodium chloride (NaCl) were purchased from Fisher. A known mass of $FeC_2O_4 \cdot 2H_2O$ was added to 50 mL of mixed $MgCl_2$ and NaCl solutions as shown in Table 3-53 below.

The experimental set-ups were periodically monitored for pH. Once stable pH readings were achieved, the concentrations of other dissolved components (ferrous iron, magnesium, sodium, and chloride) were or are measured. Experimental results gathered to date are shown in Table 3-54.

Table 3-53 Preparation of experimental set-ups for the solubility of $FeC_2O_4 \cdot 2H_2O$(s) in mixed $MgCl_2$ and NaCl solutions (the "FeOx(aq)—Mg^{2+}—Na^+" experiment).

Set-up ID	$FeC_2O_4 \cdot 2H_2O$(s) (g)	$MgCl_2$ (m)	NaCl (m)	Reference
FeOxMgNa-1-1	0.1495			
FeOxMgNa-1-2	0.1056	0.479	4.83	WIPP-Solubility-7, p.47
FeOxMgNa-1-3	0.1167			
FeOxMgNa-2-1	0.1599			
FeOxMgNa-2-2	0.1447	0.752	3.24	WIPP-Solubility-7, p.47
FeOxMgNa-2-3	0.1197			
FeOxMgNa-3-1	0.1440			
FeOxMgNa-3-2	0.1276	1.11	2.64	WIPP-Solubility-7, p.47
FeOxMgNa-3-3	0.1177			
FeOxMgNa-4-1	0.1377			
FeOxMgNa-4-2	0.1511	1.30	1.76	WIPP-Solubility-7, p.47
FeOxMgNa-4-3	0.1112			
FeOxMgNa-5-1	0.1876			
FeOxMgNa-5-2	0.1323	1.47	1.28	WIPP-Solubility-7, p.47
FeOxMgNa-5-3	0.1050			
FeOxMgNa-6-1	0.1269			
FeOxMgNa-6-2	0.1390	1.65	0.545	WIPP-Solubility-7, p.47
FeOxMgNa-6-3	0.1283			

Table 3-54 Measured data for the solubility of $FeC_2O_4 \cdot 2H_2O(s)$ in mixed $MgCl_2$ and NaCl solutions (the "FeOx(aq)—Mg^{2+}—Na^{+}" experiment).

Set-up ID	pH* (9 day)	pH* (28 days)	pH* (29 days)	pH* (80 days)	pH* (83 days)	pH* (84 days)
FeOxMgNa-1-1	6.391	6.272	-	5.529	-	-
FeOxMgNa-1-2	6.473	6.202	-	5.755	-	-
FeOxMgNa-1-3	6.325	6.289	-	5.729	-	-
Average	**6.40**	**6.25**	-	**5.67**	-	-
2*σ	**0.15**	**0.09**	-	**0.25**	-	-
FeOxMgNa-2-1	6.438	6.324	-	-	5.610	-
FeOxMgNa-2-2	6.637	6.417	-	-	5.914	-
FeOxMgNa-2-3	6.665	6.514	-	-	6.001	-
Average	**6.58**	**6.42**	-	-	**5.84**	-
2*σ	**0.25**	**0.19**	-	-	**0.41**	-
FeOxMgNa-3-1	5.925	-	5.694	-	5.347	-
FeOxMgNa-3-2	6.076	-	5.823	-	5.704	-
FeOxMgNa-3-3	6.149	-	5.916	-	5.743	-
Average	**6.05**	-	**5.81**	-	**5.60**	-
2*σ	**0.23**	-	**0.22**	-	**0.44**	-
FeOxMgNa-4-1	6.347	-	6.050	-	-	5.648
FeOxMgNa-4-2	6.376	-	6.084	-	-	5.824
FeOxMgNa-4-3	6.463	-	6.111	-	-	5.933
Average	**6.40**	-	**6.08**	-	-	**5.80**
2*σ	**0.12**	-	**0.06**	-	-	**0.29**
FeOxMgNa-5-1	5.734	-	5.458	-	5.554	-
FeOxMgNa-5-2	6.021	-	5.703	-	5.687	-
FeOxMgNa-5-3	6.070	-	5.784	-	5.755	-
Average	**5.94**	-	**5.65**	-	**5.67**	-
2*σ	**0.36**	-	**0.34**	-	**0.20**	-
FeOxMgNa-6-1	5.795	-	5.472	-	5.216	-
FeOxMgNa-6-2	5.783	-	5.426	-	5.342	-
FeOxMgNa-6-3	5.817	-	5.463	-	5.415	-
Average	**5.80**	-	**5.45**	-	**5.32**	-
2*σ	**0.03**	-	**0.05**	-	**0.20**	-
Reference	WIPP-Solubility-7, p.48	WIPP-Solubility-7, p.48	WIPP-Solubility-7, p.48	WIPP-Solubility-7, p.48	WIPP-Solubility-7, p.48	WIPP-Solubility-7, p.48

* Measured with pH electrode and meter.

3.29 Solubility of $Ca_3[Citrate]_2 \cdot 4H_2O(s)$ in NaCl solutions (the "Na^+—$CaCit^-$" experiment)

The objectives of this set of experiments are to (1) determine the solubility of earlandite ($Ca_3[Citrate]_2 \cdot 4H_2O(s)$) in NaCl (sodium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "Na^+—$CaCit^-$" (Item 29, Table 1-1).

A known mass of earlandite (calcium citrate tribasic tetrahydrate) from ACROS Organics was added to 100 mL of NaCl solutions as shown in Table 3-55 below.

The experimental set-ups are periodically monitored for pH and dissolved calcium. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, calcium, chloride, and citrate) are to be determined. Experimental results gathered to date will be shown in Table 3-56.

Table 3-55 Preparation of experimental set-ups for the solubility of $Ca_3[Citrate]_2 \cdot 4H_2O(s)$ in NaCl solutions (the "Na^+—$CaCit^-$" experiment).

Set-up ID	Earlandite (g)	NaCl (m)	Reference
ACROS-ELDT-0.01-1	2.0197	0.010	WIPP-Solubility-3, p. 62
ACROS-ELDT-0.1-1	2.0176	0.10	WIPP-Solubility-3, p. 62
ACROS-ELDT-1.0-1	2.0128	1.0	WIPP-Solubility-3, p. 62
ACROS-ELDT-2.0-1	2.0162	2.1	WIPP-Solubility-3, p. 62
ACROS-ELDT-3.0-1	2.0146	3.2	WIPP-Solubility-3, p. 62
ACROS-ELDT-5.0-1	2.0108	5.0	WIPP-Solubility-3, p. 62
ACROS-ELDT-0.01-2	2.0100	0.010	WIPP-Solubility-3, p. 62
ACROS-ELDT-0.1-2	2.0199	0.10	WIPP-Solubility-3, p. 62
ACROS-ELDT-1.0-2	2.0254	1.0	WIPP-Solubility-3, p. 62
ACROS-ELDT-2.0-2	2.0198	2.1	WIPP-Solubility-3, p. 62
ACROS-ELDT-3.0-2	2.0057	3.2	WIPP-Solubility-3, p. 62
ACROS-ELDT-5.0-2	2.0009	5.0	WIPP-Solubility-3, p. 62

Table 3-56 Measured data for the solubility of $Ca_3[Citrate]_2 \cdot 4H_2O(s)$ in NaCl solutions (the "Na^+—$CaCit^-$" experiment).

Set-up ID	pH	[Ca(II)]	[Na(I)]	$[citrate^{3-}]_T$
Reference	No Data to Date			

3.30 Solubility of $Ca_2EDTA(s)$ in NaCl solutions (the "Na^+—$HEDTA^{3-}$" experiment).

The objectives of this set of experiments are to (1) determine the solubility of Ca_2EDTA (calcium EDTA) in NaCl (sodium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "Cl^-—$HEDTA^{3-}$" (Item 30, Table 1-1).

A known mass of Ca_2EDTA from ACROS Organics was added to 100 mL of NaCl solutions as shown in Table 3-57 below.

The experimental set-ups are periodically monitored for pH and dissolved calcium. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, chloride, and EDTA) are to be determined. Experimental results gathered to date will be shown in Table 3-58.

Table 3-57 Preparation of experimental set-ups for the solubility of $Ca_2EDTA(s)$ in NaCl solutions (the "Na^+—$HEDTA^{3-}$" experiment).

Set-up ID	$Ca_2EDTA(s)$ (g)	NaCl (m)	Reference
Ca_2EDTA-0.01-1	2.0070	0.010	WIPP-Solubility-3, p. 9-10
Ca_2EDTA-0.1-1	2.0075	0.10	WIPP-Solubility-3, p. 9-10
Ca_2EDTA-1.0-1	4.0081	1.0	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-2.0-1	4.0088	2.1	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-3.0-1	4.0094	3.2	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-4.0-1	4.0093	4.4	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-5.0-1	2.0034	5.0	WIPP-Solubility-3, p. 9-10
Ca_2EDTA-0.01-2	2.0029	0.010	WIPP-Solubility-3, p. 9-10
Ca_2EDTA-0.1-2	2.0052	0.10	WIPP-Solubility-3, p. 9-10
Ca_2EDTA-1.0-2	4.0084	1.0	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-2.0-2	4.0130	2.1	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-3.0-2	4.0072	3.2	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-4.0-2	4.0087	4.4	WIPP-Solubility-3, p. 9-10, and p. 19
Ca_2EDTA-5.0-2	2.0011	5.0	WIPP-Solubility-3, p. 9-10

Table 3-58 Measured data for the solubility of $Ca_2EDTA(s)$ in NaCl solutions (the "Na^+—$HEDTA^{3-}$" experiment).

Set-up ID	pH	[Ca(II)]	[Na(I)]	$[EDTA^{4-}]_T$	[Cl(-I)]
Reference	No Data to Date				

3.31 Solubility of $Ca_2EDTA(s)$ in $MgCl_2$ solutions (the "Mg^{2+}—$HEDTA^{3-}$" experiment).

The objectives of this set of experiments are to (1) determine the solubility of Ca_2EDTA (calcium EDTA) in NaCl (sodium chloride) solutions and (2) determine the Pitzer ion-interaction parameters for the ion pair "Mg^{2+}—$HEDTA^{3-}$" (Item 31, Table 1-1).

A known mass of Ca_2EDTA from ACROS Organics was added to 100 mL of $MgCl_2$ (Fisher) solutions as shown in Table 3-59 below.

The experimental set-ups are periodically monitored for pH and dissolved calcium. Once stable pH readings are achieved, concentrations of other dissolved components (sodium, magnesium, chloride, and EDTA) are to be determined. Experimental results gathered to date will be shown in Table 3-60.

Table 3-59 Preparation of experimental set-ups for the solubility of Ca_2EDTA (s) in NaCl solutions (the "Mg^{2+}—$HEDTA^{3-}$" experiment).

Set-up ID	$Ca_2EDTA(s)$ (g)	$MgCl_2$ (m)	Reference
Ca_2EDTA-0.01$MgCl_2$-1	2.0044	0.010	WIPP-Solubility-3, p. 12
Ca_2EDTA-0.1$MgCl_2$-1	4.0031	0.10	WIPP-Solubility-3, p. 12, and 15
Ca_2EDTA-1.0$MgCl_2$-1	6.0084	1.0	WIPP-Solubility-3, p. 12, 15, and 16
Ca_2EDTA-1.5$MgCl_2$-1	8.0128	1.5	WIPP-Solubility-3, p. 12, 15, 16, and 17
Ca_2EDTA-2.0$MgCl_2$-1	8.0131	2.0	WIPP-Solubility-3, p. 12, 15, 16, and 17
Ca_2EDTA-2.5$MgCl_2$-1	10.0131	2.5	WIPP-Solubility-3, p. 12, 15, 16, 17, and 19
Ca_2EDTA-0.01$MgCl_2$-2	2.0044	0.010	WIPP-Solubility-3, p. 12
Ca_2EDTA-0.1$MgCl_2$-2	4.0062	0.10	WIPP-Solubility-3, p. 12, and 15
Ca_2EDTA-1.0$MgCl_2$-2	6.0074	1.0	WIPP-Solubility-3, p. 12, 15, and 16
Ca_2EDTA-1.5$MgCl_2$-2	8.0087	1.5	WIPP-Solubility-3, p. 12, 15, 16, and 17
Ca_2EDTA-2.0$MgCl_2$-2	8.0078	2.0	WIPP-Solubility-3, p. 12, 15, 16, and 17
Ca_2EDTA-2.5$MgCl_2$-2	10.0159	2.5	WIPP-Solubility-3, p. 12, 15, 16, 17, and 19

Table 3-60 Measured data for the solubility of Ca_2EDTA (s) in $MgCl_2$ solutions (the "Mg^{2+}—$HEDTA^{3-}$" experiment).

Set-up ID	pH	[Ca(II)]	[Mg(II)]	$[EDTA^{4-}]_T$	[Cl(-I)]
Reference	No Data to Date				

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Kim, Sungtae Sungtae Kim 5/24/2011

From: Nemer, Martin B
Sent: Thursday, May 19, 2011 3:27 PM
To: Kim, Sungtae
Subject: LDRD

Sorry I haven't gotten back to you. Swamped with work. I will try to find a name for you.

You have my permission to sign the milestone report on my behalf.

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