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First End-of-year Report on Test Plan TP 20-01 Revision 0, "Solubility and Complexation of Lead in Non-Sulfidic Brines of WIPP-Relevance"

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ABBREVIATIONS AND ACRONYMS

Abbreviation or Acronym	Definition
CO_2	carbon dioxide
DI	deionized
DDI	de-aerated deionized
ICDD	the International Centre for Diffraction Data
ID	identification
m	molality (mol/kg H_2O)
M	Molarity (mol/L)
mL	milli-Liter
N_2	nitrogen gas
NaCl	sodium chloride
$NaPb_2(CO_3)_2(OH)$	Abellaite
O_2	oxygen
Pb	lead
$PbCl_2$	Cotunnite
PbCl(OH)	Laurionite
$PbCO_3$	Cerussite
PbO	Litharge
$Pb_8O_5(OH)_2Cl_4$	Blixite
$PbSO_4$	Anglesite
PDF	Powder Diffraction File
pH	negative logarithm of hydrogen ion activity
QA	Quality Assurance
s	solid
SNL	Sandia National Laboratories
SP	Specific Procedure
TP	Test Plan
MΩ ·cm	Megohm per centimeter
WIPP	Waste Isolation Pilot Plant

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XRD X-ray Diffractometer

1.0 INTRODUCTION

The purpose of this end-of-year report is to summarize the progress completed to date for tasks related to Test Plan (TP) 20-01 Rev. 0 (Kirkes and Zhang, 2020). The goal of TP 20-01 Rev. 0 is to measure the solubility and complexation of lead in non-sulfidic brines of WIPP-relevance.

A previous TP, i.e., TP 08-02 Rev. 0 (Ismail et al., 2008), was initiated in 2008 for this goal however upon conducting final solid characterization of the experiments, formation of secondary lead solids were observed that are different from the lead solids used to initiate the solubility experiments. Lead solids such as abellaite ($NaPb_2(CO_3)_2(OH)(s)$), blixite ($Pb_8O_5(OH)_2Cl_4(s)$), and laurionite ($PbCl(OH)(s)$) were identified and documented (Kirkes and Xiong, 2019) as "unexpected" because they were not in the thermodynamic database used to calculate the equilibria. The observation of the "unexpected" lead solids indicates that we need to determine the solubility of the secondary lead solids to improve our model prediction capability by performing analytical and sequential experiments for the solids, as well as by referring to the literature information. Most of experimental work for lead under TP 08-02 Rev. 0 was on hold, and TP 20-01 Rev. 0 was initiated to experimentally evaluate the usability of the unexpected results obtained from TP 08-02 Rev. 0 (Ismail et al., 2008). It is possible that these unexpected results were a result of not maintaining the experimental environment, such as intermittent exposure of the reactors to CO_2 in the air. Unlike TP 08-02 Rev. 0, experiments in TP 20-01 Rev. 0 are prepared in a CO_2-free gloveboxes to ensure sample preservation. The results obtained from TP 20-01 Rev. 0 experiments will be used to evaluate the results from TP 08-02 Rev. 0. Analysis reports and/or memoranda will be written to compare and evaluate the data obtained from the two mentioned test plans.

The objectives of TP 20-01 Rev. 0 are the following: (i) to determine the implication of adopting sample preservation technique (i.e., CO_2-free gloveboxes), (ii) to produce experimental data for the solubility and complexation of lead in the WIPP relevant brines in

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non-sulfide systems, and (iii) to determine when the secondary lead-bearing solid phase would form under CO_2-free, anoxic conditions. The results of solubility measurement and solid characterization obtained under this TP are to be used to determine the thermodynamic quantities, i.e., equilibrium constants and Pitzer interaction parameters, needed to update the thermodynamic database to include the secondary lead solids observed from the experiments performed under TP 08-02 Rev. 0.

The following tasks have been identified for completion in this study as per TP 20-01 Rev. 0:

- *Task 1: Synthesize and Characterize Solid Starting Materials:* 14 subtasks were designed to identify/synthesize a source of individual lead-bearing solids used in Task 2 and to perform solid characterization.
- *Task 2: Long-term Solubility Experiments:* Setup experimental measurement of the solubility of each of individual lead solids in different brine matrixes: NaCl (0 – 6.0 m), $MgCl_2$ (0 - 6 m) or mixed. A total of 42 subtasks were designed to run for long time with sampling at different intervals.
- *Task 3: Scoping Experiments:* These experiments were designed to determine, in short-term, the solubility of $PbCl_2$(s) and PbO(s) in brines made of DDI water, HCl, NaOH, NaCl, and $MgCl_2$ and the complexation of lead with EDTA.

2.0 MATERIALS AND METHODS

All solutions were prepared from reagent grade chemicals and de-aerated DI (DDI) water. All DI water used for experimental set-up had a resistivity of ≥18 MΩ ·cm. The DI water for experiments was degassed inside a glove box, using pre-purified inert gas (e.g., N_2) to minimize the concentration of any potentially dissolved CO_2, O_2 and other gases. All solutions and experimental set-ups were prepared inside an anoxic (Nitrogen or 95:5 Nitrogen:Hydrogen mixture filled) glove box environment.

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High purity lead solids from Fisher Scientific or analogous vendors were used for solid starting materials.

2.1 Solid Materials Characterization

Solid starting materials were analyzed using the Bruker D8 X-ray diffractometer (XRD) as per SP 12-8 Rev. 4, "Use and Maintenance of Bruker Analytical X-Ray Systems D8 Advance X-Ray Diffractometer," (Kirkes, 2020).

2.2 Ion and Anion Concentration Measurements

To date, no ion or pH data has been collected on this TP.

3.0 RESULTS

The following sections describe the experimental preparations completed by the cut-off date of 9/14/2020.

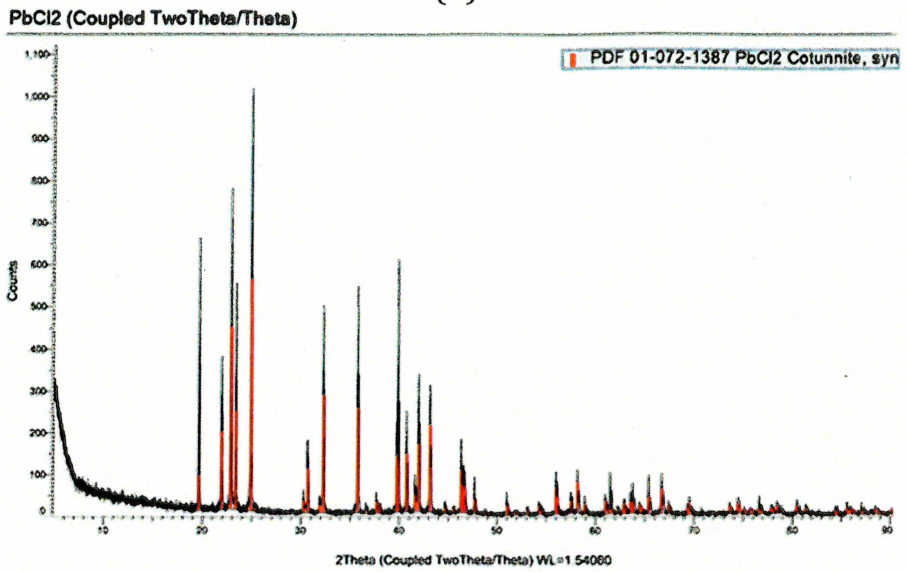
3.1 Task 1: Synthesize/Obtain and Characterize Solid Starting Materials.

The lead starting solids, $PbCl_2$(s) and $PbSO_4$(s), were purchased from Fisher Scientific or analogous vendors and characterized by XRD (See Figures 1 and 2).

XRD scans were performed to check and verify the quality of the lead solids used in this TP. Figures 1 and 2 present the XRD patterns of lead solids, $PbCl_2$(s) and $PbSO_4$(s), respectively. They match, at each peak in the patterns, with PDF patterns 01-072-1387 for Cotunnite and 01-089-7356 for Anglesite available in PDF-4+ database from ICDD (the International Centre for Diffraction Data)

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(A)



(B)

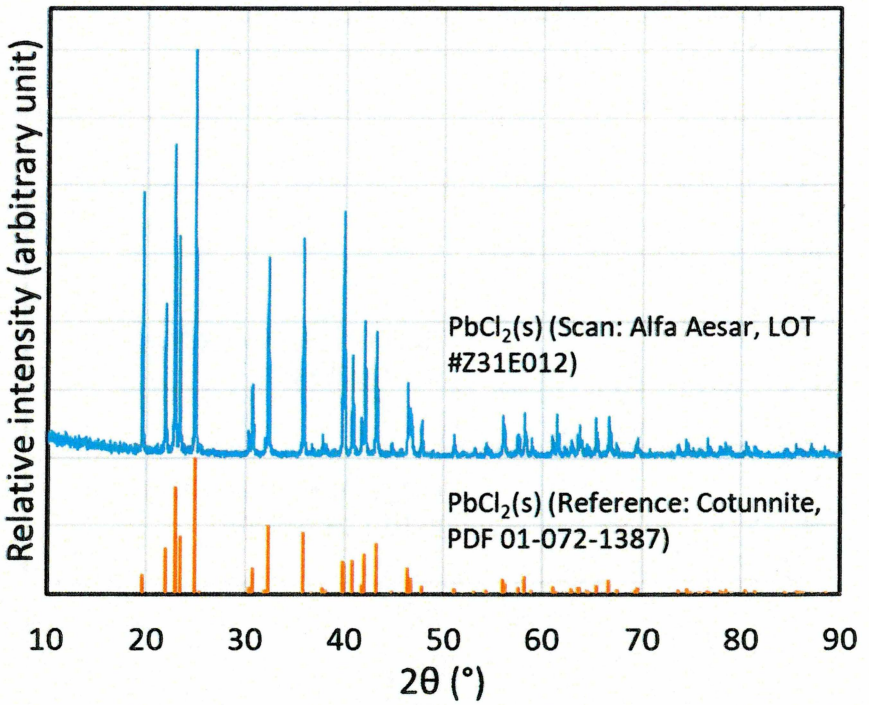
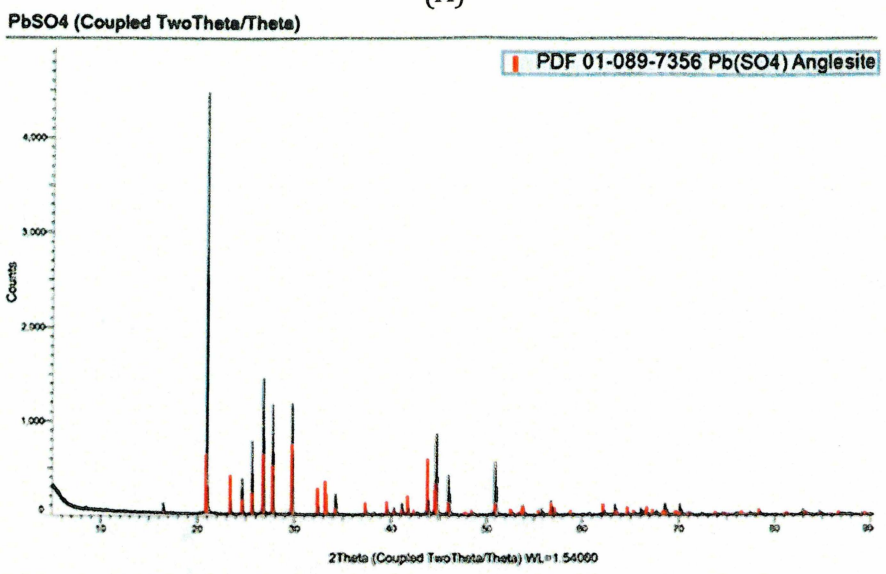


Figure 1. XRD Patterns for the starting solid $PbCl_2(s)$ (Alfa Aesar, LOT Z31E012): (A) Screenshot of the print-out that the XRD software generated (WIPP-Pb-1, page 4), (B) Same scan as in (A) reproduced using Microsoft Excel to present scan and reference peak locations separately for clarity. The Microsoft Excel file, named "000_XRD_Pb_TP2001.xlsx", is located in "/cvs/CVSLIB/WIPP_EXTERNAL/Geochem2020/milestones".

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(A)



(B)

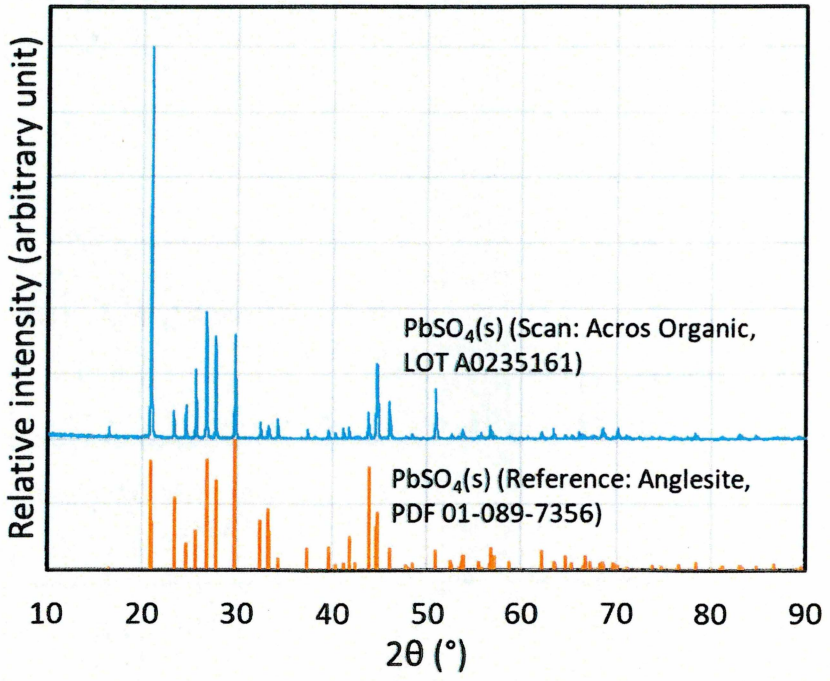


Figure 2. XRD Patterns for the starting solid $PbSO_4$(s)(Acros Organic, LOT A0235161, WIPP-Pb-1, page 5): (A) Screenshot of the print-out that the XRD software generated, (B) Same scan as in (A) reproduced using Microsoft Excel to present scan and reference peak locations separately for clarity. The Microsoft Excel file, named "000_XRD_Pb_TP2001.xlsx", is located in "/cvs/CVSLIB/WIPP_EXTERNAL/Geochem2020/milestones".

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3.2 Task 2: Long-term Solubility Experiments

Subtasks 2.1 and 2.31 in Task 2 were prepared in the lab up to the cut-off date, 9/14/2020. These experiments were set-up to determine the solubility of lead solids in various brine matrices(e.g., NaCl solution) and confirm if the individual solids are transformed during the long-term experiments at a controlled environment (anoxic condition). Table 1 shows the experimental set-up of subtask 2.1 for the solubility of cotunnite, $PbCl_2$(s). A total 15 identical glass experimental vials were prepared for each experimental ID from NaCl concentrations at 0 m (DDI water), 0.01 m, 0.1 m, 1.0 m, 2.0 m, 3.0 m, 4.0 m, 5.0 m and 6.0 m (see Table 1). For this subtask 2.1, a total of 270 vials (15 vials/condition × 9 conditions × 2 replicates=270) were prepared (See **Table 1**).

Similarly, experiments in subtask 2.31 were prepared for the solubility of anglesite ($PbSO_4$) in the NaCl solution concentrations at 0 m (DDI water), 0.01 m, 0.1 m, 1.0 m, 2.0 m, 3.0 m, 4.0 m, 5.0 m and 6.0 m (see **Table 2**).

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Table 1. Experimental set-up for the solubility of cotunnite ($PbCl_2$(s)) in NaCl matrix (0-6.0 m) (WIPP-Pb-1, pages 6-9).

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL DDI water		
1	PbCl2-NaCl-0-1-1	1.2005	PbCl2-NaCl-0-2-1	1.2002
2	PbCl2-NaCl-0-1-2	1.2033	PbCl2-NaCl-0-2-2	1.2016
3	PbCl2-NaCl-0-1-3	1.2017	PbCl2-NaCl-0-2-3	1.2014
4	PbCl2-NaCl-0-1-4	1.2010	PbCl2-NaCl-0-2-4	1.2022
5	PbCl2-NaCl-0-1-5	1.2008	PbCl2-NaCl-0-2-5	1.2001
6	PbCl2-NaCl-0-1-6	1.2001	PbCl2-NaCl-0-2-6	1.2016
7	PbCl2-NaCl-0-1-7	1.2014	PbCl2-NaCl-0-2-7	1.2013
8	PbCl2-NaCl-0-1-8	1.2017	PbCl2-NaCl-0-2-8	1.2002
9	PbCl2-NaCl-0-1-9	1.2011	PbCl2-NaCl-0-2-9	1.2010
10	PbCl2-NaCl-0-1-10	1.2015	PbCl2-NaCl-0-2-10	1.2001
11	PbCl2-NaCl-0-1-11	1.2026	PbCl2-NaCl-0-2-11	1.2008
12	PbCl2-NaCl-0-1-12	1.2052	PbCl2-NaCl-0-2-12	1.2032
13	PbCl2-NaCl-0-1-13	1.2053	PbCl2-NaCl-0-2-13	1.2024
14	PbCl2-NaCl-0-1-14	1.2013	PbCl2-NaCl-0-2-14	1.2020
15	PbCl2-NaCl-0-1-15	1.2062	PbCl2-NaCl-0-2-15	1.2014
		20 mL 0.01 m NaCl		
1	PbCl2-NaCl-0.01-1-1	1.2007	PbCl2-NaCl-0.01-2-1	1.2001
2	PbCl2-NaCl-0.01-1-2	1.2010	PbCl2-NaCl-0.01-2-2	1.2001
3	PbCl2-NaCl-0.01-1-3	1.2017	PbCl2-NaCl-0.01-2-3	1.2009
4	PbCl2-NaCl-0.01-1-4	1.2020	PbCl2-NaCl-0.01-2-4	1.2019
5	PbCl2-NaCl-0.01-1-5	1.2004	PbCl2-NaCl-0.01-2-5	1.2007
6	PbCl2-NaCl-0.01-1-6	1.2013	PbCl2-NaCl-0.01-2-6	1.2012
7	PbCl2-NaCl-0.01-1-7	1.2014	PbCl2-NaCl-0.01-2-7	1.2012
8	PbCl2-NaCl-0.01-1-8	1.2005	PbCl2-NaCl-0.01-2-8	1.2003
9	PbCl2-NaCl-0.01-1-9	1.2012	PbCl2-NaCl-0.01-2-9	1.2000
10	PbCl2-NaCl-0.01-1-10	1.2010	PbCl2-NaCl-0.01-2-10	1.2001
11	PbCl2-NaCl-0.01-1-11	1.2040	PbCl2-NaCl-0.01-2-11	1.2016
12	PbCl2-NaCl-0.01-1-12	1.2023	PbCl2-NaCl-0.01-2-12	1.2019
13	PbCl2-NaCl-0.01-1-13	1.2014	PbCl2-NaCl-0.01-2-13	1.2021
14	PbCl2-NaCl-0.01-1-14	1.2025	PbCl2-NaCl-0.01-2-14	1.2026
15	PbCl2-NaCl-0.01-1-15	1.2018	PbCl2-NaCl-0.01-2-15	1.2011

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Table 1 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL 0.1 m NaCl		
1	PbCl2-NaCl-0.1-1-1	1.1999	PbCl2-NaCl-0.1-2-1	1.2010
2	PbCl2-NaCl-0.1-1-2	1.2002	PbCl2-NaCl-0.1-2-2	1.2010
3	PbCl2-NaCl-0.1-1-3	1.2016	PbCl2-NaCl-0.1-2-3	1.1999
4	PbCl2-NaCl-0.1-1-4	1.2016	PbCl2-NaCl-0.1-2-4	1.2019
5	PbCl2-NaCl-0.1-1-5	1.2001	PbCl2-NaCl-0.1-2-5	1.2000
6	PbCl2-NaCl-0.1-1-6	1.2015	PbCl2-NaCl-0.1-2-6	1:2000
7	PbCl2-NaCl-0.1-1-7	1.2006	PbCl2-NaCl-0.1-2-7	1.2012
8	PbCl2-NaCl-0.1-1-8	1.2001	PbCl2-NaCl-0.1-2-8	1.2014
9	PbCl2-NaCl-0.1-1-9	1.2011	PbCl2-NaCl-0.1-2-9	1.2001
10	PbCl2-NaCl-0.1-1-10	1.2006	PbCl2-NaCl-0.1-2-10	1.2008
11	PbCl2-NaCl-0.1-1-11	1.2040	PbCl2-NaCl-0.1-2-11	1.2075
12	PbCl2-NaCl-0.1-1-12	1.2010	PbCl2-NaCl-0.1-2-12	1.2055
13	PbCl2-NaCl-0.1-1-13	1.2070	PbCl2-NaCl-0.1-2-13	1.2048
14	PbCl2-NaCl-0.1-1-14	1.2056	PbCl2-NaCl-0.1-2-14	1.2045
15	PbCl2-NaCl-0.1-1-15	1.2058	PbCl2-NaCl-0.1-2-15	1.2044
		20 mL 1.0 m NaCl		
1	PbCl2-NaCl-1.0-1-1	1.2008	PbCl2-NaCl-1.0-2-1	1.2008
2	PbCl2-NaCl-1.0-1-2	1.2000	PbCl2-NaCl-1.0-2-2	1.2009
3	PbCl2-NaCl-1.0-1-3	1.2019	PbCl2-NaCl-1.0-2-3	1.2003
4	PbCl2-NaCl-1.0-1-4	1.2004	PbCl2-NaCl-1.0-2-4	1.2000
5	PbCl2-NaCl-1.0-1-5	1.2013	PbCl2-NaCl-1.0-2-5	1.2014
6	PbCl2-NaCl-1.0-1-6	1.2005	PbCl2-NaCl-1.0-2-6	1.2002
7	PbCl2-NaCl-1.0-1-7	1.2020	PbCl2-NaCl-1.0-2-7	1.2015
8	PbCl2-NaCl-1.0-1-8	1.2008	PbCl2-NaCl-1.0-2-8	1.2001
9	PbCl2-NaCl-1.0-1-9	1.2002	PbCl2-NaCl-1.0-2-9	1.2001
10	PbCl2-NaCl-1.0-1-10	1.2016	PbCl2-NaCl-1.0-2-10	1.2000
11	PbCl2-NaCl-1.0-1-11	1.2018	PbCl2-NaCl-1.0-2-11	1.2038
12	PbCl2-NaCl-1.0-1-12	1.2028	PbCl2-NaCl-1.0-2-12	1.2050
13	PbCl2-NaCl-1.0-1-13	1.2047	PbCl2-NaCl-1.0-2-13	1.2009
14	PbCl2-NaCl-1.0-1-14	1.2020	PbCl2-NaCl-1.0-2-14	1.2022
15	PbCl2-NaCl-1.0-1-15	1.2019	PbCl2-NaCl-1.0-2-15	1.2012

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Table 1 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL 2.0 m NaCl		
1	PbCl2-NaCl-2.0-1-1	1.2008	PbCl2-NaCl-2.0-2-1	1.2004
2	PbCl2-NaCl-2.0-1-2	1.2006	PbCl2-NaCl-2.0-2-2	1.2019
3	PbCl2-NaCl-2.0-1-3	1.2002	PbCl2-NaCl-2.0-2-3	1.1999
4	PbCl2-NaCl-2.0-1-4	1.2007	PbCl2-NaCl-2.0-2-4	1.2024
5	PbCl2-NaCl-2.0-1-5	1.2020	PbCl2-NaCl-2.0-2-5	1.2008
6	PbCl2-NaCl-2.0-1-6	1.2005	PbCl2-NaCl-2.0-2-6	1.2008
7	PbCl2-NaCl-2.0-1-7	1.2007	PbCl2-NaCl-2.0-2-7	1.2015
8	PbCl2-NaCl-2.0-1-8	1.2015	PbCl2-NaCl-2.0-2-8	1.2013
9	PbCl2-NaCl-2.0-1-9	1.2016	PbCl2-NaCl-2.0-2-9	1.2003
10	PbCl2-NaCl-2.0-1-10	1.2007	PbCl2-NaCl-2.0-2-10	1.2014
11	PbCl2-NaCl-2.0-1-11	1.2057	PbCl2-NaCl-2.0-2-11	1.2037
12	PbCl2-NaCl-2.0-1-12	1.2079	PbCl2-NaCl-2.0-2-12	1.2053
13	PbCl2-NaCl-2.0-1-13	1.2067	PbCl2-NaCl-2.0-2-13	1.2056
14	PbCl2-NaCl-2.0-1-14	1.2014	PbCl2-NaCl-2.0-2-14	1.2027
15	PbCl2-NaCl-2.0-1-15	1.2016	PbCl2-NaCl-2.0-2-15	1.2016
		20 mL 3.0 m NaCl		
1	PbCl2-NaCl-3.0-1-1	1.2006	PbCl2-NaCl-3.0-2-1	1.2002
2	PbCl2-NaCl-3.0-1-2	1.2006	PbCl2-NaCl-3.0-2-2	1.2006
3	PbCl2-NaCl-3.0-1-3	1.2012	PbCl2-NaCl-3.0-2-3	1.2013
4	PbCl2-NaCl-3.0-1-4	1.2000	PbCl2-NaCl-3.0-2-4	1.1997
5	PbCl2-NaCl-3.0-1-5	1.2001	PbCl2-NaCl-3.0-2-5	1.2009
6	PbCl2-NaCl-3.0-1-6	1.2003	PbCl2-NaCl-3.0-2-6	1.2014
7	PbCl2-NaCl-3.0-1-7	1.2008	PbCl2-NaCl-3.0-2-7	1.2001
8	PbCl2-NaCl-3.0-1-8	1.2019	PbCl2-NaCl-3.0-2-8	1.2000
9	PbCl2-NaCl-3.0-1-9	1.2010	PbCl2-NaCl-3.0-2-9	1.2018
10	PbCl2-NaCl-3.0-1-10	1.2004	PbCl2-NaCl-3.0-2-10	1.2011
11	PbCl2-NaCl-3.0-1-11	1.2062	PbCl2-NaCl-3.0-2-11	1.2050
12	PbCl2-NaCl-3.0-1-12	1.2023	PbCl2-NaCl-3.0-2-12	1.2016
13	PbCl2-NaCl-3.0-1-13	1.2051	PbCl2-NaCl-3.0-2-13	1.2039
14	PbCl2-NaCl-3.0-1-14	1.2005	PbCl2-NaCl-3.0-2-14	1.2045
15	PbCl2-NaCl-3.0-1-15	1.2012	PbCl2-NaCl-3.0-2-15	1.2047

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Table 1 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL 4.0 m NaCl		
1	PbCl2-NaCl-4.0-1-1	1.2008	PbCl2-NaCl-4.0-2-1	1.2008
2	PbCl2-NaCl-4.0-1-2	1.2018	PbCl2-NaCl-4.0-2-2	1.2000
3	PbCl2-NaCl-4.0-1-3	1.1998	PbCl2-NaCl-4.0-2-3	1.1998
4	PbCl2-NaCl-4.0-1-4	1.1998	PbCl2-NaCl-4.0-2-4	1.2008
5	PbCl2-NaCl-4.0-1-5	1.2001	PbCl2-NaCl-4.0-2-5	1.2020
6	PbCl2-NaCl-4.0-1-6	1.2005	PbCl2-NaCl-4.0-2-6	1.2009
7	PbCl2-NaCl-4.0-1-7	1.2009	PbCl2-NaCl-4.0-2-7	1.2002
8	PbCl2-NaCl-4.0-1-8	1.2017	PbCl2-NaCl-4.0-2-8	1.2003
9	PbCl2-NaCl-4.0-1-9	1.2001	PbCl2-NaCl-4.0-2-9	1.2008
10	PbCl2-NaCl-4.0-1-10	1.2013	PbCl2-NaCl-4.0-2-10	1.2012
11	PbCl2-NaCl-4.0-1-11	1.2017	PbCl2-NaCl-4.0-2-11	1.2072
12	PbCl2-NaCl-4.0-1-12	1.2057	PbCl2-NaCl-4.0-2-12	1.2017
13	PbCl2-NaCl-4.0-1-13	1.2058	PbCl2-NaCl-4.0-2-13	1.2042
14	PbCl2-NaCl-4.0-1-14	1.2027	PbCl2-NaCl-4.0-2-14	1.2016
15	PbCl2-NaCl-4.0-1-15	1.2013	PbCl2-NaCl-4.0-2-15	1.2019
		20 mL 5.0 m NaCl		
1	PbCl2-NaCl-5.0-1-1	1.2004	PbCl2-NaCl-5.0-2-1	1.2015
2	PbCl2-NaCl-5.0-1-2	1.2025	PbCl2-NaCl-5.0-2-2	1.2016
3	PbCl2-NaCl-5.0-1-3	1.2016	PbCl2-NaCl-5.0-2-3	1.2024
4	PbCl2-NaCl-5.0-1-4	1.2018	PbCl2-NaCl-5.0-2-4	1.2000
5	PbCl2-NaCl-5.0-1-5	1.2018	PbCl2-NaCl-5.0-2-5	1.2006
6	PbCl2-NaCl-5.0-1-6	1.2015	PbCl2-NaCl-5.0-2-6	1.2000
7	PbCl2-NaCl-5.0-1-7	1.2012	PbCl2-NaCl-5.0-2-7	1.2012
8	PbCl2-NaCl-5.0-1-8	1.2006	PbCl2-NaCl-5.0-2-8	1.2005
9	PbCl2-NaCl-5.0-1-9	1.2003	PbCl2-NaCl-5.0-2-9	1.2000
10	PbCl2-NaCl-5.0-1-10	1.2008	PbCl2-NaCl-5.0-2-10	1.2003
11	PbCl2-NaCl-5.0-1-11	1.2061	PbCl2-NaCl-5.0-2-11	1.2053
12	PbCl2-NaCl-5.0-1-12	1.2028	PbCl2-NaCl-5.0-2-12	1.2016
13	PbCl2-NaCl-5.0-1-13	1.2042	PbCl2-NaCl-5.0-2-13	1.2031
14	PbCl2-NaCl-5.0-1-14	1.2029	PbCl2-NaCl-5.0-2-14	1.2046
15	PbCl2-NaCl-5.0-1-15	1.2032	PbCl2-NaCl-5.0-2-15	1.2034

Information Only

Table 1 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL 6.0 m NaCl		
1	PbCl2-NaCl-6.0-1-1	1.2007	PbCl2-NaCl-6.0-2-1	1.2012
2	PbCl2-NaCl-6.0-1-2	1.1999	PbCl2-NaCl-6.0-2-2	1.1997
3	PbCl2-NaCl-6.0-1-3	1.2025	PbCl2-NaCl-6.0-2-3	1.2018
4	PbCl2-NaCl-6.0-1-4	1.2004	PbCl2-NaCl-6.0-2-4	1.2000
5	PbCl2-NaCl-6.0-1-5	1.2005	PbCl2-NaCl-6.0-2-5	1.1997
6	PbCl2-NaCl-6.0-1-6	1.2009	PbCl2-NaCl-6.0-2-6	1.2005
7	PbCl2-NaCl-6.0-1-7	1.2018	PbCl2-NaCl-6.0-2-7	1.2014
8	PbCl2-NaCl-6.0-1-8	1.2000	PbCl2-NaCl-6.0-2-8	1.2015
9	PbCl2-NaCl-6.0-1-9	1.2007	PbCl2-NaCl-6.0-2-9	1.2014
10	PbCl2-NaCl-6.0-1-10	1.2006	PbCl2-NaCl-6.0-2-10	1.2011
11	PbCl2-NaCl-6.0-1-11	1.2063	PbCl2-NaCl-6.0-2-11	1.2055
12	PbCl2-NaCl-6.0-1-12	1.2009	PbCl2-NaCl-6.0-2-12	1.2045
13	PbCl2-NaCl-6.0-1-13	1.2040	PbCl2-NaCl-6.0-2-13	1.2007
14	PbCl2-NaCl-6.0-1-14	1.2039	PbCl2-NaCl-6.0-2-14	1.2016
15	PbCl2-NaCl-6.0-1-15	1.2020	PbCl2-NaCl-6.0-2-15	1.2023

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Table 2. Experimental set-up for the solubility of anglesite ($PbSO_4(s)$) in NaCl matrix (0-6.0 m) (WIPP-Pb-1, pages 20-23).

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL DDI water		
1	PbSO4-NaCl-0-1-1	1.0000	PbSO4-NaCl-0-2-1	1.0008
1	PbSO4-NaCl-0-1-2	0.9999	PbSO4-NaCl-0-2-2	1.0008
3	PbSO4-NaCl-0-1-3	0.9998	PbSO4-NaCl-0-2-3	1.0015
4	PbSO4-NaCl-0-1-4	1.0008	PbSO4-NaCl-0-2-4	0.9999
5	PbSO4-NaCl-0-1-5	1.0019	PbSO4-NaCl-0-2-5	1.0020
6	PbSO4-NaCl-0-1-6	1.0019	PbSO4-NaCl-0-2-6	1.0010
7	PbSO4-NaCl-0-1-7	1.0007	PbSO4-NaCl-0-2-7	1.0002
8	PbSO4-NaCl-0-1-8	1.0016	PbSO4-NaCl-0-2-8	1.0001
9	PbSO4-NaCl-0-1-9	1.0001	PbSO4-NaCl-0-2-9	1.0005
10	PbSO4-NaCl-0-1-10	1.0013	PbSO4-NaCl-0-2-10	1.0015
11	PbSO4-NaCl-0-1-11	1.0018	PbSO4-NaCl-0-2-11	1.0009
12	PbSO4-NaCl-0-1-12	1.0019	PbSO4-NaCl-0-2-12	1.0011
13	PbSO4-NaCl-0-1-13	1.0013	PbSO4-NaCl-0-2-13	1.0009
14	PbSO4-NaCl-0-1-14	1.0016	PbSO4-NaCl-0-2-14	1.0007
15	PbSO4-NaCl-0-1-15	1.0004	PbSO4-NaCl-0-2-15	1.0017
		20 mL 0.01 m NaCl		
1	PbSO4-NaCl-0.01-1-1	1.0005	PbSO4-NaCl-0.01-2-1	1.0012
1	PbSO4-NaCl-0.01-1-2	1.0005	PbSO4-NaCl-0.01-2-2	1.0005
3	PbSO4-NaCl-0.01-1-3	1.0014	PbSO4-NaCl-0.01-2-3	1.0000
4	PbSO4-NaCl-0.01-1-4	1.0016	PbSO4-NaCl-0.01-2-4	1.0023
5	PbSO4-NaCl-0.01-1-5	1.0001	PbSO4-NaCl-0.01-2-5	1.0006
6	PbSO4-NaCl-0.01-1-6	1.0006	PbSO4-NaCl-0.01-2-6	1.0001
7	PbSO4-NaCl-0.01-1-7	1.0008	PbSO4-NaCl-0.01-2-7	1.0003
8	PbSO4-NaCl-0.01-1-8	1.0009	PbSO4-NaCl-0.01-2-8	1.0000
9	PbSO4-NaCl-0.01-1-9	1.0011	PbSO4-NaCl-0.01-2-9	1.0006
10	PbSO4-NaCl-0.01-1-10	1.0018	PbSO4-NaCl-0.01-2-10	1.0010
11	PbSO4-NaCl-0.01-1-11	1.0001	PbSO4-NaCl-0.01-2-11	1.0013
12	PbSO4-NaCl-0.01-1-12	1.0001	PbSO4-NaCl-0.01-2-12	1.0002
13	PbSO4-NaCl-0.01-1-13	1.0019	PbSO4-NaCl-0.01-2-13	1.0015
14	PbSO4-NaCl-0.01-1-14	1.0017	PbSO4-NaCl-0.01-2-14	1.0013
15	PbSO4-NaCl-0.01-1-15	1.0002	PbSO4-NaCl-0.01-2-15	1.0013

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Table 2 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL 0.1 m NaCl		
1	PbSO4-NaCl-0.1-1-1	1.0001	PbSO4-NaCl-0.1-2-1	1.0010
1	PbSO4-NaCl-0.1-1-2	1.0009	PbSO4-NaCl-0.1-2-2	0.9997
3	PbSO4-NaCl-0.1-1-3	1.0004	PbSO4-NaCl-0.1-2-3	0.9995
4	PbSO4-NaCl-0.1-1-4	0.9998	PbSO4-NaCl-0.1-2-4	1.0015
5	PbSO4-NaCl-0.1-1-5	1.0022	PbSO4-NaCl-0.1-2-5	1.0021
6	PbSO4-NaCl-0.1-1-6	1.0019	PbSO4-NaCl-0.1-2-6	1.0001
7	PbSO4-NaCl-0.1-1-7	1.0005	PbSO4-NaCl-0.1-2-7	1.0003
8	PbSO4-NaCl-0.1-1-8	1.0011	PbSO4-NaCl-0.1-2-8	1.0015
9	PbSO4-NaCl-0.1-1-9	1.0000	PbSO4-NaCl-0.1-2-9	1.0002
10	PbSO4-NaCl-0.1-1-10	1.0008	PbSO4-NaCl-0.1-2-10	1.0019
11	PbSO4-NaCl-0.1-1-11	1.0004	PbSO4-NaCl-0.1-2-11	1.0002
12	PbSO4-NaCl-0.1-1-12	1.0009	PbSO4-NaCl-0.1-2-12	1.0012
13	PbSO4-NaCl-0.1-1-13	1.0012	PbSO4-NaCl-0.1-2-13	1.0007
14	PbSO4-NaCl-0.1-1-14	1.0012	PbSO4-NaCl-0.1-2-14	1.0006
15	PbSO4-NaCl-0.1-1-15	1.0010	PbSO4-NaCl-0.1-2-15	1.0003
		20 mL 1.0 m NaCl		
1	PbSO4-NaCl-1.0-1-1	1.0018	PbSO4-NaCl-1.0-2-1	1.0016
1	PbSO4-NaCl-1.0-1-2	0.9998	PbSO4-NaCl-1.0-2-2	1.0009
3	PbSO4-NaCl-1.0-1-3	1.0010	PbSO4-NaCl-1.0-2-3	1.0019
4	PbSO4-NaCl-1.0-1-4	1.0005	PbSO4-NaCl-1.0-2-4	1.0020
5	PbSO4-NaCl-1.0-1-5	1.0000	PbSO4-NaCl-1.0-2-5	1.0014
6	PbSO4-NaCl-1.0-1-6	1.0000	PbSO4-NaCl-1.0-2-6	1.0009
7	PbSO4-NaCl-1.0-1-7	1.0015	PbSO4-NaCl-1.0-2-7	1.0013
8	PbSO4-NaCl-1.0-1-8	1.0004	PbSO4-NaCl-1.0-2-8	1.0018
9	PbSO4-NaCl-1.0-1-9	1.0000	PbSO4-NaCl-1.0-2-9	1.0006
10	PbSO4-NaCl-1.0-1-10	1.0010	PbSO4-NaCl-1.0-2-10	1.0005
11	PbSO4-NaCl-1.0-1-11	1.0006	PbSO4-NaCl-1.0-2-11	1.0013
12	PbSO4-NaCl-1.0-1-12	1.0004	PbSO4-NaCl-1.0-2-12	1.0018
13	PbSO4-NaCl-1.0-1-13	1.0011	PbSO4-NaCl-1.0-2-13	1.0015
14	PbSO4-NaCl-1.0-1-14	1.0011	PbSO4-NaCl-1.0-2-14	1.0013
15	PbSO4-NaCl-1.0-1-15	1.0011	PbSO4-NaCl-1.0-2-15	1.0011

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Table 2 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
	20 mL 2.0 m NaCl			
1	PbSO4-NaCl-2.0-1-1	1.0007	PbSO4-NaCl-2.0-2-1	1.0003
1	PbSO4-NaCl-2.0-1-2	1.0020	PbSO4-NaCl-2.0-2-2	1.0000
3	PbSO4-NaCl-2.0-1-3	1.0005	PbSO4-NaCl-2.0-2-3	1.0016
4	PbSO4-NaCl-2.0-1-4	1.0022	PbSO4-NaCl-2.0-2-4	1.0013
5	PbSO4-NaCl-2.0-1-5	0.9998	PbSO4-NaCl-2.0-2-5	1.0014
6	PbSO4-NaCl-2.0-1-6	1.0008	PbSO4-NaCl-2.0-2-6	1.0003
7	PbSO4-NaCl-2.0-1-7	1.0005	PbSO4-NaCl-2.0-2-7	1.0003
8	PbSO4-NaCl-2.0-1-8	1.0003	PbSO4-NaCl-2.0-2-8	1.0002
9	PbSO4-NaCl-2.0-1-9	1.0002	PbSO4-NaCl-2.0-2-9	1.0003
10	PbSO4-NaCl-2.0-1-10	1.0008	PbSO4-NaCl-2.0-2-10	1.0003
11	PbSO4-NaCl-2.0-1-11	1.0005	PbSO4-NaCl-2.0-2-11	1.0010
12	PbSO4-NaCl-2.0-1-12	1.0017	PbSO4-NaCl-2.0-2-12	1.0014
13	PbSO4-NaCl-2.0-1-13	1.0026	PbSO4-NaCl-2.0-2-13	1.0007
14	PbSO4-NaCl-2.0-1-14	1.0009	PbSO4-NaCl-2.0-2-14	1.0001
15	PbSO4-NaCl-2.0-1-15	1.0009	PbSO4-NaCl-2.0-2-15	1.0019
	20 mL 3.0 m NaCl			
1	PbSO4-NaCl-3.0-1-1	1.0001	PbSO4-NaCl-3.0-2-1	1.0014
1	PbSO4-NaCl-3.0-1-2	1.0019	PbSO4-NaCl-3.0-2-2	1.0009
3	PbSO4-NaCl-3.0-1-3	1.0020	PbSO4-NaCl-3.0-2-3	1.0017
4	PbSO4-NaCl-3.0-1-4	1.0009	PbSO4-NaCl-3.0-2-4	1.0005
5	PbSO4-NaCl-3.0-1-5	1.0025	PbSO4-NaCl-3.0-2-5	1.0013
6	PbSO4-NaCl-3.0-1-6	1.0006	PbSO4-NaCl-3.0-2-6	1.0014
7	PbSO4-NaCl-3.0-1-7	1.0014	PbSO4-NaCl-3.0-2-7	1.0010
8	PbSO4-NaCl-3.0-1-8	1.0019	PbSO4-NaCl-3.0-2-8	1.0012
9	PbSO4-NaCl-3.0-1-9	1.0005	PbSO4-NaCl-3.0-2-9	1.0016
10	PbSO4-NaCl-3.0-1-10	1.0000	PbSO4-NaCl-3.0-2-10	1.0002
11	PbSO4-NaCl-3.0-1-11	1.0008	PbSO4-NaCl-3.0-2-11	1.0015
12	PbSO4-NaCl-3.0-1-12	1.0005	PbSO4-NaCl-3.0-2-12	1.0010
13	PbSO4-NaCl-3.0-1-13	1.0014	PbSO4-NaCl-3.0-2-13	1.0000
14	PbSO4-NaCl-3.0-1-14	1.0003	PbSO4-NaCl-3.0-2-14	1.0011
15	PbSO4-NaCl-3.0-1-15	1.0012	PbSO4-NaCl-3.0-2-15	1.0008

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Table 2 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL 4.0 m NaCl		
1	PbSO4-NaCl-4.0-1-1	1.0007	PbSO4-NaCl-4.0-2-1	1.0011
1	PbSO4-NaCl-4.0-1-2	1.0000	PbSO4-NaCl-4.0-2-2	0.9998
3	PbSO4-NaCl-4.0-1-3	1.0019	PbSO4-NaCl-4.0-2-3	1.0008
4	PbSO4-NaCl-4.0-1-4	1.0018	PbSO4-NaCl-4.0-2-4	1.0020
5	PbSO4-NaCl-4.0-1-5	1.0010	PbSO4-NaCl-4.0-2-5	1.0005
6	PbSO4-NaCl-4.0-1-6	1.0018	PbSO4-NaCl-4.0-2-6	1.0006
7	PbSO4-NaCl-4.0-1-7	1.0003	PbSO4-NaCl-4.0-2-7	1.0012
8	PbSO4-NaCl-4.0-1-8	1.0009	PbSO4-NaCl-4.0-2-8	1.0003
9	PbSO4-NaCl-4.0-1-9	1.0007	PbSO4-NaCl-4.0-2-9	1.0000
10	PbSO4-NaCl-4.0-1-10	1.0009	PbSO4-NaCl-4.0-2-10	1.0019
11	PbSO4-NaCl-4.0-1-11	1.0005	PbSO4-NaCl-4.0-2-11	1.0002
12	PbSO4-NaCl-4.0-1-12	1.0015	PbSO4-NaCl-4.0-2-12	1.0008
13	PbSO4-NaCl-4.0-1-13	1.0004	PbSO4-NaCl-4.0-2-13	1.0008
14	PbSO4-NaCl-4.0-1-14	1.0013	PbSO4-NaCl-4.0-2-14	1.0010
15	PbSO4-NaCl-4.0-1-15	1.0003	PbSO4-NaCl-4.0-2-15	1.0005
		20 mL 5.0 m NaCl		
1	PbSO4-NaCl-5.0-1-1	1.0023	PbSO4-NaCl-5.0-2-1	1.0018
1	PbSO4-NaCl-5.0-1-2	1.0003	PbSO4-NaCl-5.0-2-2	1.0017
3	PbSO4-NaCl-5.0-1-3	1.0000	PbSO4-NaCl-5.0-2-3	1.0008
4	PbSO4-NaCl-5.0-1-4	1.0015	PbSO4-NaCl-5.0-2-4	1.0012
5	PbSO4-NaCl-5.0-1-5	1.0020	PbSO4-NaCl-5.0-2-5	1.0003
6	PbSO4-NaCl-5.0-1-6	1.0000	PbSO4-NaCl-5.0-2-6	1.0002
7	PbSO4-NaCl-5.0-1-7	1.0016	PbSO4-NaCl-5.0-2-7	1.0000
8	PbSO4-NaCl-5.0-1-8	1.0002	PbSO4-NaCl-5.0-2-8	1.0015
9	PbSO4-NaCl-5.0-1-9	1.0019	PbSO4-NaCl-5.0-2-9	1.0000
10	PbSO4-NaCl-5.0-1-10	1.0019	PbSO4-NaCl-5.0-2-10	1.0001
11	PbSO4-NaCl-5.0-1-11	1.0018	PbSO4-NaCl-5.0-2-11	1.0013
12	PbSO4-NaCl-5.0-1-12	1.0005	PbSO4-NaCl-5.0-2-12	1.0007
13	PbSO4-NaCl-5.0-1-13	1.0006	PbSO4-NaCl-5.0-2-13	1.0004
14	PbSO4-NaCl-5.0-1-14	1.0009	PbSO4-NaCl-5.0-2-14	1.0014
15	PbSO4-NaCl-5.0-1-15	1.0013	PbSO4-NaCl-5.0-2-15	1.0000

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Table 2 (continued)

Vial #	Sample ID	Measured $PbCl_2$ (g)	Sample ID	Measured $PbCl_2$ (g)
		20 mL 6.0 m NaCl		
1	PbSO4-NaCl-6.0-1-1	1.0014	PbSO4-NaCl-6.0-2-1	1.0019
1	PbSO4-NaCl-6.0-1-2	1.0000	PbSO4-NaCl-6.0-2-2	1.0009
3	PbSO4-NaCl-6.0-1-3	1.0008	PbSO4-NaCl-6.0-2-3	0.9998
4	PbSO4-NaCl-6.0-1-4	1.0008	PbSO4-NaCl-6.0-2-4	1.0015
5	PbSO4-NaCl-6.0-1-5	1.0006	PbSO4-NaCl-6.0-2-5	1.0015
6	PbSO4-NaCl-6.0-1-6	1.0014	PbSO4-NaCl-6.0-2-6	1.0005
7	PbSO4-NaCl-6.0-1-7	1.0004	PbSO4-NaCl-6.0-2-7	1.0020
8	PbSO4-NaCl-6.0-1-8	1.0012	PbSO4-NaCl-6.0-2-8	1.0001
9	PbSO4-NaCl-6.0-1-9	1.0001	PbSO4-NaCl-6.0-2-9	1.0003
10	PbSO4-NaCl-6.0-1-10	1.0015	PbSO4-NaCl-6.0-2-10	1.0000
11	PbSO4-NaCl-6.0-1-11	1.0016	PbSO4-NaCl-6.0-2-11	1.0002
12	PbSO4-NaCl-6.0-1-12	1.0014	PbSO4-NaCl-6.0-2-12	1.0004
13	PbSO4-NaCl-6.0-1-13	1.0014	PbSO4-NaCl-6.0-2-13	1.0009
14	PbSO4-NaCl-6.0-1-14	1.0007	PbSO4-NaCl-6.0-2-14	1.0001
15	PbSO4-NaCl-6.0-1-15	1.0002	PbSO4-NaCl-6.0-2-15	1.0003

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3.3 Task 3: Scoping Experiments

Task 3 in TP 20-01 Rev. 0 is a set of six subtasks designed to investigate (i) the solubility and stability of $PbCl_2(s)$ (Subtasks 3.1, 3.2, 3.3, 3.4, and 3.5), and (ii) the dissolution of PbO(s) by complexation of $EDTA^{4-}$ in the presence of $Mg(OH)_2(s)$ (Subtask 3.6). Chloride was excluded in designing Subtask 3.6 to avoid the formation of secondary Pb-Cl-OH solid(s) at the expense of PbO(s) as reported in Kirkes and Xiong (2019).

For this end-of-year report, subtask 3.1 was prepared to measure the solubility of $PbCl_2(s)$ (cotunnite) in DDI water. The goal of this subtask 3.1 is to prove or disprove the following hypotheses: (i) SNL WIPP Geochemistry can reproduce the solubility of $PbCl_2(s)$ (cotunnite) in pure water reported in the literature, i.e., 0.039 M (Burrage, 1926; Deacon, 1927; Carmody, 1929; Wilkinson et al., 1937; Tan et al., 1987), within the uncertainty associated with overall laboratory operation, and (ii) the solubility of $PbCl_2(s)$ is independent of the solid/solution ratio. The preparation is described in the **Table 3**. Three $PbCl_2(s)$ of different sources were used at three different solid/solution ratios.

Table 3. Experimental set-up for the solubility $PbCl_2(s)$ (cotunnite) in DDI water (WIPP-Pb-1, pages 13, 14, and 28).

Reactor ID	Measured $PbCl_2(s)$ (g)	Source of $PbCl_2(s)$	Volume of DDI water (mL)
PbCl2DI-1-1	1.0008	Acros Organics, 99%, LOT AO387918	50
PbCl2DI-1-2	4.9999	Acros Organics, 99%, LOT AO387918	50
PbCl2DI-1-3	15.0012	Acros Organics, 99%, LOT AO387918	50
PbCl2DI-2-1	1.0007	Alfa Aesar, 99.999%, LOT Z31E012	50
PbCl2DI-2-2	5.0006	Alfa Aesar, 99.999%, LOT Z31E012	50
PbCl2DI-2-3	15.0001	Alfa Aesar, 99.999%, LOT Z31E012	50
PbCl2DI-3-1	1.0003	Alfa Aesar, 99.999%, LOT 23244	50
PbCl2DI-3-2	5.0006	Alfa Aesar, 99.999%, LOT 23244	50
PbCl2DI-3-3	14.9999	Alfa Aesar, 99.999%, LOT 23244	50

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4.0 ACKNOWLEDGEMENTS

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5.0 REFERENCES

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