X-Ray Diffraction and Scanning Electron Microscope Analysis of COPR Samples

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April 20, 2007

CONTENTS

TABLES 2
APPENDICES 2

1.0 INTRODUCTION 3

2.0 SAMPLE ANALYSIS PROCEDURE 4
2.1 X-Ray Diffraction 4
2.2 Scanning Electron Microscopy/Electron Microprobe 4

3.0 RESULTS 5
3.1 X-Ray Diffraction 5
3.2 SEM Images and EDS Analysis 9

4.0 DISCUSSION 11
4.1 Mineralogical Characteristics of COPR 11

TABLES

Table 1. Samples Received for Analysis 3
Table 2a. XRD Results in Weight Percent of Crystalline Matter (Priority 1 Samples) 6
Table 2b. XRD Results in Weight Percent of Crystalline Matter (Priority 2 Samples) 8
Table 3. Chemical and Crystallographic Data for Phases Present in Test Samples 9
Table 4. Summary of SEM/EDS Results for Images in Appendix B 10

APPENDICES

APPENDIX A: Mineralogical Samples
APPENDIX B: XRD Patterns
APPENDIX C: Quality Assurance
APPENDIX D: SEM Files

1.0 INTRODUCTION

Battelle received twenty-eight samples in December, 2006, from CH2M-Hill to be analyzed by x-ray diffraction (XRD) and scanning electron microscopy (SEM). These samples consisted of chrome ore processing residue (COPR) excavated from the Dundalk Marine Terminal (DMT) site in Baltimore, Maryland.

CH2M-Hill has delineated these samples in three categories based on the priority for their mineralogical investigations: (1) Priority 1 Samples, (2) Priority 2 samples, and (3) Priority 3 samples. Battelle received 14 priority 1 samples, 8 priority 2 samples and 6 priority 3 samples. Table 1 lists all the samples according to their priority. Appendix A lists the sample identification and description sheet that Battelle received from CH2M-Hill

The purpose of the mineralogical and chemical analyses was to determine the abundant minerals present in these samples and identify the minerals, which lead to the delineation of the subsurface into Grey Black (GB) and Hard Brown (HB) COPR samples.

Table 1. Samples Received for Analysis

Sample ID	Depth (ft)	Brief Description
Priority 1 Samples		
INC-12-SOI-1516	15.4-15.5	HB
INC-12-SOI-1616	16.0-16.1	GB
INC-12-SOI-1111	10.9-11.1	HB
INC-12-SOI-1414	14.3-14.4	HB
INC-12-SOI-1515	15.0-15.1	HB
INC-29S-SOI-14-1	14.2-14.4	HB
INC-29S-SOI-14-2	14.6-14.7	Transitional sample between GB and HB
INC-29S-SOI-14-3	14.8-14.9	GB
INC-18-SOI-0809	8.6-8.7	HB
INC-18-SOI-0910 GB	9.6-9.7	GB portion of a lithified sample that was primarily HB with lenses of GB
INC-18-SOI-0910 HB	9.6-9.7	HB portion of a lithified sample that was primarily HB with lenses of GB
INC-18-SOI-1010	10.0-10.1	GB
INC-18-SOI-1212	11.9-12.0	GB
Priority 2 Samples		
INC-10-SOI-1113	11-13	GB
INC-10-SOI-1314	13.0-14.0	HB
INC-3-SOI-1113	11-13	GB
INC-3-SOI-1819	18-19	HB
INC-9-SOI-1010	10-10.1	GB
INC-18-SOI-1111	10.8-10.9	GB

Table 1. Samples Received for Analysis (continued)

Sample ID	Depth (ft)	Brief Description
INC-19-SOI-1921	19-21	GB
INC-19-SOI-2223	22-23	GB
INC-20-SOI-0305	3.5-5.0	Particulate HB
DMT-33S-SOI-1518	15.5-18.0	GB
Priority 3 Samples		
INC-9-SOI-0707	7.0-7.5	HB
INC-11-3839	38-39	GB
INC-13-SOI-0305	3.5-5.0	HB
INC-18-SOI-0710	7-10	HB
INC-18-SOI-1314	13.6-13.7	HB
INC-18-SOI-1012	10-12	GB

2.0 SAMPLE ANALYSIS PROCEDURE

2.1 X-Ray Diffraction

Samples sent by CH2M-Hill were analyzed by x-ray diffraction (XRD) using a Rigaku wide angle powder diffractometer running Materials Data, Inc., (MDI) automation and software (JADE 7+). The x-ray radiation source used was a copper normal focus x-ray tube operated at 40 kV and 30 mA. The goniometer slit choices were the standard 0.5 degree DS and SS, 0.3 RS and a 0.6 slit in front of the scintillation counter. Further, a curved graphite monochromator was employed to remove the copper K-beta lines and much of the x-ray background from the patterns. An external standard (NBS 640b Silicon powder) was run on a regular basis to ensure that the mechanical alignment of the instrument was within quality assurance (QA) specifications.

A representative portion of each supplied sample was air-dried for at least 24 hours in an environmentally-controlled room. One to two grams of the dried material were then ground with an agate mortar and pestle to pass through a 400 mesh sieve (<38 µm). A portion of this material was carefully packed into a standard Rigaku 0.5 mm recessed glass holder and scanned in the XRD from 5°–70° 2θ, using a step size was 0.024° 2θ and a speed of 0.24° 2θ/minute.

2.2 Scanning Electron Microscopy

Samples received by CH2M-Hill were analyzed in the JEOL 840A scanning electron microscope (SEM) with an attached Oxford INCA 300 Energy Dispersive Spectrometer. Unpolished mounts that were coated with gold were examined three dimensionally. Several small fragments (representative of the sample) were carefully adhered to double-sided carbon tape that was attached to a one inch carbon planchet. These three-dimensional mounts were gold coated to reduce charging during examination. Images were obtained using secondary and backscattered electrons at magnifications capable of ranging from 10 to 50,000 times. Accelerating voltage and beam current are varied depending on the experiment but typically nominal conditions are 20 kV and 1 nA. These samples could be rotated and tilted to improve viewing approach. Semi-quantitative chemistry information (elemental identification and approximate abundance) were collected on various spots or areas of interest using the Oxford Energy Dispersive Spectrometer (EDS). Calibration and QA protocols are described in Appendix C.

3.0 RESULTS

3.1 X-Ray Diffraction

The raw data (collected) patterns were processed by first modeling and then removing the background inherent in the x-ray trace as well as the $K\alpha_2$ line contributions. The derived pattern was then run through the extensive search/match programming module with accessibility to approximately 255,000 crystalline (inorganic and organic) indexed substances.[1] Several levels of search/match filtering for chemistry, preferred orientation, and crystallography were preformed to give initial identification of the phases present in these soils. Computer identification was then followed up with manual processing and identification to obtain the list of phases present in the DMT samples. Table 2a-2b lists the minerals or inorganic compounds that were identified in the priority 1 and 2 samples. Table 3 lists the chemical composition of these minerals. Raw data patterns with superimposed diffraction lines for identified peaks are presented in Appendix B.

[1] Powder Diffraction File-4+ 2006 RDB and the Inorganic Crystal Structure Database/NIST version 2006-2

Table 2a. XRD Results in Weight Percent of Crystalline Matter (Priority 1 Samples)

XRD Units weight percent

Sample ID	Depth (ft)	Brief Description	Brownmillerite	Periclase	Brucite	Portlandite	Calcite	Hydrotalcite	Hydrocalumite	Hydrogarnet (katoite)	Hydrogarnet (Ca3Fe2(OH)12)	Ettringite	Afwillite	Ca4Al2O6(CrO4) 9H2O	Ca4Al2CrO10 12H2O	3CaOAl2O3CaCrO4 14H2O	NaCa4Al2O6(CrO4)1.5 15H2O	Kuzelite	Ca4Al2O6(SO4) 14H2O	Ca4Al2(OH)12(CO3)(H2O)5	Ca8Al4O14CO2 24H2O	Feldspar	Quartz	Clinochlore	SUM
Samples received by Battelle on 11th November, 2006																									
INC-12-SOI-1516	15.4-15.5	HB	3.2	1.8	5.4	0.0	0.0	2.1	0.0	39.7	0.0	0.0	0.0	2.4	0.0	10.1	5.0	0.0	0.0	20.5	0.0	0.0	9.8	0.0	100.0
INC-12-SOI-1616	16.0-16.1	GB	37.5	5.0	2.8	0.0	2.9	2.2	0.0	22.6	0.0	1.3	0.0	0.0	0.0	14.9	5.4	0.0	0.0	2.6	0.0	0.0	2.8	0.0	100.0
INC-12-SOI-1111	10.9-11.1	HB	3.2	1.8	6.2	0.0	0.0	2.8	0.0	39.0	0.0	0.0	0.0	1.6	0.0	2.6	5.7	0.0	0.0	27.7	0.0	0.0	9.3	0.0	99.9
INC-12-SOI-1414	14.3-14.4	HB	2.2	1.3	4.5	0.0	0.7	2.0	0.0	42.5	0.0	0.0	0.0	0.0	0.0	8.4	1.9	0.0	0.0	24.0	0.0	0.0	12.4	0.0	99.9
INC-12-SOI-1515	15.0-15.1	HB	3.2	2.4	5.6	0.0	0.0	2.3	0.0	34.5	0.0	0.0	0.0	1.8	0.0	1.6	2.9	0.0	0.0	34.5	0.0	0.0	11.3	0.0	100.1
INC-29S-SOI-14-1	14.2-14.4	HB	1.7	0.3	4.4	0.0	0.0	10.0	0.0	12.4	12.6	0.0	0.0	16.1	0.0	1.9	0.0	0.0	0.0	38.2	0.0	0.0	2.4	0.0	100.0
INC-29S-SOI-14-2	14.6-14.7	Transitional sample between GB and HB	42.6	3.0	3.7	0.0	4.0	5.3	0.0	13.9	0.0	0.0	0.0	1.1	0.0	0.0	1.9	0.0	0.0	24.5	0.0	0.0	0.0	0.0	100.0
INC-29S-SOI-14-3	14.8-14.9	GB	58.5	4.0	4.7	1.5	1.4	3.5	0.0	13.3	0.0	0.0	0.0	1.1	0.0	1.4	5.3	0.0	0.0	5.4	0.0	0.0	0.0	0.0	100.1

Table 2a. XRD Results in Weight Percent of Crystalline Matter (Priority 1 Samples) (continued)

Sample ID	Depth (ft)	Brief Description	Brownmillerite	Periclase	Brucite	Portlandite	Calcite	Hydrotalcite	Hydrocalumite	Hydrogarnet (katoite)	Hydrogarnet (Ca3Fe2(OH)12)	Ettringite	Afwillite	Ca4Al2O6(CrO4) 9H2O	Ca4Al2CrO10 12H2O	3CaOAl2O3CaCrO4 14H2O	NaCa4Al2O6(CrO4)1.5 15H2O	Kuzelite	Ca4Al2O6(SO4) 14H2O	Ca4Al2(OH)12(CO3)(H2O)5	Ca8Al4O14CO2 24H2O	Feldspar	Quartz	Clinochlore	SUM
Samples received by Battelle on 15th December, 2006																									
INC-18-SOI-0809	8.6-8.7	HB	1.6	0.8	3.3	0.0	0.0	6.4	9.1	33.4	0.0	0.0	0.0	26.2	0.0	0.0	0.0	0.0	0.0	19.2	0.0	0.0	0.0	0.0	100.0
INC-18-SOI-0910 GB	9.6-9.7	GB portion of a lithified sample that was primarily HB with lenses of GB	6.1	2.4	2.6	0.0	0.0	0.8	3.1	35.1	0.0	0.0	0.0	0.0	0.0	1.1	2.4	0.0	0.0	6.5	0.0	0.0	38.5	1.3	99.9
INC-18-SOI-0910 HB	9.6-9.7	HB portion of a lithified sample that was primarily HB with lenses of GB	1.5	1.1	5.0	0.0	0.0	3.2	0.0	25.0	0.0	0.0	0.0	11.0	0.0	6.3	3.5	0.0	0.0	43.3	0.0	0.0	0.0	0.0	99.9
INC-18-SOI-1010	10.0-10.1	GB	28.6	4.2	1.6	0.0	2.5	0.8	0.0	12.6	0.0	0.0	0.0	0.0	0.0	5.7	2.5	0.0	0.0	2.5	0.0	0.0	39.0	0.0	100.0
INC-18-SOI-1212	11.9-12.0	GB	40.1	5.9	2.6	2.4	2.7	2.2	0.0	20.0	0.0	0.0	0.0	0.0	0.0	13.8	4.0	0.0	0.0	4.4	0.0	0.0	1.9	0.0	100.0
INC-10-SOI-1113	11-13	GB	55.3	4.5	5.3	0.0	4.0	6.5	0.0	8.8	0.0	1.5	0.0	1.3	0.0	3.3	0.0	0.0	0.0	7.1	0.0	0.0	2.4	0.0	100.0
INC-10-SOI-1314	13.0-14.0	HB	0.7	0.0	5.0	0.0	0.0	1.5	6.7	13.2	14.1	0.0	0.0	46.5	0.0	0.0	0.0	0.0	0.0	12.4	0.0	0.0	0.0	0.0	100.1

Table 2b. XRD Results in Weight Percent of Crystalline Matter (Priority 2 Samples)

Sample ID	Depth (ft)	Brief Description	Brownmillerite	Periclase	Brucite	Portlandite	Calcite	Hydrotalcite (Sjoegrenite)	Hydrocalumite	Hydrogarnet (katoite)	Hydrogarnet (Ca3Fe2(OH)12)	Ettringite	Afwillite	Ca4Al2O6(CrO4) 9H2O	Ca4Al2CrO10 12H2O	3CaOAl2O3CaCrO4 14H2O	NaCa4Al2O6(SO4)1.5 15H2O	Kuzelite	Ca4Al2O6(SO4) 14H2O	Ca4Al2(OH)12(CO3)(H2O)5	Ca8Al4O14CO2 24H2O	Feldspar	Quartz	Clinochlore	SUM
INC-3-SOI-1113	11-13	GB	56.9	4.5	3.7	0.9	3.3	2.3	0.0	9.1	0.0	0.0	1.1	0.0	0.0	13.1	3.0	0.0	0.0	2.1	0.0	0.0	0.0	0.0	100.0
INC-3-SOI-1819	18-19	HB	4.0	3.8	2.8	0.0	0.0	27.6	24.1	27.3	0.0	0.0	0.0	0.0	0.0	1.4	6.0	0.0	0.0	3.0	0.0	0.0	0.0	0.0	100.0
INC-9-SOI-1010	10-10.1	GB	62.1	4.5	9.9	0.5	1.5	3.1	0.0	9.3	0.0	0.9	0.0	0.0	0.0	4.9	1.4	0.0	0.0	2.0	0.0	0.0	0.0	0.0	100.1
INC-18-SOI-1111	10.8-10.9	GB	29.0	4.4	1.4	0.0	2.3	0.8	0.0	15.2	0.0	0.0	0.0	0.0	0.0	10.4	3.6	0.0	0.0	10.2	0.0	1.6	19.9	1.3	100.1
INC-19-SOI-1921	19-21	GB	47.1	2.9	4.8	4.0	3.0	3.8	0.0	16.5	0.0	0.0	0.0	0.0	0.0	11.0	4.5	0.0	0.0	2.4	0.0	0.0	0.0	0.0	100.0
INC-19-SOI-2223	22-23	GB	1.3	0.0	4.5	0.0	0.0	0.9	3.9	33.3	0.0	24.2	0.0	0.0	0.0	17.0	0.0	13.0	0.0	1.9	0.0	0.0	0.0	0.0	100.0
INC-20-SOI-0305	3.5-5.0	Particulate HB	35.7	2.8	3.4	2.2	10.4	3.6	0.0	13.9	0.0	0.0	0.0	0.0	0.0	1.7	5.0	0.0	0.0	14.8	0.0	0.0	6.4	0.0	99.9
DMT-33S-SOI-1518	15.5-18.0	GB	49.4	3.7	7.8	7.2	4.5	3.4	0.0	16.4	0.0	0.0	1.3	0.0	0.0	0.6	4.0	0.0	0.0	1.7	0.0	0.0	0.0	0.0	100.0

Table 3. Chemical and Crystallographic Data for Phases Present in Test Samples

Mineral Name	Chemical Composition
Brownmillerite	$Ca_2(Al,Fe^{+3}\ Cr^{+3})_2O_5$
Periclase	MgO
Brucite	$Mg(OH)_2$
Portlandite	$Ca(OH)_2$
Hydrogarnet (katoite series)	$Ca_3Al_2(SiO_4)_{3-x}(OH)_{4x}$ (x=1.5-3)
Hydrotalcite	$Mg_6Al_2(CO_3)(OH)_{16} \cdot 4H_2O$
Hydrocalumite	$Ca_2Al(OH)_{6.5}Cl_{0.5} \cdot 3H_2O$
Gypsum	$CaSO_4{\cdot}2H_2O$
Ettringite	$Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$
Calcium iron hydroxide	$Ca_3Fe_2\ (OH)_{12}$
Calcium aluminum chromate hydrate	$Ca_4Al_2O_6(CrO_4) \cdot (9, 12, 14)H_2O$
Calcium aluminum sulfate hydrate	$Ca_4Al_2O_6(SO_4) \cdot 14H_2O$
Sodium calcium aluminum sulfate hydrate	$NaCa_4Al_2O_6\ (SO_4)_{1.5} \cdot 15H_2O$
Kuzelite	$Ca_4Al_2(SO_4)(OH)_{12} \cdot 6H_2O$
Calcium aluminum sulfate hydrate	$Ca_4Al_2O_6(SO_4) \cdot 14H_2O$
Calcium aluminum oxide carbonate hydrate	$Ca_4Al_2(OH)_{12}(CO_3)(H_2O)_5$
Calcium aluminum oxide carbonate hydrate	$Ca_8Al_4O_{14}CO_2 \cdot 24H_2O$
Lepidocrocite	γ–FeOOH
Calcite	$CaCO_3$
Quartz	SiO_2

3.2 SEM Images and EDS Analysis

SEM images of representative particles are presented in Appendix B, where the locations of spectral analysis are identified by numbers on each image. A cross-hair indicates where the x-ray beam was focused on a small-diameter point. The (pink) boxes illustrate where the x-ray beam was rastered over the defined area to produce an average composition.

The chemistry of selected particles was determined by EDS and summarized in Table 4. Results are considered semiquantitative because sample surfaces are very irregular and the EDS instrumentation was not calibrated to mineral standards. Normally, quantitative analyses are performed on epoxy-mounted, polished samples using an electron microprobe with appropriate calibration standards. Nevertheless, an attempt was made to identify certain minerals of interest in Table 4, based on atom (or mole) percentages by EDS and, where possible, crystal morphology. However, highly accurate analyses are not possible by this method and some mineral identifications are merely estimates.

Table 4. Summary of SEM/EDS Results for Images in Appendix B

Magnification	Spectrum Number	Description
INC-18-SOI-0809		
400X	1	AFm phases: (1) has low Al/Fe ratio; (2) has high (Al/Fe) ratio and may have CO3-2 substituting for CrO4-2
	2	
400 X(2)	1	All phases are calcium monochromate-type AFm
	2	
	3	
	4	
400X(3)	1	All phases are calcium monochromate-type AFm
	2	
	3	
2000X	1	AFm phases: (1) and (2) have high Al/Fe ratio; Al/Fe~1 in (3)
	2	
	3	
INC-18-SOI-0910-GB		
2000X	1	Both areas are intergranular cement which has been exposed after plucking another nodule away from the COPR. The composition suggests that several minerals are present in the cement.
	2	
INC-18-SOI-0910-HB		
300X	1	(1) Calcium monochromate with higher than usual iron content; (2) thin-walled tubule on surface of COPR nodule.
2500X	1	
INC-12-SOI-1616		
2500X	1	(1), (3) Acicular mineral that resembles a Cr-rich AFt phase (Cr-ettringite); (2), (4), AFm phase High Mg background in all solids
	2	
	3	
	4	
2500X(2)	1	(1), (2) Acicular mineral that resembles a Cr-rich AFt phase (Cr-ettringite); (3), (4), (5) AFm phases; measurable Na in (4) and (5) High Mg background in all solids
	2 3 4 5	
1000X	1	(1) Acicular mineral that resembles a Cr-rich AFt phase (Cr-ettringite); (2) AFm platelet
	2	

4.0 DISCUSSION

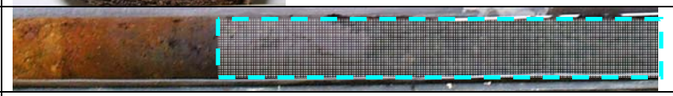
4.1 Mineralogical Characteristics of COPR

At the time COPR was deposited at DMT it is thought to have contained three primary minerals: brownmillerite, periclase, and portlandite (hydrated lime). After exposure to surface water and groundwater, brownmillerite slowly reacted to form several types of hydration products, including hydrogarnets, hydrotalcites, AFm phases (calcium aluminum hydrates of chromate, carbonate, and sulfate) an AFt phase (ettringite), calcium silicate hydrate (e.g. afwillite and amorphous C-S-H), and hydrous (and usually amorphous) ferric and chromic compounds (see compositions listed in Table 3). Hydration of periclase releases magnesium which produces brucite and hydrotalcite. Magnesium compounds are much more limited in number than those containing calcium.

Brownmillerite hydration is essential to the formation of aluminum-bearing mineral phases that cement COPR particles (nodules) together and provides the driving force for expansion. The AFm group accommodates several types of anions, including chromate. Therefore, mechanisms that reduce hexavalent chromium must first liberate Cr(VI) from calcium aluminum chromate hydrate (or simply referred to as *monochromate*). In pure water brownmillerite hydration begins with formation of metastable precursor phases composed of calcium, aluminum, and water (C-A-H in cement chemistry nomenclature). Alumina in these hydrated phases is more reactive than in the parent brownmillerite. This allows chromate to react with the hydrated alumina to form monochromates. It is thought that chromate accelerates the hydration of brownmillerite, similar to mechanism by which sulfate (as gypsum) accelerates the conversion of brownmillerite to ettringite. The reaction of carbonate with C-A-H compounds results in either of two calcium aluminum carbonate hydrates.

APPENDIX A

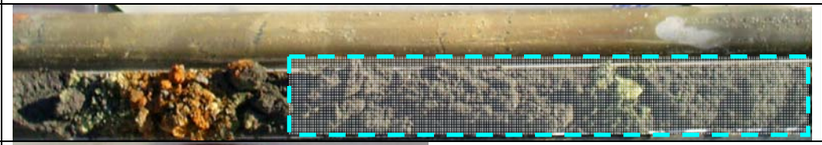
MINERALOGICAL SAMPLES

Field Sample ID	Start Depth (ft)	End Depth (ft)	Classification	Photo	Lithology Description	XRD Priority[1]	SEM	Comments
INC-3-SOI-1113	11.0	13.0	GB-SM		Silty sand, moist, particulate, 10YR 3/1 very dark gray, 10YR 2/1 black, trace bright green	2		Near the top of a column of GB material (9.5 – 18.0) overlying HB from 18.0 – 19.0
INC-3-SOI-1819	18.0	19.0	HB		Gravelly sand, wet, strongly indurated, 7.5YR 5/8 strong brown,	2		Gravel-size particles are cemented HB COPR, which can be broken apart with hammer to smaller fragments of HB.
INC-5-SOI-0305	3.5	5.0	GB		Sand, very fine to fine, 7.5YR 3/1 very dark gray and 7.5YR 7.5/1 black	2		HB layer immediately above
INC-9-SOI-0707	7.0	7.5	HB		Sand, fine to medium, dry, 7.5YR 3/3 dark brown	3		Do not conduct mineralogy yet as may be able to retrieve better sample from unextruded Pitch tube from 5.0 – 8.0
INC-9-SOI-1010	10.4	10.5	GB		Sand, moist, fine to medium, 7.5YR 3/1 very dark gray, trace bright green	2		Water table shown at 12.5 ft on log based on transition from moist to wet samples
INC-10-SOI-1113	11.0	13.0	GB		Silty sand, wet, particulate to weakly indurated, 10YR 2/1 black, 10YR 3/1 very dark gray, some bright green	1		No recover immediately above this sample from 10.0 – 11.0.

Field Sample ID	Start Depth (ft)	End Depth (ft)	Classification	Photo	Lithology Description	XRD Priority[1]	SEM	Comments
INC-10-SOI-1314	13.0	14.0	HB		Silty sand, trace gravel, wet, particulate to moderately indurated, 7.5YR 5/8 strong brown	1		Gravel is indurated HB COPR.
INC-11-3839	38.0	39.0	GB?	No photo available	Up to 38.5: Sand, medium to coarse, mottled colors, brown, black, red, some gray, trace green, no plasticity, moist	3		May be transition to non-COPR, do not test but hold for archive.
INC-12-SOI-1111	10.9	11.1	HB		No recovery with DPT – drill cuttings are DPC ++ and 7.5YR 5/6 strong to 5YR 5/8 yellowish red (moderately indurated per pitcher tube sample)	1		Driller hit refusal at 10.5. Must use 2-3/4 auger to open hole and loosen material. While auguring, driller states "like rock coring…eases up at around 12 ft". XRD sample is from Pitcher sample.
INC-12-SOI-1414	14.3	14.4	HB		Silty sand, moist, weakly indurated, 7.5YR 5/8 strong brown and trace bright green specks	1		Particulate HB above sample from 13.5 – 14.0 and GB above that from 13.0 – 13.5

Field Sample ID	Start Depth (ft)	End Depth (ft)	Classification	Photo	Lithology Description	XRD Priority[1]	SEM	Comments
INC-12-SOI-1515	15.0	15.1	HB		As above, except less strong brown, more mottled	1		
								Pitcher tube (PT3) advanced 13.0 – 15.5, photo shown is 14.0 – 15.5
INC-12-SOI-1516	15.4	15.5	HB		Silty sand, moist, hard, strongly indurated, 7.5YR 5/8 strong brown, trace bright green	1		The lithology description is from a disturbed sample. Transition to GB material is abrupt at about 15.8 ft bgs
INC-12-SOI-1616	16.0	16.1	GB		Soil boring log did not describe this transition	1		Sample from Pitcher tube, not DPT
INC-13-SOI-0305	3.5	5.0	HB?	No Picture Available?	Disturbed description: Sand and Gravel, hard, dry, no plasticity, mostly 5YR 5/8 and some 10YR 5/8 strong brown	3		Need photo to confirm what type of COPR this is prior to any mineralogical analyses
INC-18-SOI-	7.0	10.0	HB		See below for descriptions of discrete	3		Do NOT analyze – use

Field Sample ID	Start Depth (ft)	End Depth (ft)	Classification	Photo	Lithology Description	XRD Priority[1]	SEM	Comments
0710					intervals			discrete samples described below
INC-18-SOI-0809	8.6	8.7	HB		Sand, medium to coarse, some fines, hard, dry, no plasticity, 7.5YR 4/6 strong brown COPR	1		The lithology description is from a disturbed sample. Photo shown is an undisturbed sample from a pitcher tube, which is what was sent for analysis. The driller noted that DPT pushing increasingly difficult beginning at 5.0, with shortened hammer strokes.
INC-18-SOI-0910	9.6	9.7	HB		Same as above	1		Driller notes “like concrete”. Note the inclusion of some zones of gray-black material.
INC-18-SOI-1012	10.0	12.0	GB		See below for descriptions of discrete intervals	3		Do NOT analyze – use discrete samples described below
INC-18-SOI-1010	10.0	10.1	GB		Sand with some silt, hard, no plasticity, slightly damp, 10YR 6/1, gray, 10YR 3/1 very dark gray, trace “bright” green, fine- to medium-sized COPR particles.	1		This is likely a zone in transition. GB begins at 9.9 per the Pitcher barrel log.
INC-18-SOI-1111	10.8	10.9	GB		Same as above.	2		Transition to GB complete at this depth.

Field Sample ID	Start Depth (ft)	End Depth (ft)	Classification	Photo	Lithology Description	XRD Priority[1]	SEM	Comments
INC-18-SOI-1212	11.9	12.0	GB		Same as above.	1		Note the "classical" GB COPR appearance.
INC-18-SOI-1314	13.6	13.7	HB		No description in soil boring log.	3		This material sits immediately above a non-COPR silty sand layer.
INC-19-SOI-1921	19.0	21.0	GB	No photo available?	Sand, moist, fine to medium-sized, GLEY2 2.5/10B bluish black	2		Ask Battelle to take a photo prior to grinding, if not too late
INC-19-SOI-2223	22.0	23.0	Particulate HB?	No photo available?	As above (19-21), except 7.5YR 5/8 strong brown	2		Ask Battelle to take a photo prior to grinding, if not too late
INC-20-SOI-0305	3.5	5.0	GB		Sand, fine to medium, slightly moist, GLEY 4/1 1013 dark bluish gray, trace bright green, trace 5YR 5/8 yellowish red (3.5-3.8)	2		
INC-29S-SOI-14-1	14.2	14.4	HB		Disturbed description: Fine to coarse sand, moist to wet, 2.5YR 4/4 reddish brown, weakly indurated	1		During pitcher tube collection, driller states "it felt real granular, not hard like usual"
INC-29S	14	16						Photo shown is disturbed DPT sample from 13.6 – 15.5. Transitions from weakly indurated HB from

Field Sample ID	Start Depth (ft)	End Depth (ft)	Classification	Photo	Lithology Description	XRD Priority[1]	SEM	Comments
								13.8 – 14.6 to GB at approximately 14.6 – 14.8
INC-29S-SOI-14-2	14.6	14.7		See Above	Very fine to fine sand, GLEY1 2.5/N black, moist to wet, trace gravel	1		Transition sample
INC-29S-SOI-14-3	14.8	14.9		See Above	As above (14.6 – 14.7)	1		Completed transition to GB
DMT-33S-SOI-1518	15.5	18.0	GB-SM		Silty sand, wet, particulate 10YR 2/1 black and 10YR 3/1 dark gray, slight plasticity, trace bright green	2		Just below the water table in a column of GB material (9.0 – 18.0)

XRD Priorities:

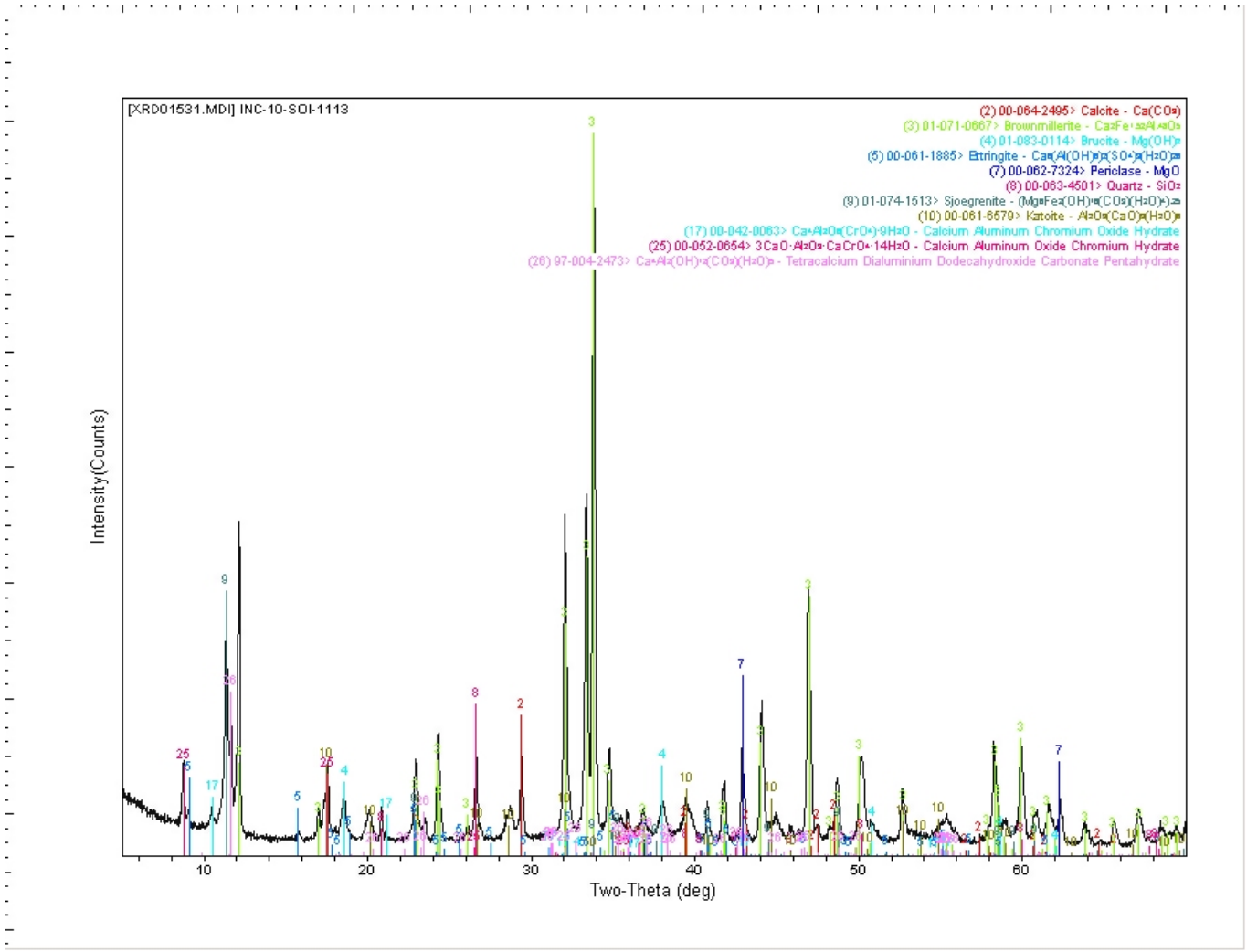
1 = Highest Priority

2 = Second Level Priority

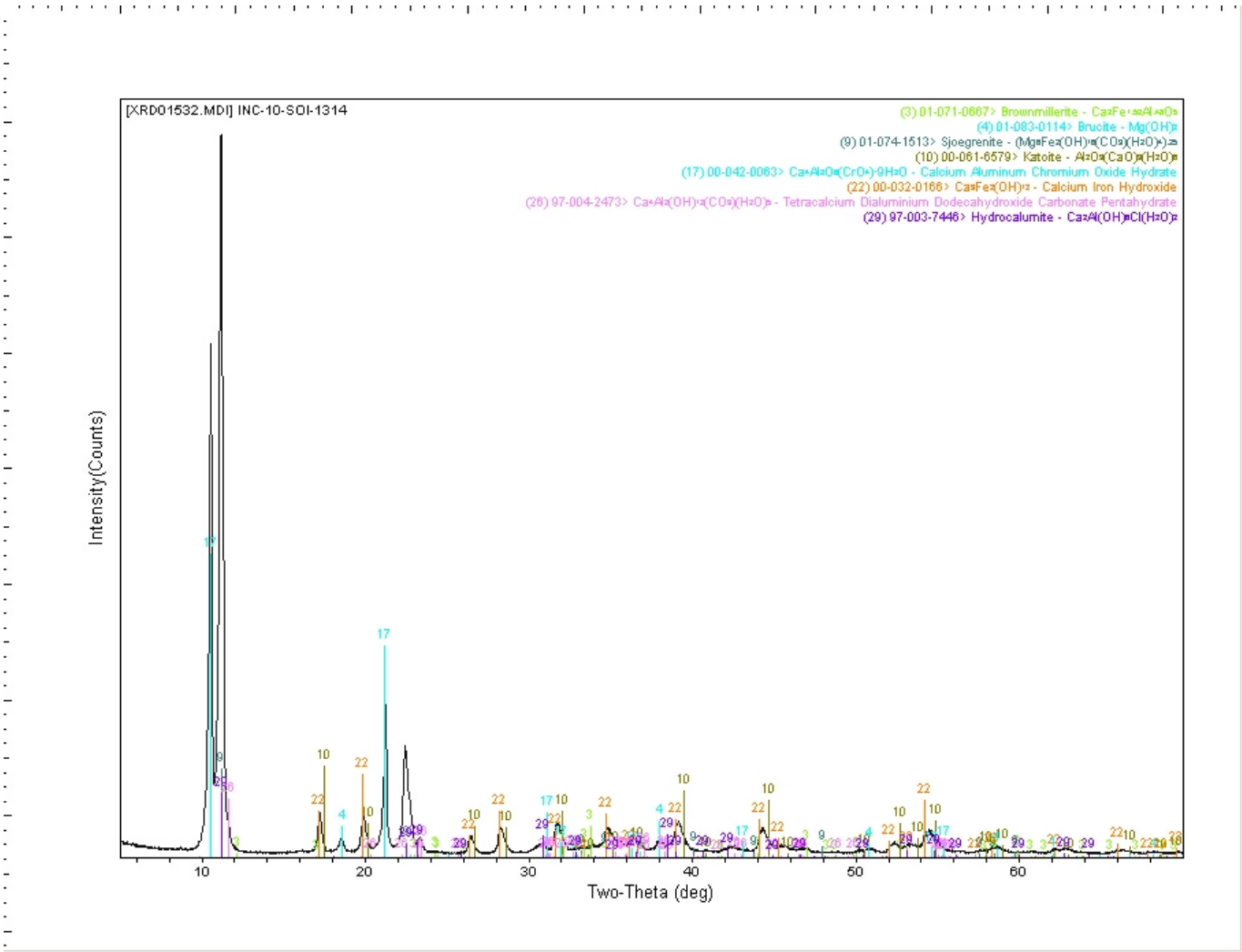
3 = NO analyses at this time, archive

APPENDIX B

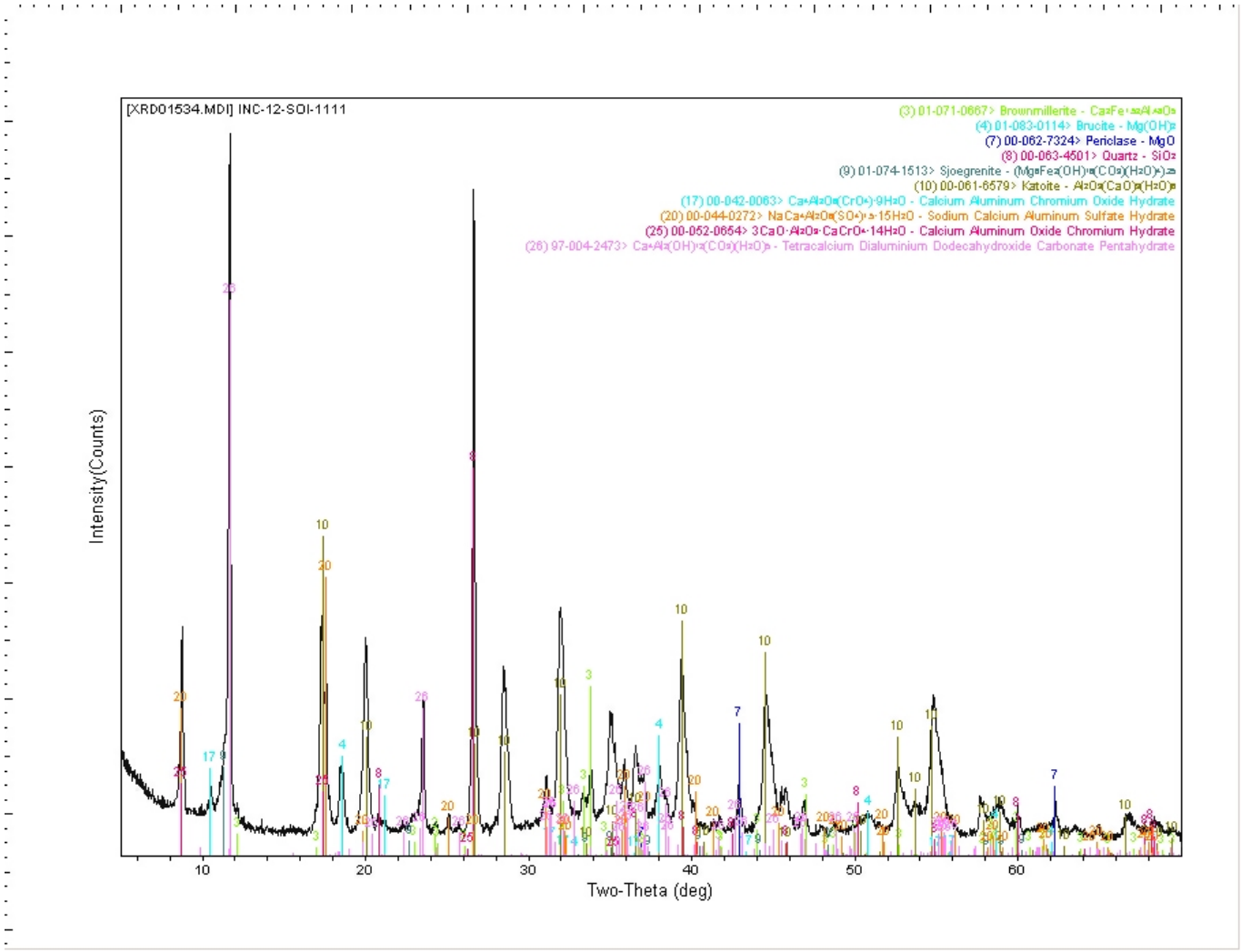
XRD PATTERNS



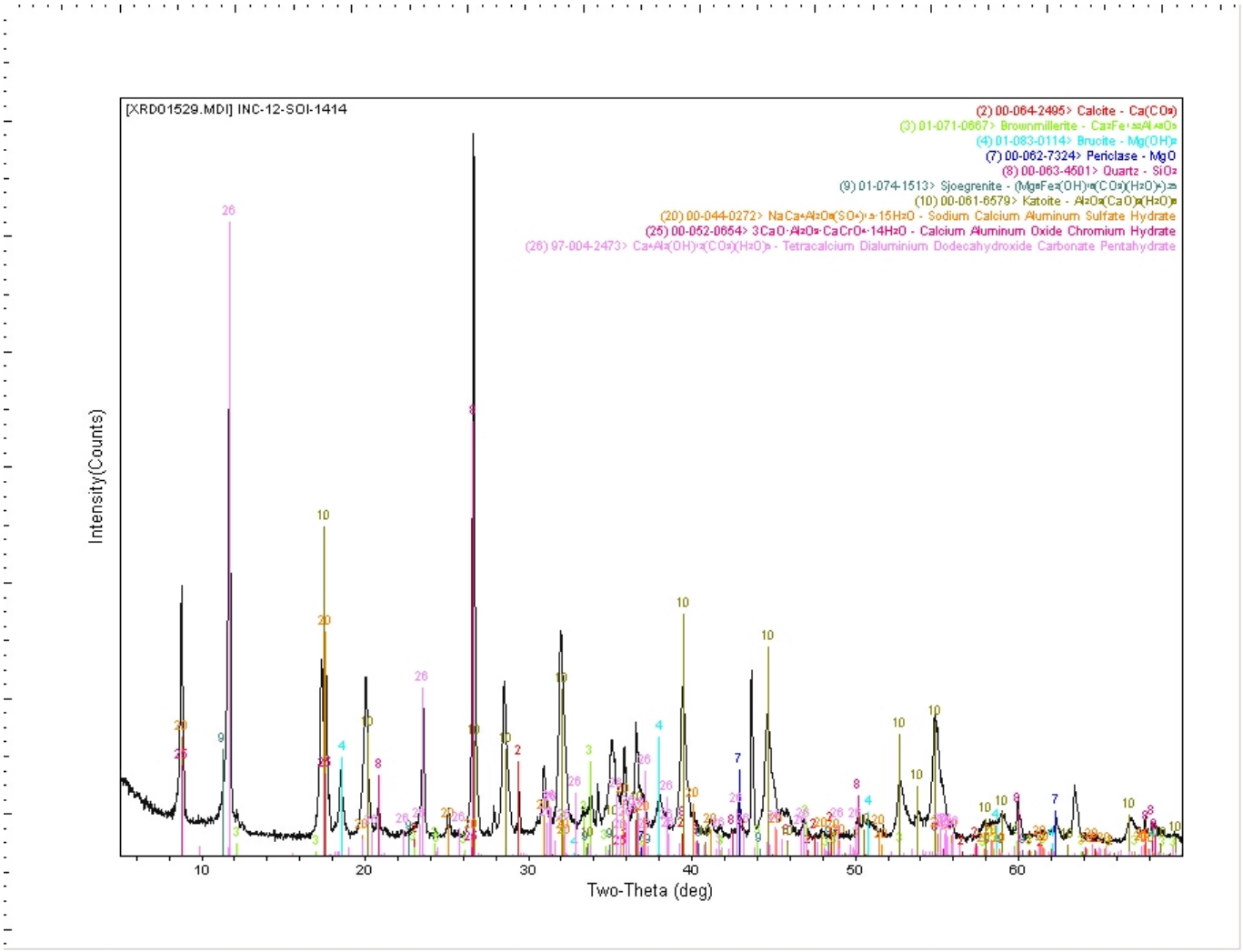
Sample INC-10-SOI-1113



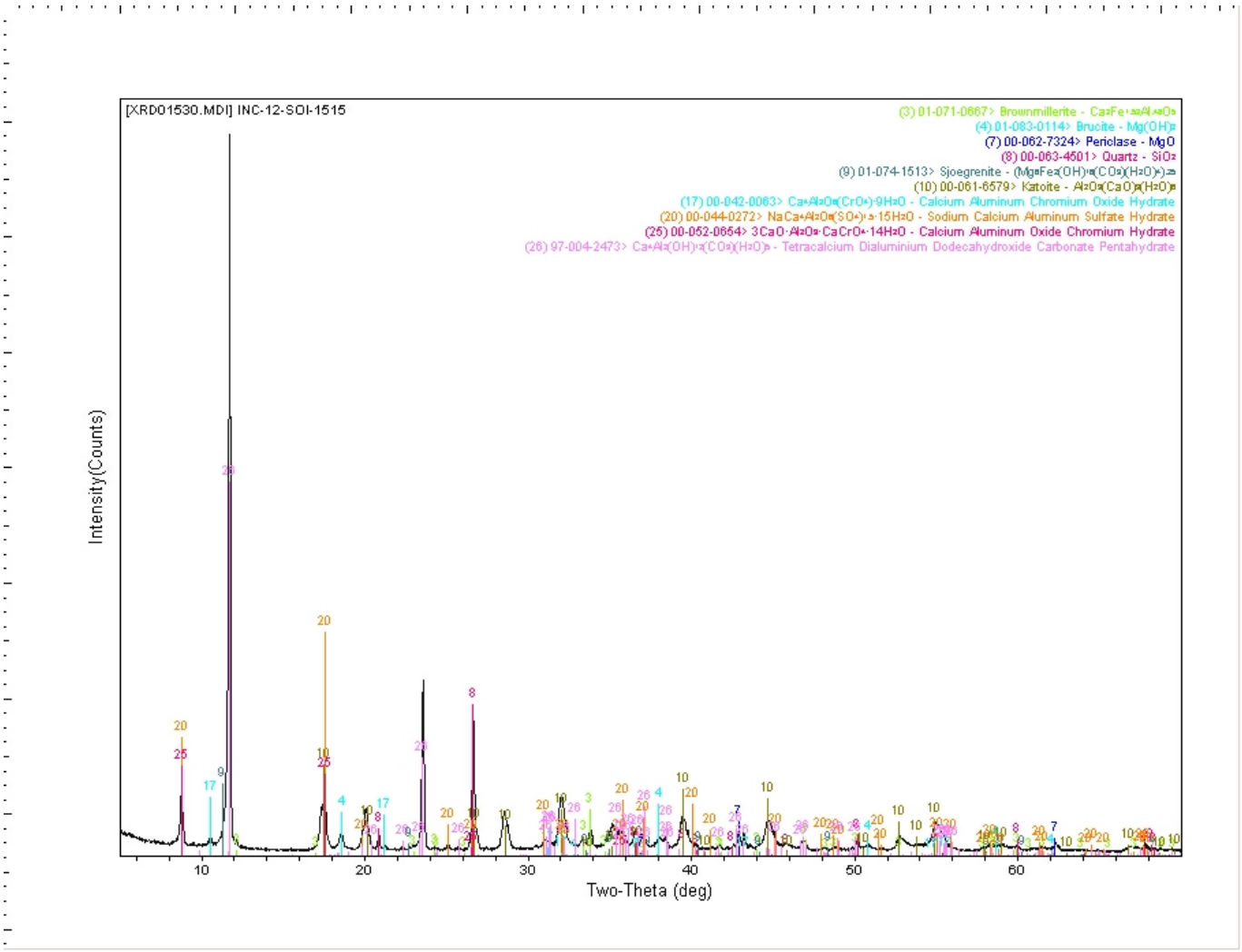
Sample INC-10-SOI-1314



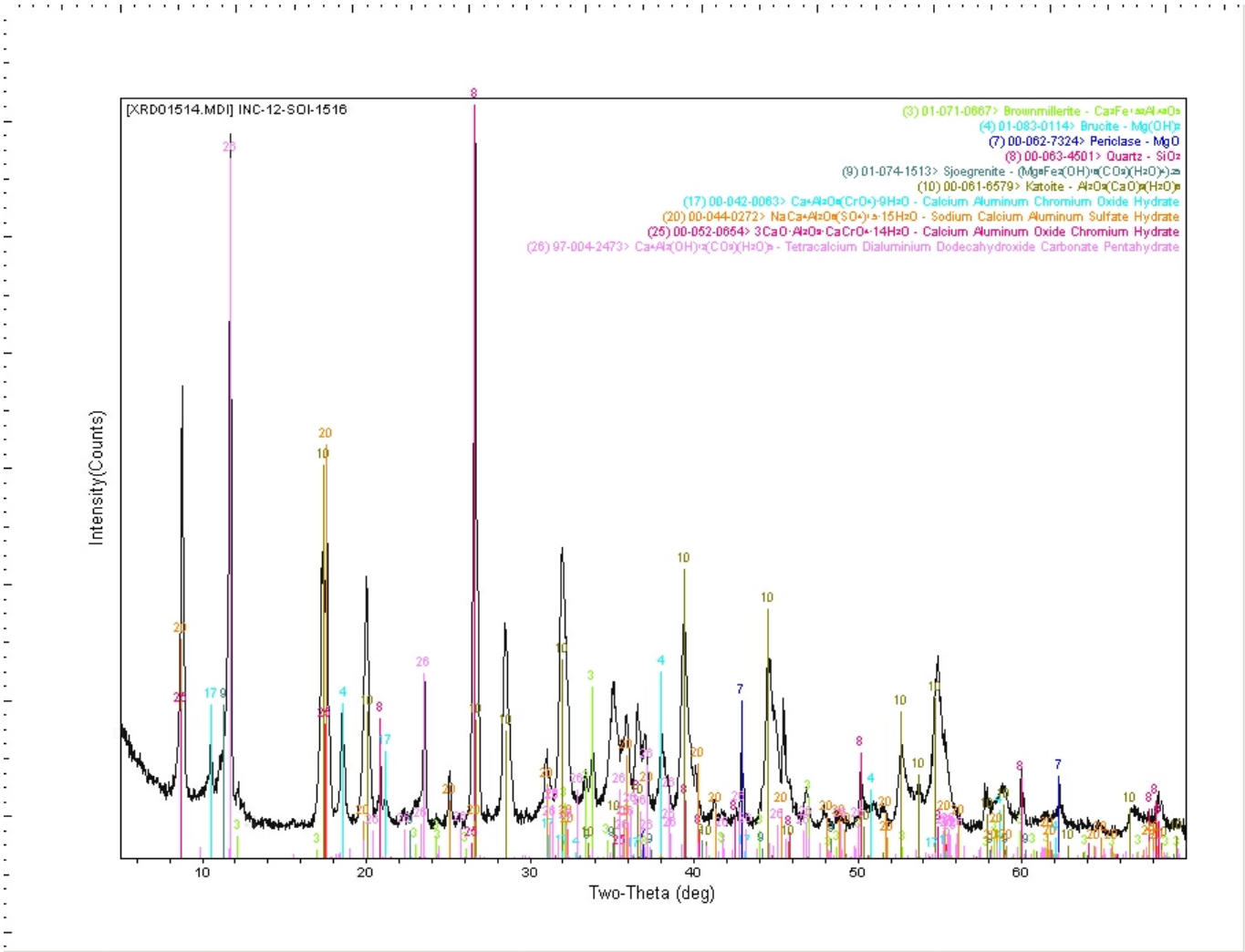
Sample INC-12-SOI-1111



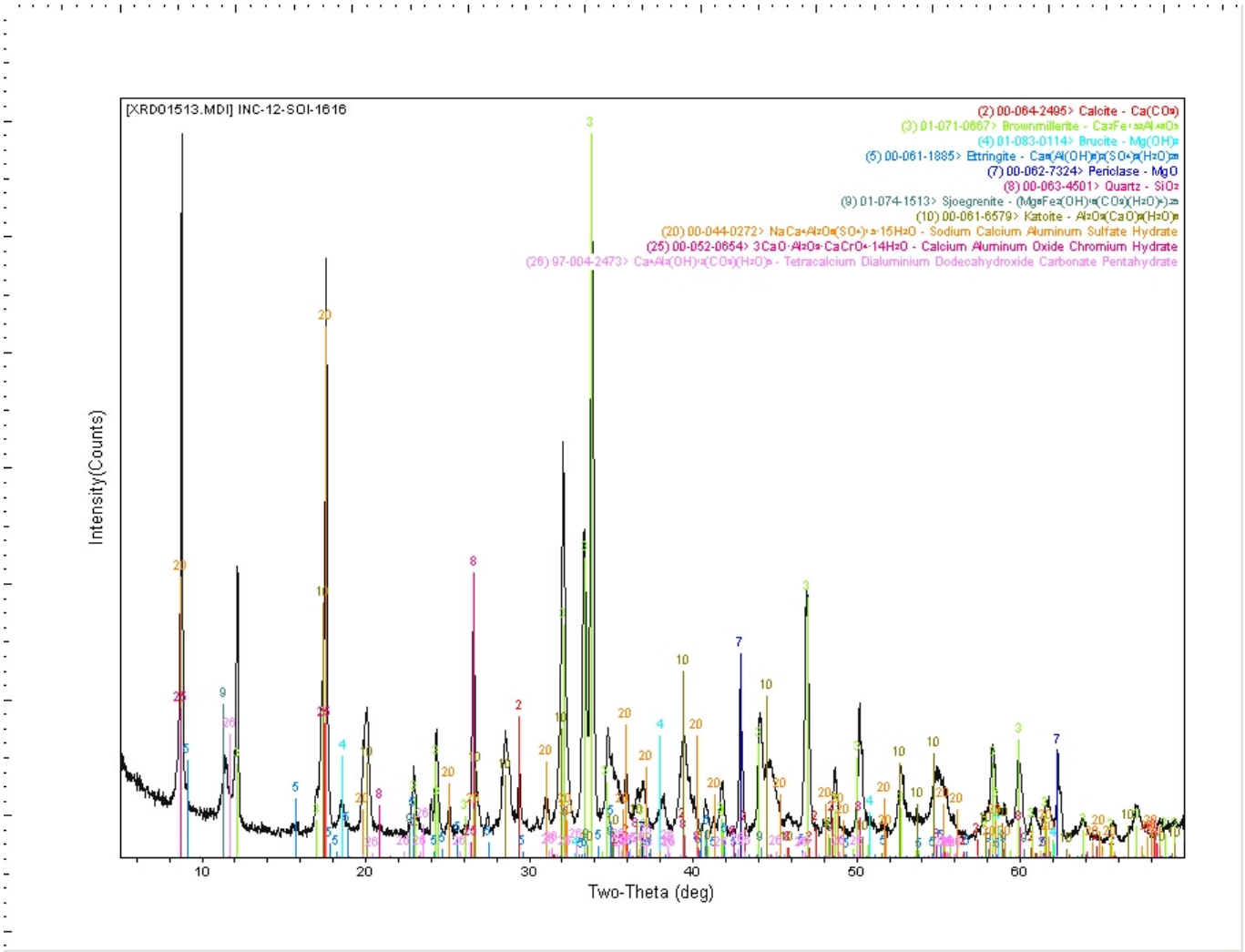
Sample INC-12-SOI-1414



Sample INC-12-SOI-1515



Sample INC-12-SOI-1516



Sample INC-12-SOI-1616

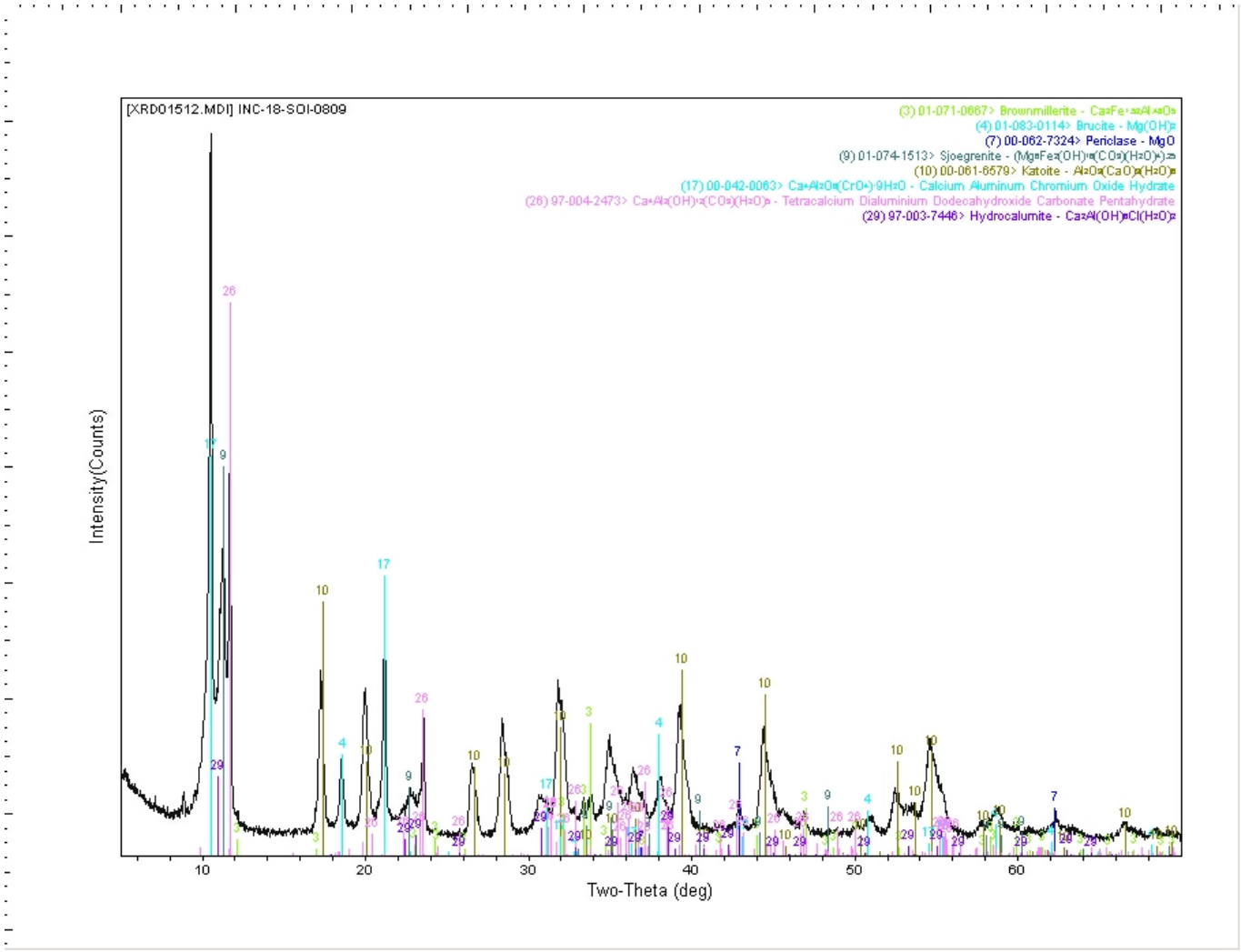
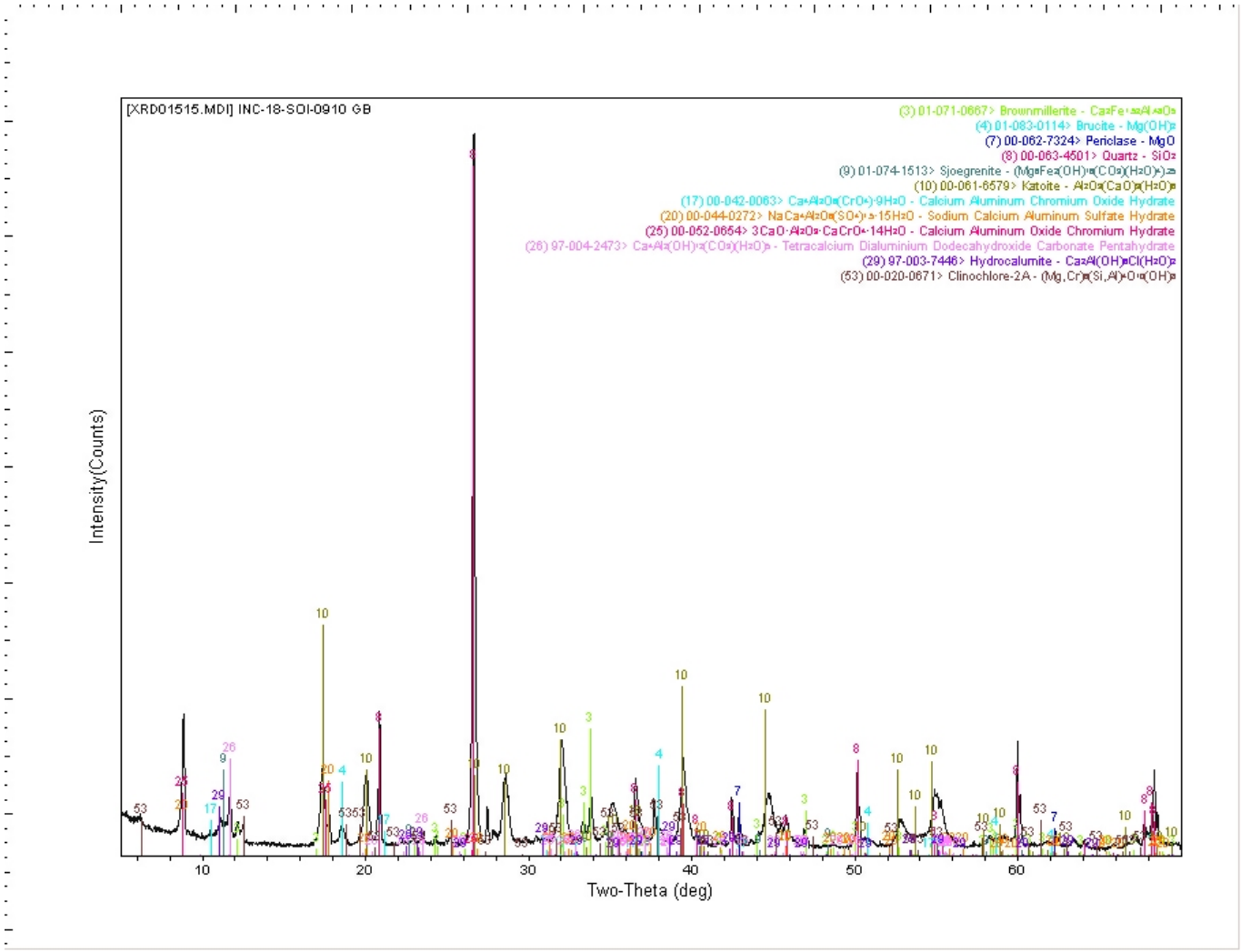
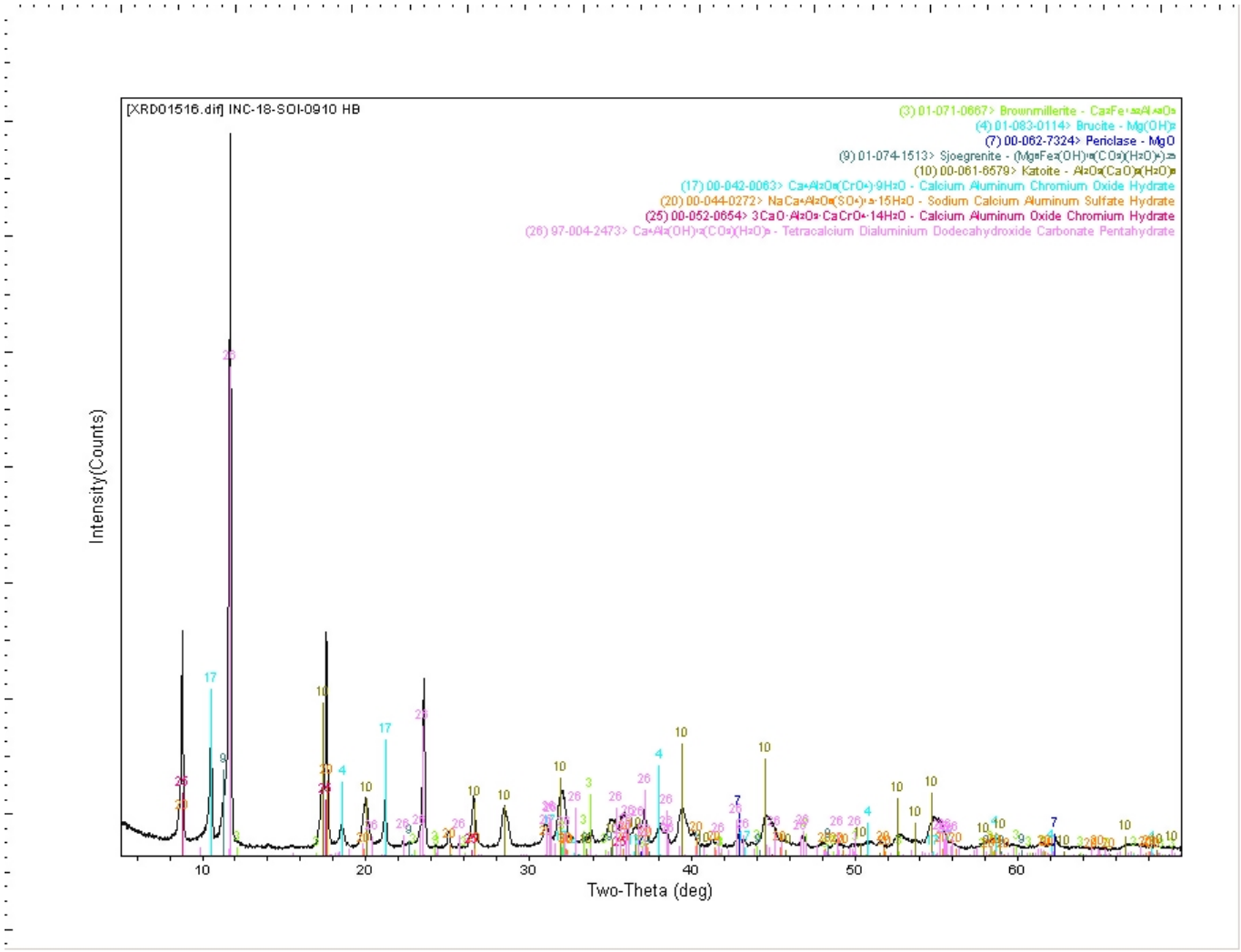


Figure 1INC-18-SOI-0809



Sample INC-18-SOI-0910



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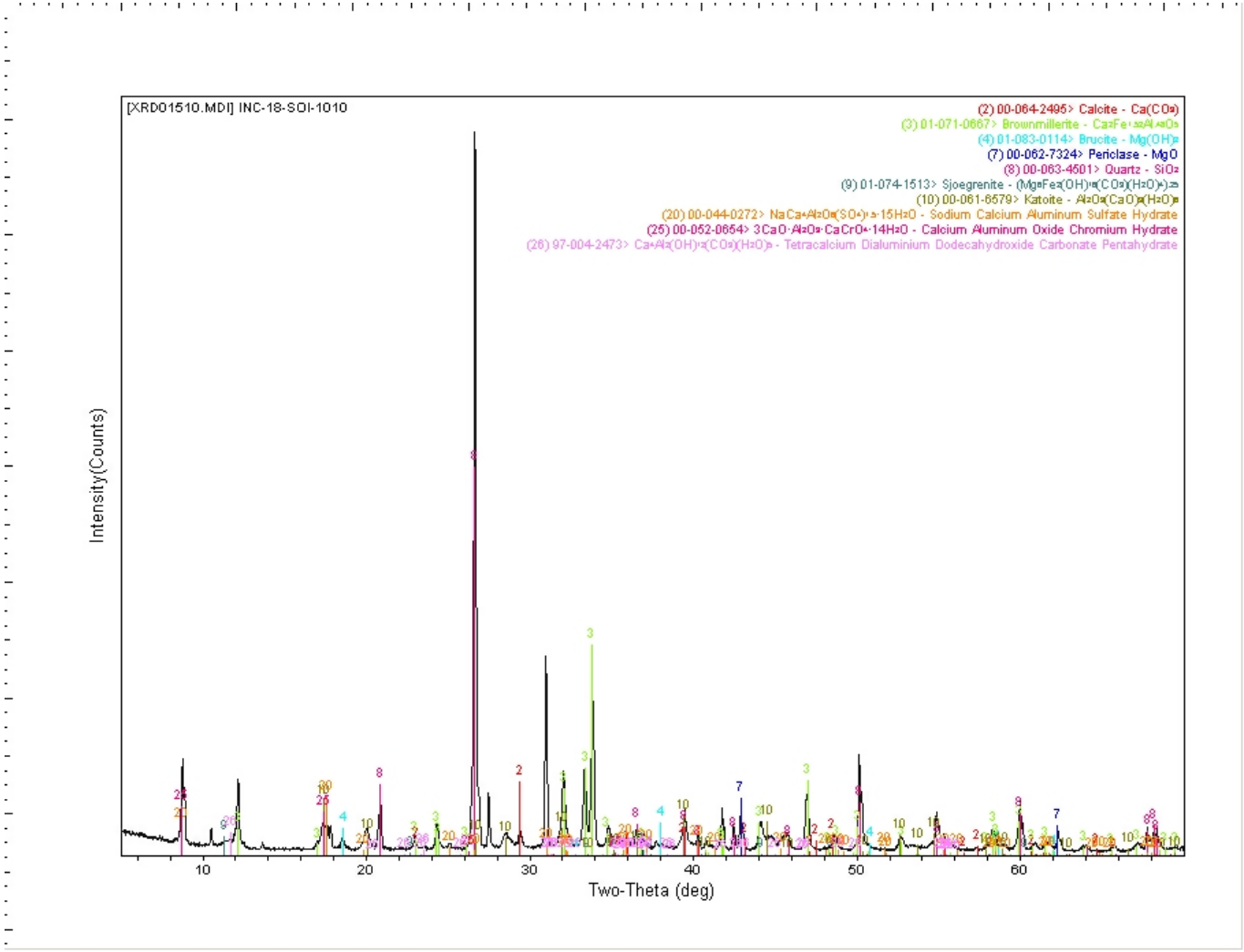
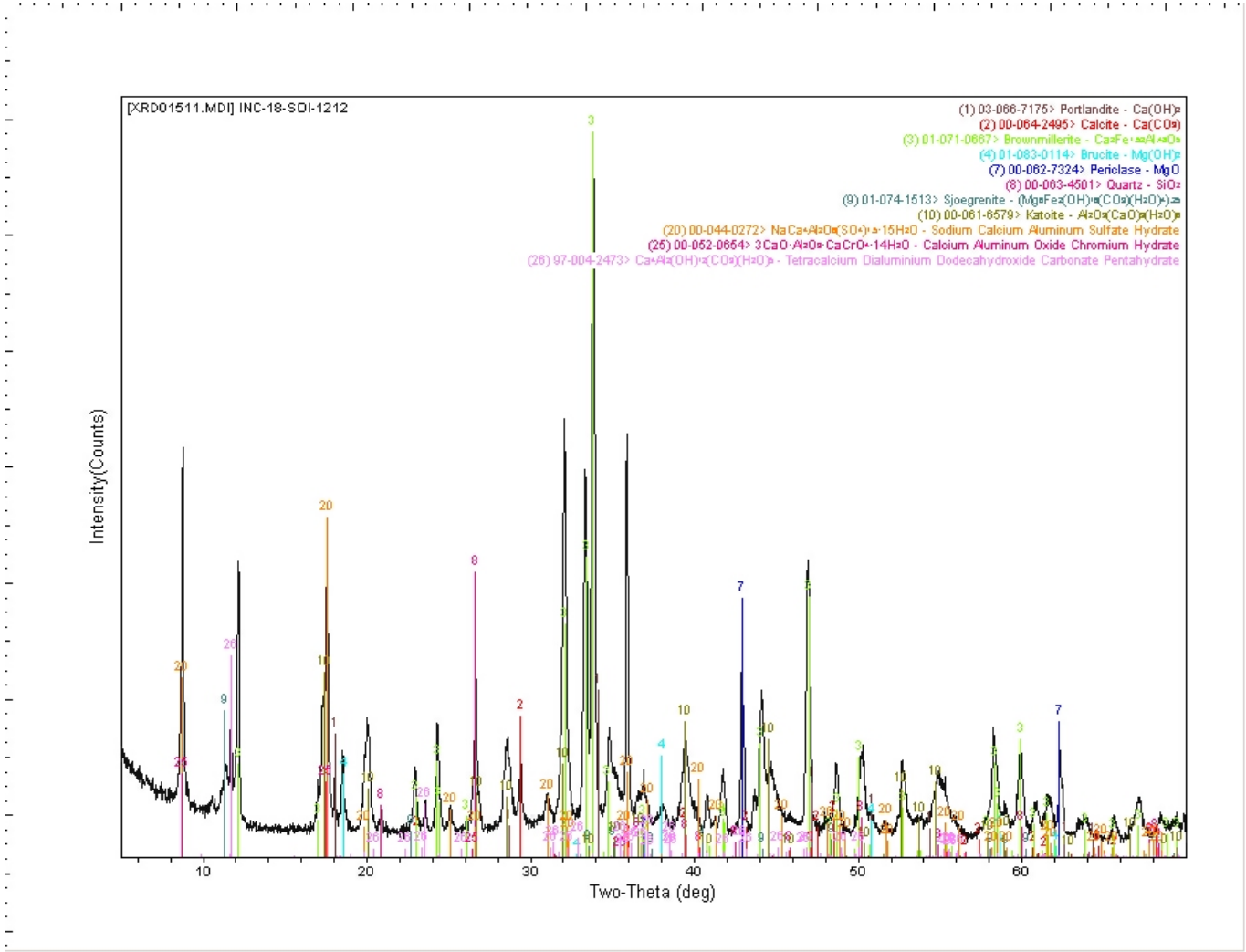
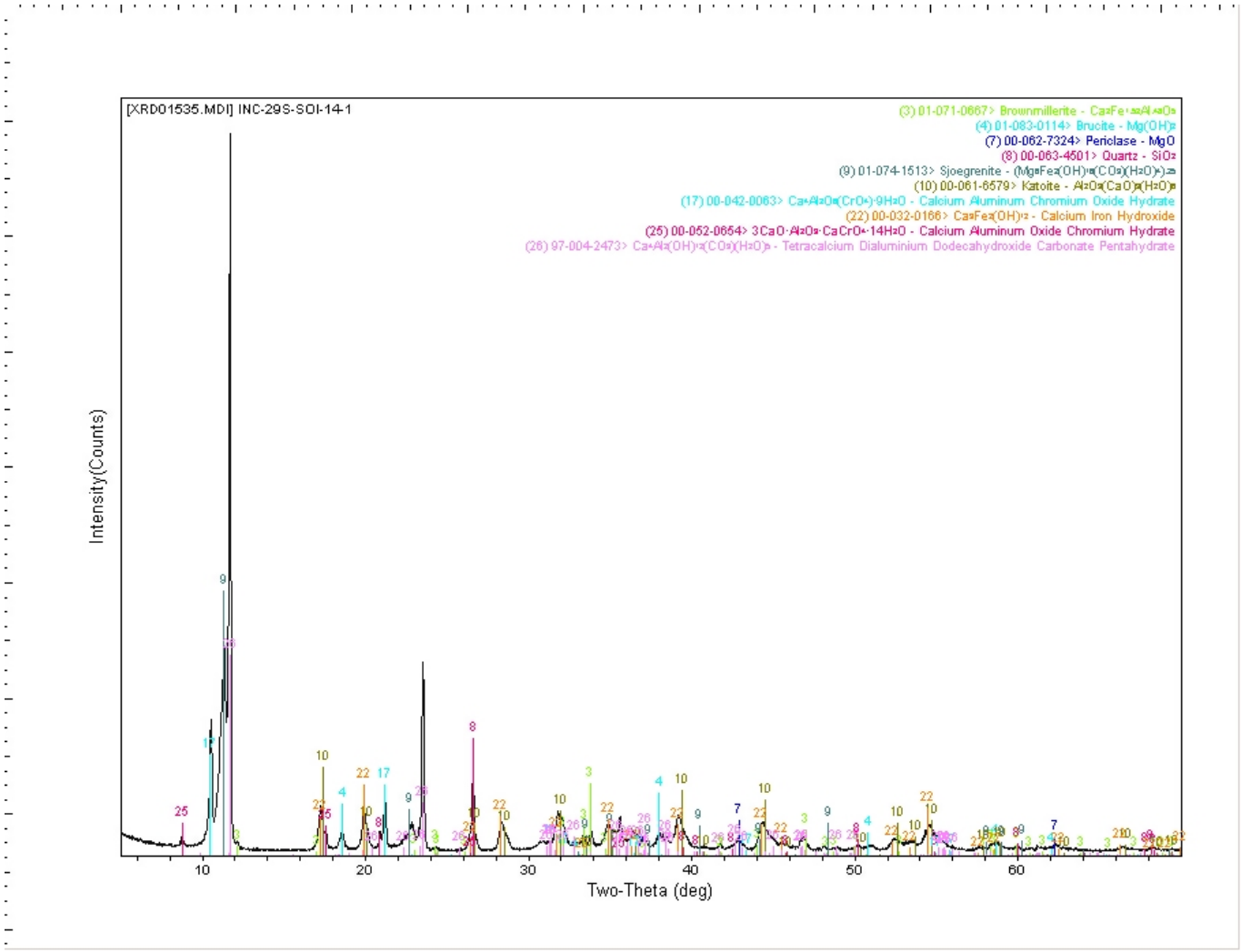


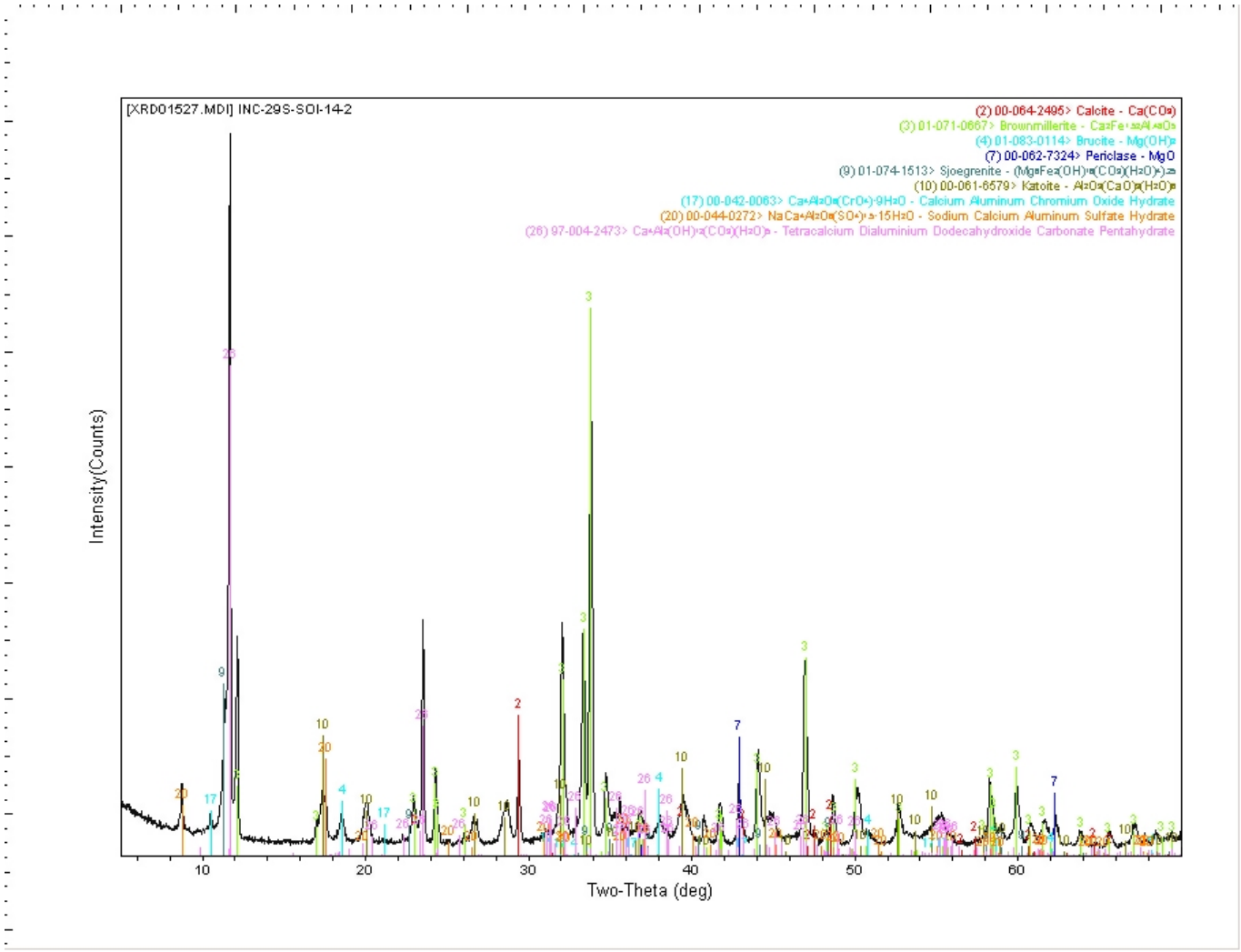
Sample INC-18-SOI-1010



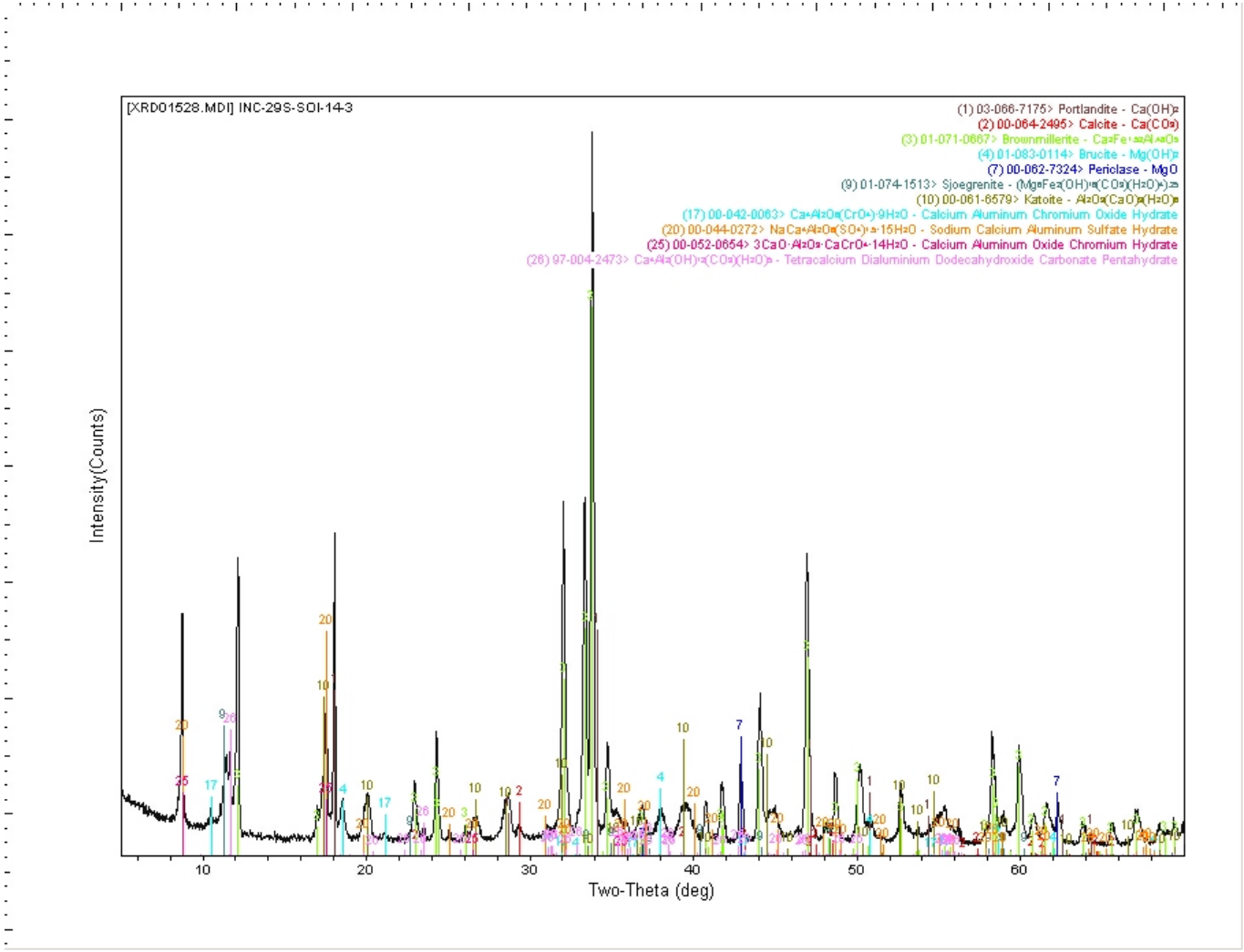
Sample INC-18-SOI-1212



Sample INC-29S-SOI-14-1



Sample INC-29S-SOI-14-2



Sample INC-29S-SOI-14-3

APPENDIX C

QUALITY ASSURANCE

QA PROCEDURES

C.1 XRD

Instrument: Rigaku D/MAX Wide Area X-Ray Diffractometer (XRD) with Materials Data, Inc., PC-based automation system and JADE7+ analysis software package.

JADE does sophisticated x-ray pattern processing and features an extensive Search/Match programming module with accessibility to approximately 255,000 crystalline (inorganic and organic) indexed substances (Powder Diffraction File-4+ 2006 RDB and the Inorganic Crystal Structure Database/ NIST version 2006-2).

Routine maintenance and alignment is preformed by the lab manager. Standard operating parameters for the XRD are: 40kV and 30mA, using a Copper normal focus x-ray tube. The standard divergence and scatter slits of 0.5 degree and receiving slit of 0.3 mm are employed. The XRD is further equipped with a curved single crystal (graphite) monochromator and a 0.6 mm slit in front of the scintillation counter.

Instrument performance is evaluated by running an external standard, NBS 640b Silicon. An XRD pattern is collected for the six relevant peaks located between 25 and 90 degrees two-theta to examine the linear calibration of the goniometer and intensities collected. The primary silicon standard (in a 0.2 mm glass Rigaku holder) is run utilizing the same conditions as the unknown scans. A calibration curve can be made and then applied, if necessary, to the unknown x-ray patterns. This test is run on a regular basis to keep the instrument in excellent calibration and compliance across two-theta range of interest. Further, a rapid line scan of the Silicon (111) peak is employed daily to check the goniometer alignment.

C.2 SEM

Instrument: JEOL 840A Scanning Electron Microscope (SEM) with an Oxford INCA 300 (Issue 16 Microanalysis Suite) Energy Dispersive Spectroscopy (EDS) system and software.

A service contract is maintained on the JEOL 840A to keep the instrument in top working order. All facets of operation are checked and adjusted, including magnification and image resolution.

Verification of the performance of the Oxford EDS system is accomplished by two tests. First, a Quant Optimization is done before collecting any EDS data. This procedure allows the software to calculate the exact peak positions and check the resolution of the EDS detector for optimal results. Cobalt metal is used in this test. The second test employed is to collect a copper energy dispersive spectrum (K and L lines) to demonstrate the Quant Optimization results. These tests are run on a regular basis to keep the instrument in excellent calibration and compliance across energy spectrum of interest.

APPENDIX D

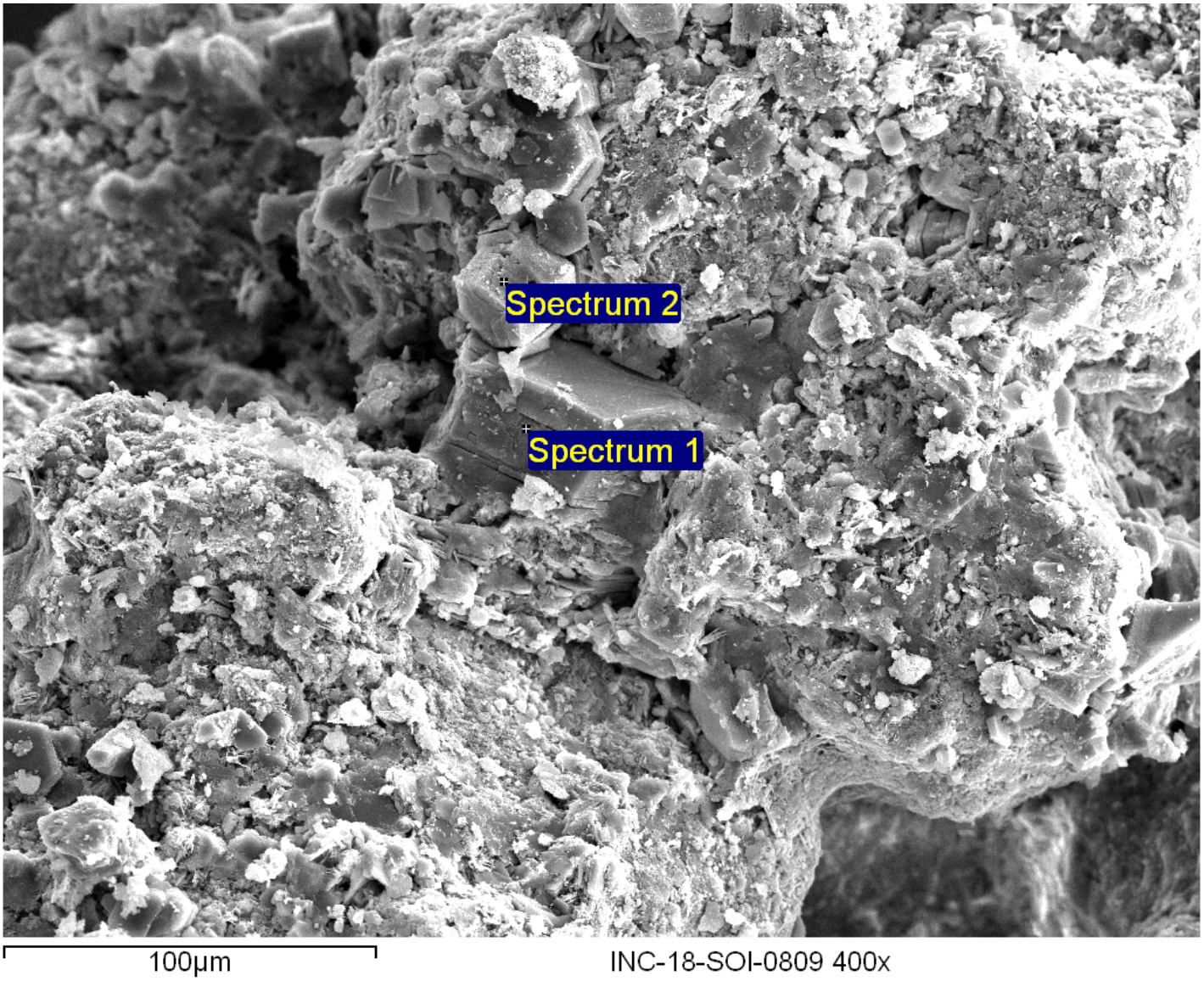
SEM FILES

INC-18-SOI-0809

Spectrum	In stats.	O	Na	Mg	Al	S	Cl	Ca	Cr	Fe
Spectrum 1	Yes	42.87	0.00	1.38	1.75	0.00	0.77	37.57	2.12	13.54
Spectrum 2	Yes	77.48	0.00	0.00	5.82	0.00	0.44	13.64	0.30	2.33
Mean		60.18	0.00	0.69	3.78	0.00	0.60	25.60	1.21	7.93
Std. deviation		24.47	0.00	0.98	2.88	0.00	0.24	16.92	1.29	7.93

All results in atomic percent.

Magnification 400x

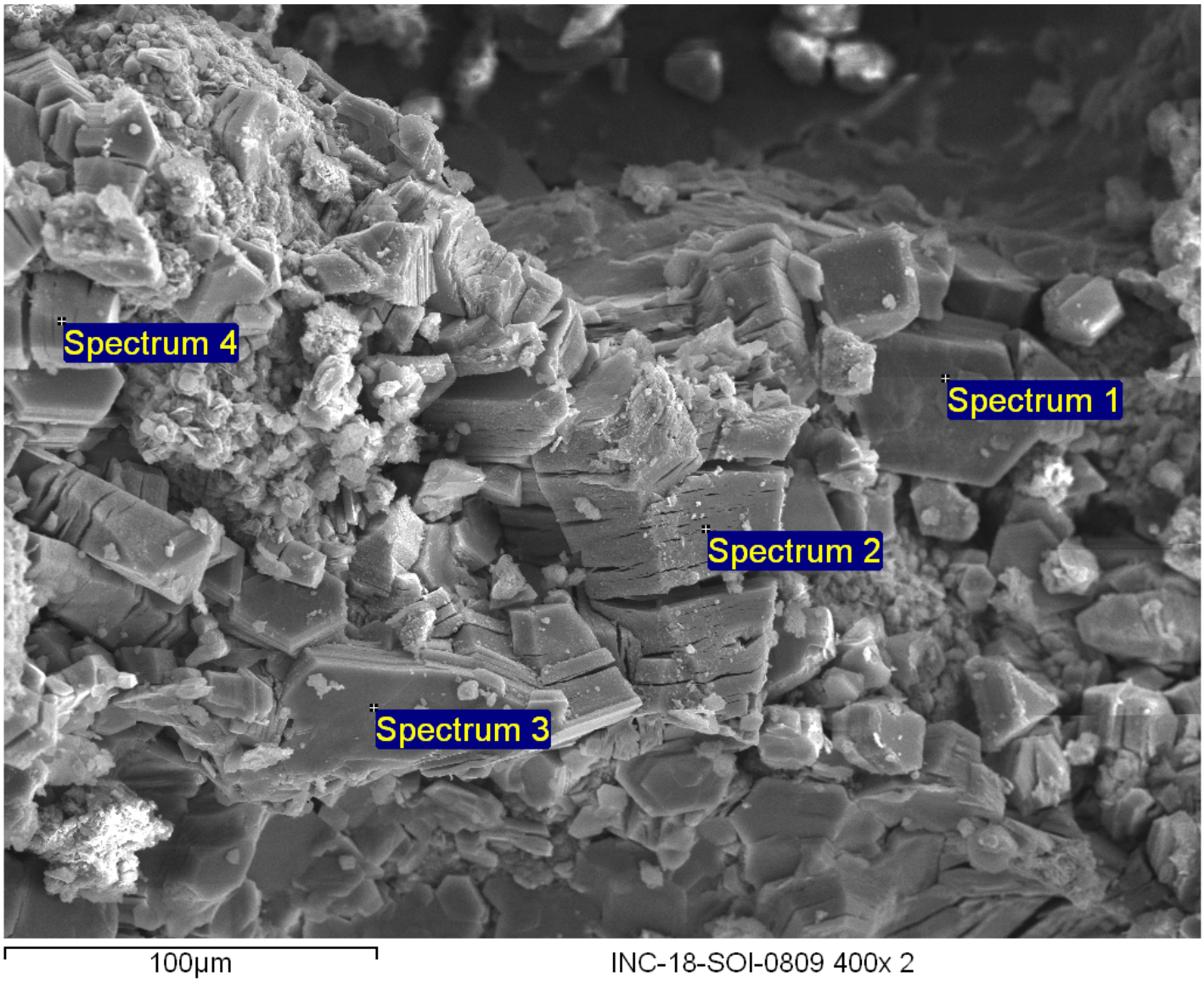


INC-18-SOI-0809

Spectrum	In stats.	O	Na	Mg	Al	S	Cl	Ca	Cr	Fe
Spectrum 1	Yes	71.38	0.00	0.00	6.70	0.00	0.00	16.51	3.38	2.03
Spectrum 2	Yes	80.83	0.00	0.00	5.10	0.00	0.27	10.92	1.17	1.71
Spectrum 3	Yes	65.40	0.00	0.00	5.48	0.00	0.33	21.51	4.65	2.63
Spectrum 4	Yes	73.18	0.00	0.00	4.85	0.00	0.00	15.29	2.88	3.79
Mean		72.70	0.00	0.00	5.53	0.00	0.15	16.06	3.02	2.54
Std. deviation		6.36	0.00	0.00	0.82	0.00	0.18	4.36	1.44	0.91

All results in atomic percent.

Magnification 400x 2

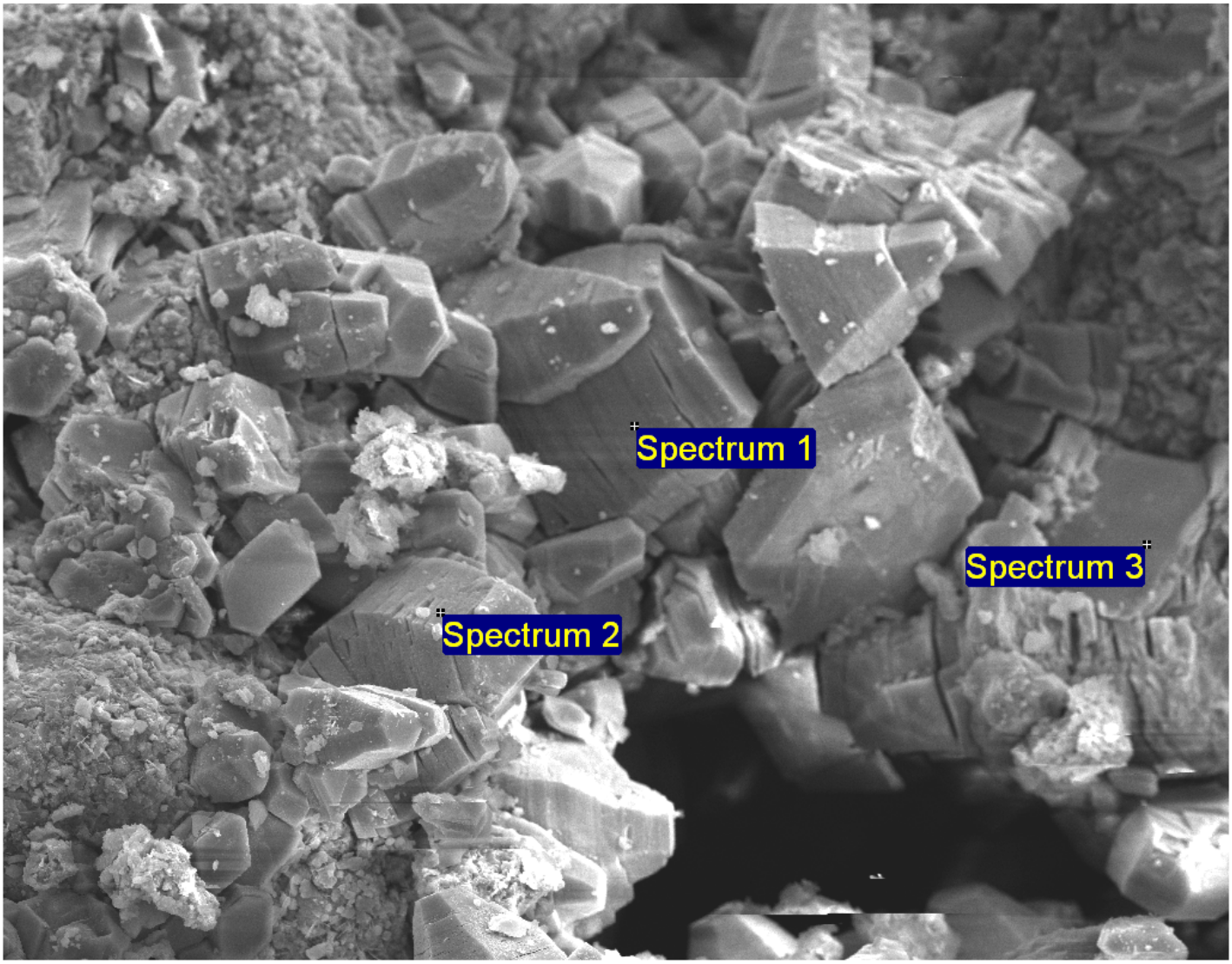


INC-18-SOI-0809

Spectrum	In stats.	O	Na	Mg	Al	S	Cl	Ca	Cr	Fe
Spectrum 1	Yes	65.76	0.00	0.00	6.69	0.00	0.33	22.81	1.89	2.52
Spectrum 2	Yes	81.93	0.00	0.00	5.17	0.00	0.00	10.18	1.16	1.56
Spectrum 3	Yes	74.51	0.00	0.00	6.95	0.00	0.55	14.69	2.09	1.21
Mean		74.07	0.00	0.00	6.27	0.00	0.29	15.89	1.72	1.76
Std. deviation		8.09	0.00	0.00	0.96	0.00	0.28	6.40	0.49	0.68

All results in atomic percent.

Magnification 400x 3



100µm

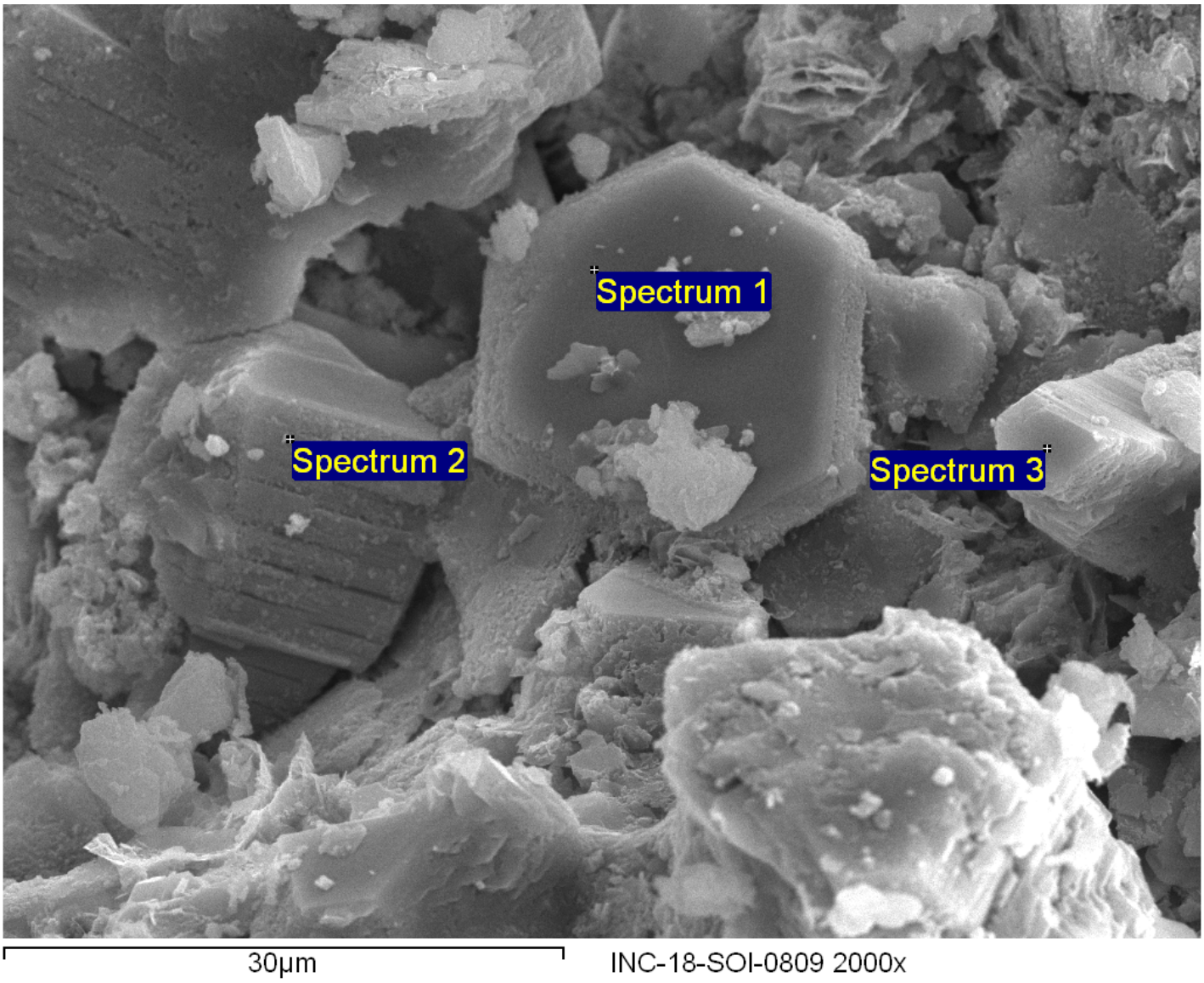
INC-18-SOI-0809 400x 3

INC-18-SOI-0809

Spectrum	In stats.	O	Na	Mg	Al	S	Cl	Ca	Cr	Fe
Spectrum 1	Yes	72.46	0.00	0.00	6.25	0.00	0.56	17.24	1.22	2.26
Spectrum 2	Yes	79.15	0.00	0.00	4.68	0.00	0.46	12.52	0.37	2.82
Spectrum 3	Yes	70.55	0.00	0.00	5.35	0.00	0.82	18.55	0.25	4.48
Mean		74.06	0.00	0.00	5.43	0.00	0.61	16.10	0.61	3.19
Std. deviation		4.52	0.00	0.00	0.79	0.00	0.18	3.17	0.53	1.16

All results in atomic percent.

Magnification 2000x

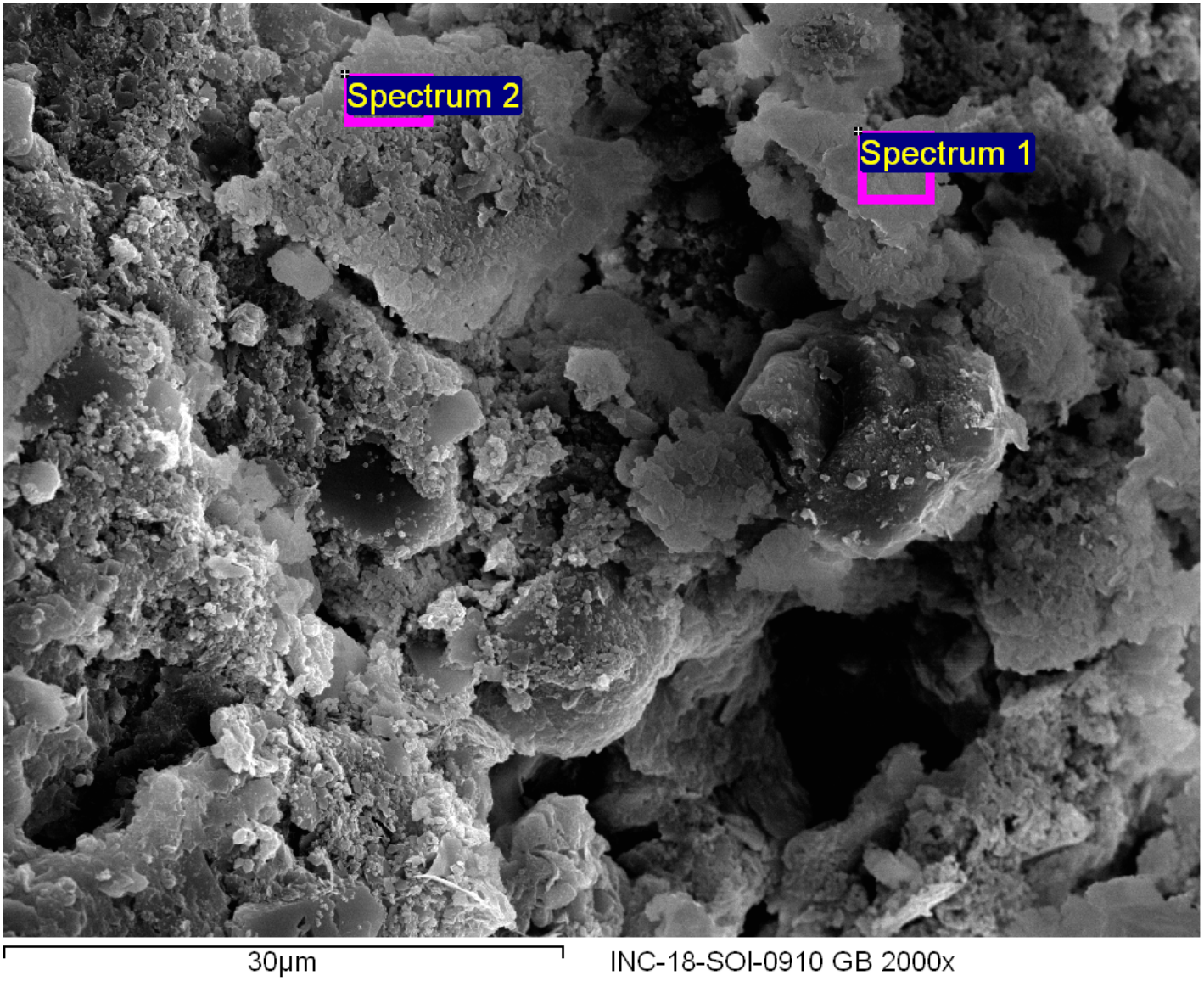


INC-18-SOI-0910 GB

Spectrum	In stats.	O	Na	Mg	Al	Si	S	K	Ca	Cr	Fe
Spectrum 1	Yes	73.00	0.00	10.04	2.96	6.60	0.00	0.00	4.72	0.45	2.21
Spectrum 2	Yes	72.50	0.00	1.11	7.51	9.19	0.00	0.90	6.00	0.42	2.37
Mean		72.75	0.00	5.58	5.24	7.89	0.00	0.45	5.36	0.44	2.29
Std. deviation		0.35	0.00	6.32	3.22	1.83	0.00	0.64	0.90	0.02	0.11

All results in atomic percent.

Magnification 2000x

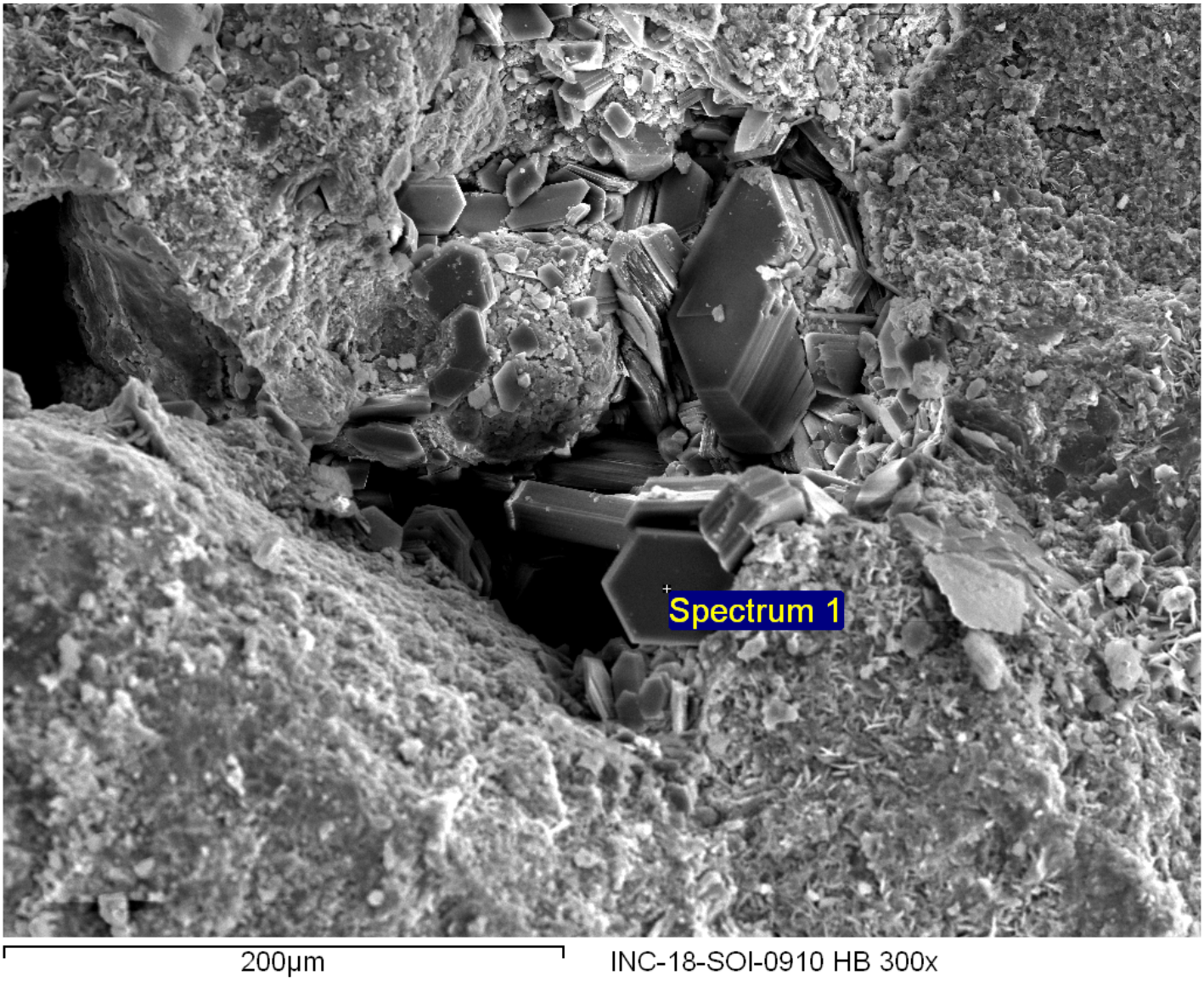


INC-18-SOI-0910 HB

Spectrum	In stats.	O	Na	Mg	Al	S	Ca	Cr	Fe
Spectrum 1	Yes	64.17	0.00	0.00	3.40	0.00	19.81	5.35	7.28
Mean		64.17	0.00	0.00	3.40	0.00	19.81	5.35	7.28
Std. deviation		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

All results in atomic percent.

Magnification 300x

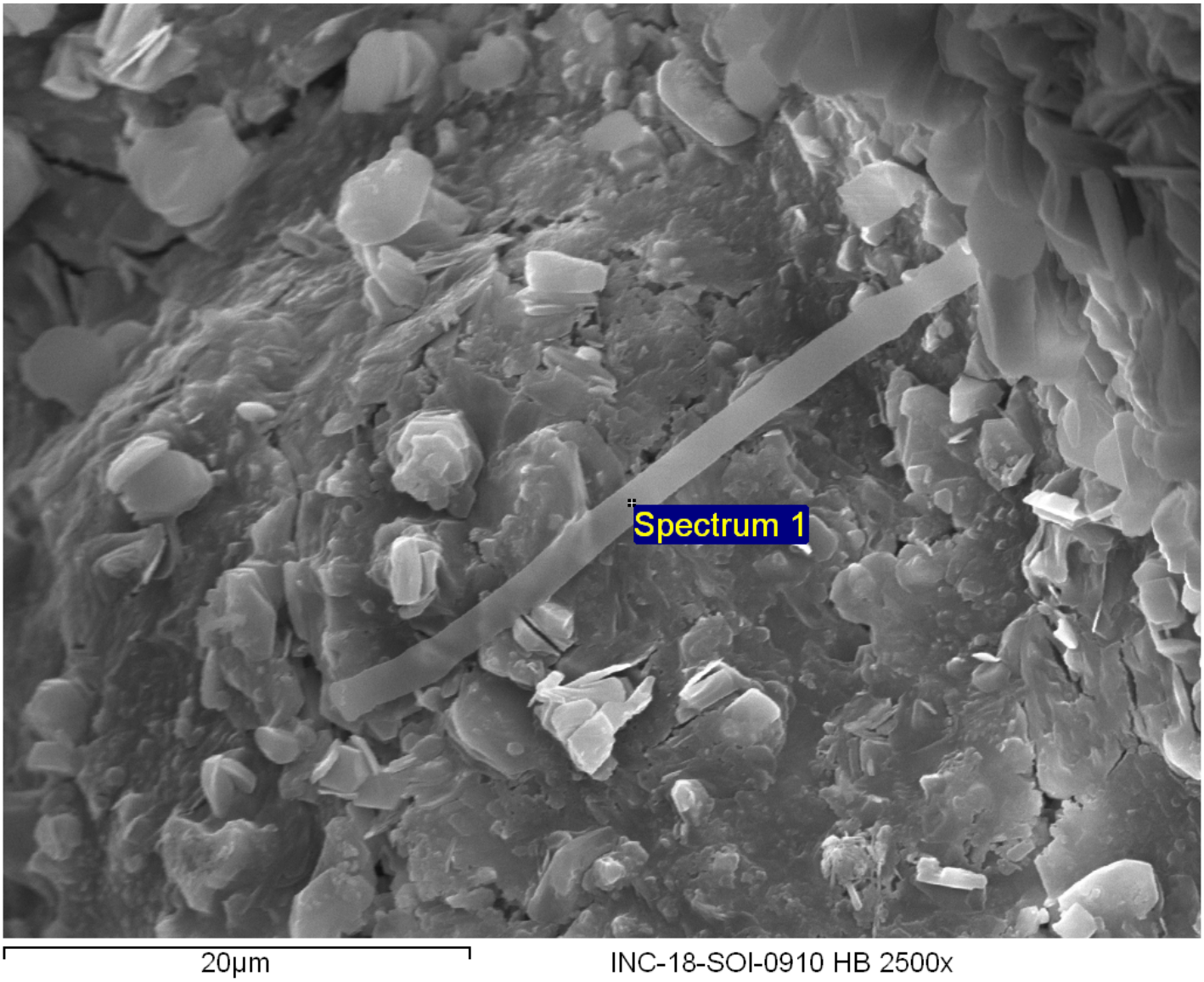


INC-18-SOI-0910 HB

Spectrum	In stats.	O	Na	Mg	Al	S	Ca	Cr	Fe
Spectrum 1	Yes	69.77	0.00	0.00	3.79	0.00	14.65	3.74	8.05

All results in atomic percent.

Magnification 2500x

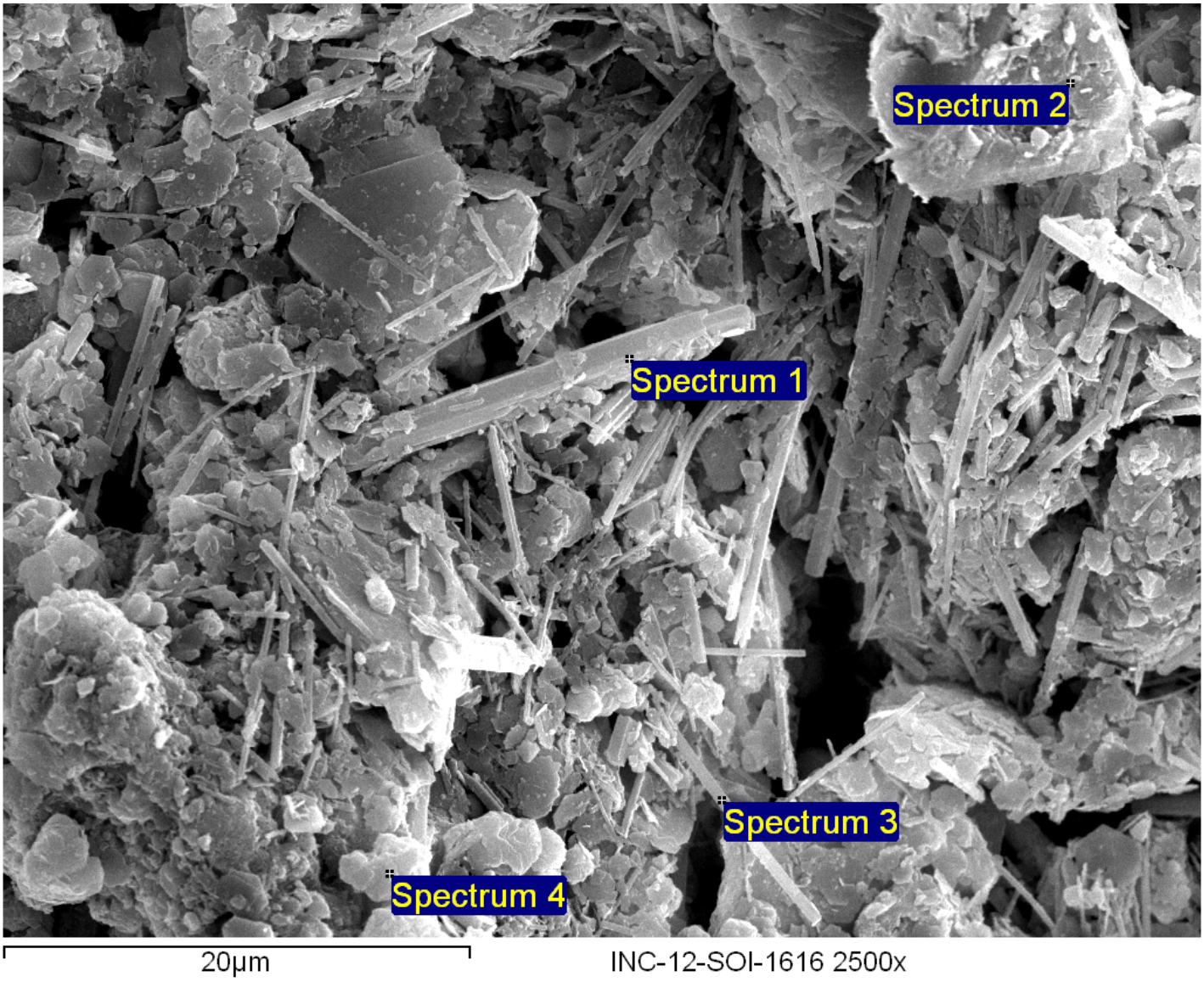


INC-12-SOI-1616

Spectrum	In stats.	O	Na	Mg	Al	Si	S	Ca	Cr	Fe
Spectrum 1	Yes	47.65	0.00	1.97	3.50	1.42	0.00	35.27	3.95	6.25
Spectrum 2	Yes	77.18	1.70	1.36	5.44	0.73	0.00	9.70	2.39	1.50
Spectrum 3	Yes	78.97	0.65	2.65	4.51	0.96	1.01	9.31	0.73	1.22
Spectrum 4	Yes	76.59	0.00	4.89	5.84	0.95	0.00	8.89	0.90	1.94
Mean		70.10	0.59	2.72	4.82	1.01	0.25	15.79	1.99	2.73
Std. deviation		15.00	0.80	1.54	1.04	0.29	0.51	12.99	1.51	2.36

All results in atomic percent.

Magnification 2500x

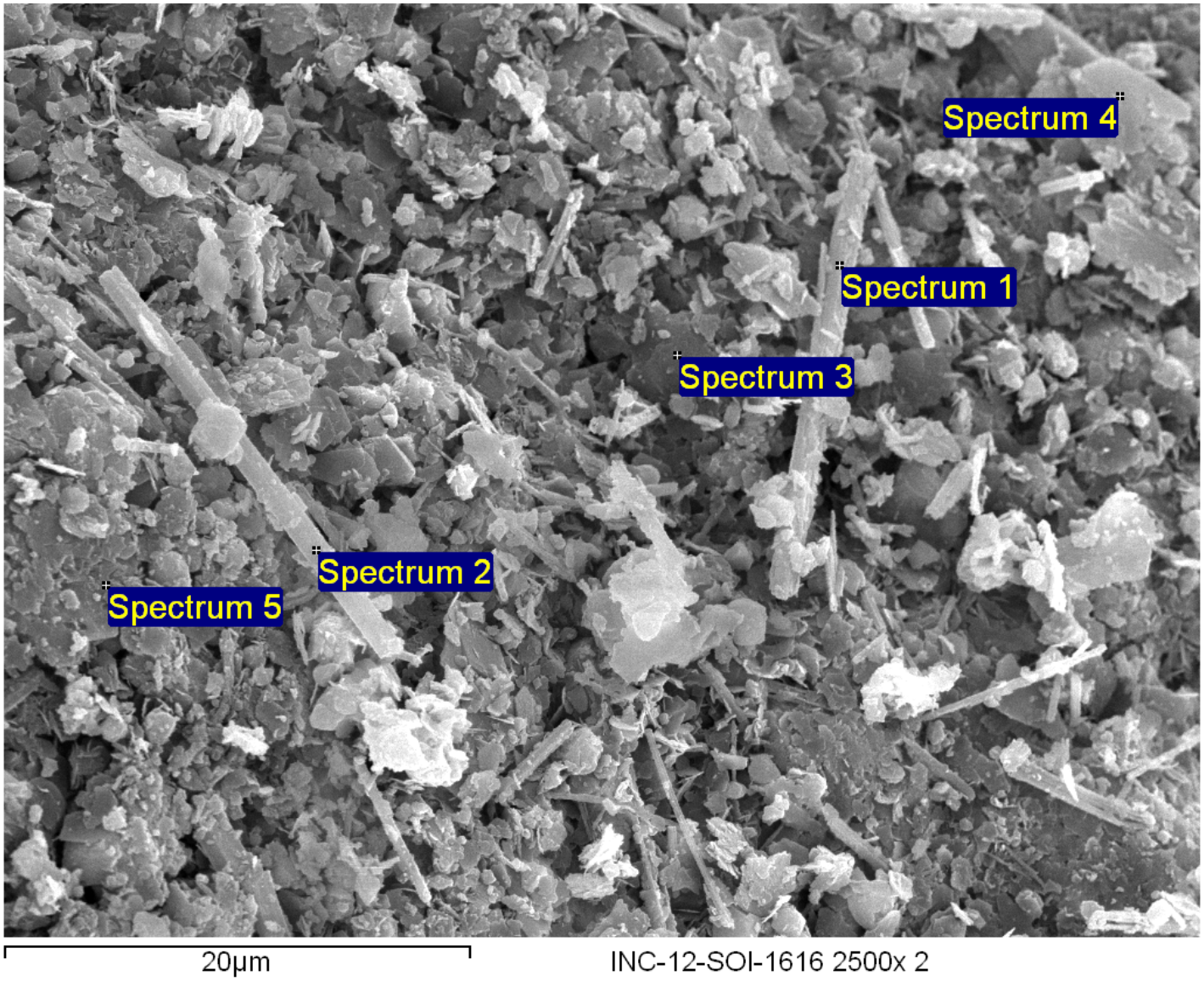


INC-12-SOI-1616

Spectrum	In stats.	O	Na	Mg	Al	Si	S	Ca	Cr	Fe
Spectrum 1	Yes	78.42	0.00	7.01	5.60	1.21	0.00	6.66	0.30	0.81
Spectrum 2	Yes	78.99	0.00	4.07	5.86	1.51	0.00	8.00	0.34	1.22
Spectrum 3	Yes	71.96	0.00	9.10	5.02	1.63	0.00	10.60	0.42	1.27
Spectrum 4	Yes	76.32	0.95	6.21	5.99	1.35	0.00	7.25	1.18	0.75
Spectrum 5	Yes	72.33	1.12	7.73	6.44	1.09	0.00	8.62	1.63	1.05
Mean		75.60	0.41	6.83	5.78	1.36	0.00	8.22	0.77	1.02
Std. deviation		3.31	0.57	1.87	0.53	0.22	0.00	1.52	0.60	0.23

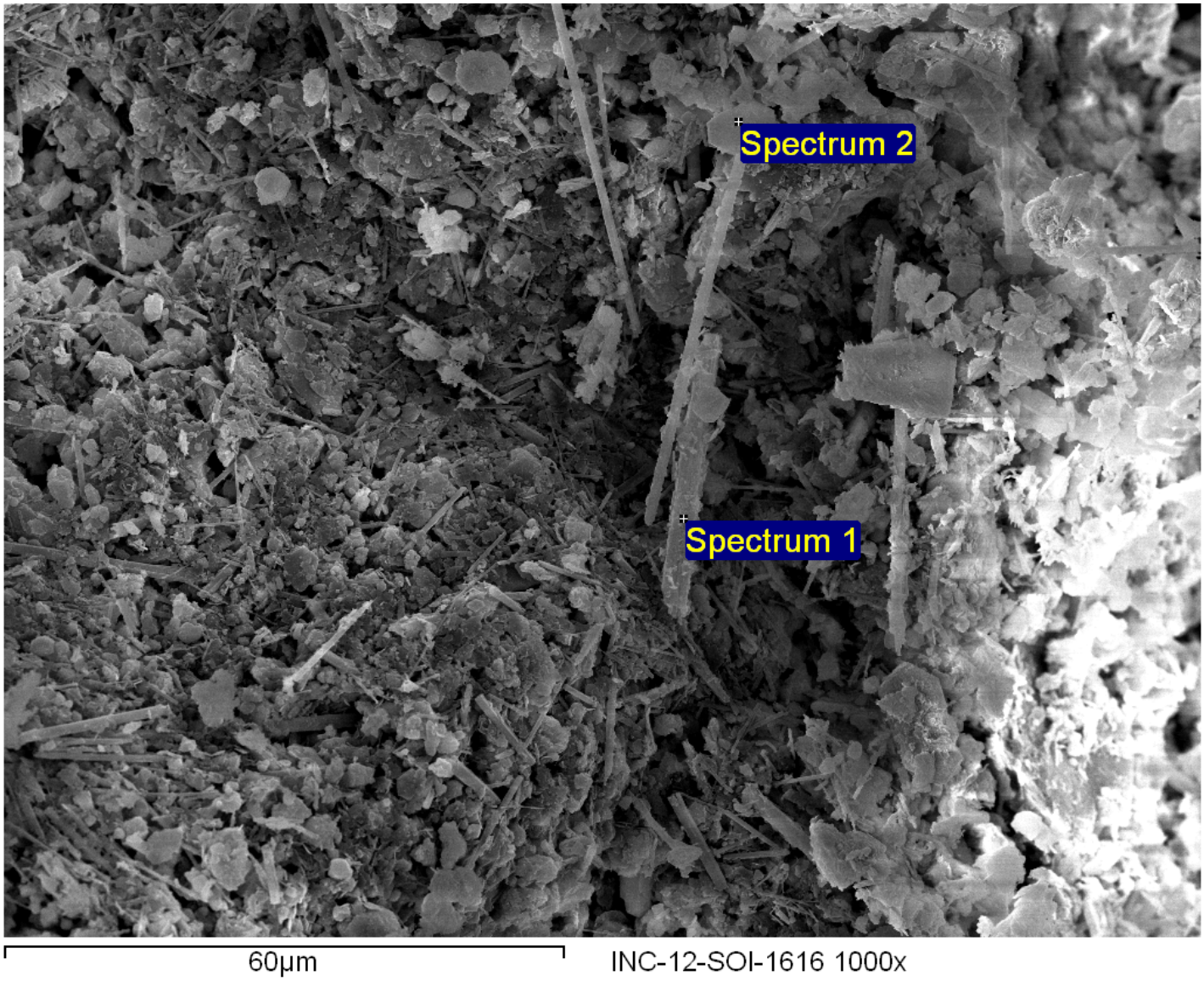
All results in atomic percent.

Magnification 2500x 2



INC-12-SOI-1616

Spectrum	In stats.	O	Na	Mg	Al	Si	S	Ca	Cr	Fe
Spectrum 1	Yes	79.72	0.56	1.94	5.03	0.69	2.32	9.13	0.15	0.46
Spectrum 2	Yes	71.49	0.73	2.40	5.89	1.35	0.00	13.32	3.07	1.76
Mean		75.60	0.65	2.17	5.46	1.02	1.16	11.23	1.61	1.11
Std. deviation		5.82	0.12	0.33	0.61	0.47	1.64	2.96	2.06	0.92

All results in atomic percent.

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