Appendix C

Technical Memorandum from Don Richard et al. at Barr to PolyMet Project File. Dated May 28, 2008 Regarding Attenuation of Inorganics in Groundwater at the NorthMet Mine Site

Barr Engineering Company
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Minneapolis, MN • Hibbing, MN • Duluth, MN • Ann Arbor, MI • Jefferson City, MO

Technical Memorandum

To: PolyMet Project File
From: Don Richard, Dale Kolstad, Eric Lund, Todd DeJournett
Subject: Attenuation of Inorganics in Groundwater at the NorthMet Mine Site
Date: May 28, 2008
Project: 23\69-862-006-001
c: John Borovsky

Introduction

Inorganic compounds including arsenic, nickel, copper, cobalt, antimony and vanadium may be present in seepage beneath waste rock stockpiles at the Mine Site and in West Pit groundwater during filling. Seepage and mine pit infiltration upon filling will contribute to the concentrations of these chemicals in the groundwater flowing from the Mine Site and potentially discharging to the Partridge River. Along the groundwater flow paths, the movement of inorganic compounds with the groundwater is impeded by several interactive processes with the aquifer sediment materials. The purpose of this memo is to discuss, in very general terms, the primary mechanisms for attenuation of inorganic compounds in groundwater at the NorthMet site and provide a basis for selecting inputs to the site groundwater models that can be used to evaluate future attenuation of these chemicals.

Background

Migration and attenuation of chemicals flowing with the groundwater is an extensive area of study, which is discussed in most groundwater hydrogeology texts. For dissolved chemicals, and in particular charged ionic species similar to those that are predicted to potentially occur in groundwater at the NorthMet site, three primary references on this subject include

- *Aquatic Chemistry: An Introduction Emphasizing Chemical Equilibria in Natural Waters*, by Stumm and Morgan (1981),
- *Surface Complexation Modeling, Hydrous Ferric Oxide,* by Dzombak and Morel (1990), and
- *Chemistry of the Solid Water Interface,* by Stumm (1992).

The ratio between the mass of chemical that will remain in solution and the mass that will be sorbed (both adsorbed and absorbed) is generally referred to as a sorption (or partition or distribution) coefficient, K_d. The sorption coefficient is based upon achieving equilibrium between the dissolved and sorbed mass, which is generally a reasonable assumption for relatively slow-moving groundwater. The sorption coefficient is generally linear in nature; however, this linearity occurs over the range where additional sorption sites remain available on the solid phase. Once all of the sorption sites have been filled, the linear range of the sorption coefficient is exceeded and any additional mass in solution will remain. In this case, the soil will no longer be able to attenuate the migration of the chemical mass in the groundwater relative to the rate of groundwater flow.

It is generally understood that the primary attraction of dissolved ions to solid surfaces is due to the presence of an un-equalized electrical charge that is present on the solid surfaces. The strength of this surface charge is dependent on the chemical nature of the solid material, and as implied by the title of Dzombak and Morel's work, the presence of iron oxide in the solid phase has a significant role in the adsorption of dissolved species to the solid surfaces. Moreover, in a study by Carillo and Drever (1998), the portion of iron oxide in the solid phase was the singular factor, other than pH, needed to correlate predicted and observed attenuation arsenic in groundwater. The other primary factors that affect the sorption of dissolved inorganics onto solid surfaces include pH and the amount of organic matter.

In a recent compilation of literature values for partition coefficients (U.S. EPA, 2005), the sorption coefficients for 20 inorganic compounds were evaluated. Where possible, the literature search effort also endeavored to obtain information on the pH, total organic carbon, and iron oxide content of the soils used in the individual studies. These values were then used in an equilibrium partitioning-based modeling effort to fill any data gaps in the literature-based data set. Table 1summarizes the results of the combined results from the literature search for sorption coefficients and the modeling based effort to fill data gaps in the literature based values for the 20 inorganic compounds of interest to the EPA.

Table 1

Soil Water Partition Coefficients for Inorganic Compounds (log K_d in units of L/Kg)

Inorganic	Median	Mean	Std. Dev.	Minimum	Maximum
Ag(I)	2.6	2.6	0.8	1.0	4.5
As	3.4	3.2	0.7	0.3	4.3
Ba(II)	--	2.0	0.7	0.7	3.4
Be(II)	--	2.2	1.0	1.7	4.1
Cd(II)	2.9	2.7	0.8	0.1	5.0
Co(II)	2.1	2.1	1.2	-1.2	4.1
Cr(III)	3.9	3.8	0.4	1.0	4.7
Cr(VI)	1.1	0.8	0.8	-0.7	3.3
Cu(II)	2.7	2.5	0.6	0.1	3.6
Hg(II)	3.8	3.6	0.7	2.2	5.8
MeHg	2.8	2.7	0.6	1.3	4.8
Mo(IV)	1.1	1.3	0.6	-0.4	2.7
Ni(II)	3.1	2.9	0.5	1.0	3.8
Pb(II)	4.1	3.7	1.2	0.7	5.0
Sb	--	2.3	1.1	0.1	2.7
Se(IV)	1.4	1.3	0.4	-0.3	2.4
Se(VI)	--	-0.2	1.1	-2.0	2.0
Sn(II)	--	2.7	0.7	2.1	4.0
Tl(I)	--	0.5	0.9	-1.2	1.5
V(V)	--	1.7	1.5	0.5	2.5
Zn(II)	3.1	2.7	1.0	-1.0	5.0
CN-	--	0.7	1.6	-2.4	1.3

Ref: See Table 3 of U.S. EPA, 2005

The U.S. EPA efforts to compile sorption coefficients from a wide array of published research with site specific soils provides a basis for the selection of sorption coefficients for modeling work at NorthMet, provided the characteristics of the soils at the site are similar to those included in the population used to develop the EPA data base.

Site Specific Data Collection

The published sorption coefficients identified by the U.S. EPA can be used to model attenuation of inorganics in groundwater at the NorthMet site, provided the pH, organic carbon content and iron oxide content are within the range of the available research. To compare site soils to those used in the compilation of published sorption coefficients, soil samples were collected from the site in January 2008 (see Figure 1 for sample locations). The primary purpose for this sampling effort was to complete an overburden geochemical characterization in accordance with a work plan developed for PolyMet by SRK. The soil pH was determined in the field during sample collection. Then, unconsolidated, overburden soils were collected by Barr field staff and submitted to Columbia Analytical Services (CAS) in Kelso, Washington for analysis of total iron, total organic carbon, and cation exchange capacity. Split samples were also submitted to the Iowa State University College of Engineering, Materials Analysis and Research Laboratory for analysis of iron oxide content. The laboratory results of these analyses are included in Appendix A and summarized in Table 2 (attached).

The field pH of soils ranged from a low of 5.4 in near surface samples to approximately 7.2. All samples below 10 feet and at depths that would likely be in contact with groundwater were between 6.3 and 7.2, which is in the middle of the range of pH values for partition coefficients identified by the EPA (4 to 10). Total organic carbon was highest in the shallow sample and was significantly different than the other samples from greater depths. The total organic carbon in the shallow sample was 1.73 percent while the value of the other four samples obtained below 10 feet ranged from 0.11 to 0.48 percent. These values are at the very low end of the values for organic carbon reported by the U.S. EPA, where the lower 10th percentile had an organic carbon content of 0.41 percent or less. Finally, the results of the laboratory analyses from CAS showed that the unconsolidated, overburden soils contain approximately 2 percent total iron. The results from Iowa State showed that the soil samples had a total iron oxide content of between 5 and 9 percent. This result suggests that most, if not all, of the iron present in the overburden soil is in the form of iron oxide. Similar to the carbon content, the iron oxide content of the NorthMet soils are within the range of soils used in the development of the U.S. EPA data base, but generally in the lower half of the range – for those samples where iron oxide content was available.

Estimation of Sorption Coefficients for Groundwater Attenuation Modeling

A simple comparison of the site specific field data for the NorthMet site to the ranges of these parameters in soils used in the compilation of sorption coefficients by the U.S. EPA shows that all of the parameters that have the potential to affect sorption of inorganics to soils were within the observed ranges. In the case of organic carbon, the values, especially for the deeper soil samples were generally at the lower end of the observed range. Because the site specific values for the parameters that influence sorption (pH, organic carbon, and iron oxide content) are all within the range of those identified in the U.S. EPA literature review, the published values are a reasonable data base for the selection of site specific values. Because the pH of site soils are generally below 7 and the values of organic carbon in the samples below 10 feet are near the lower 10th percentile for the published results, the values for the sorption coefficients used at the NorthMet site should generally be chosen near the lower end of the potential range.

References

Dzombak, D.A. and F.M.M Morel (1990) *Surface Complexation Modeling, Hydrous Ferric Oxide*. John Wiley & Sons, Inc. New York, N.Y.

Carillo, A and J.I Drever (1998) Adsorption of Arsenic by Natural Aquifer Material in the San Antonio-El Triunfo Mining Area. *Environmental Geology*, **4:**251-257.

Stumm, W. (1992) *Chemistry of the Solid Water Interface.* John Wiley & Sons, Inc. New York, N.Y.

Stumm, W. and J.J. Morgan (1981) *Aquatic Chemistry: An Introduction Emphasizing Chemical Equilibria in Natural Waters, 2nd Edition.* John Wiley & Sons, Inc. New York, N.Y.

U.S. Environmental Protection Agency (2005) *Partition Coefficients for Metals in Surface Water, Soils, and Waste.* EPA-600-R05-074. U.S. EPA, Office of Solid Waste and Emergency Response, Washington, D.C.

Table 2
Analytical Data Summary
Overburden Soil: Northmet Site
Polymet Mining Corporation
(units as noted)

Location **Date**	**RS-04 10-15'** **1/17/2008**	**RS-11 17-25'** **1/30/2008**	**RS-11 25-31'** **1/30/2008**	**RS-12 5.5-15'** **1/26/2008**	**RS-14B 0-5'** **1/26/2008**
Metals					
Iron, mg/kg	**19700**	**24900**	**18900**	--	--
Iron Oxide (Fe_2O_3), %	**5.77**	**7.61**	**8.80**	**5.71**	**7.19**
Measurements					
Solids, %	**84.6**	**88.5**	**88.0**	**92.4**	**83.9**
Cation Exchange Capacity, mEq/100g	**10**	**6.9**	**4.0**	**3.1**	**27**
General Parameter					
Carbon, total organic, %	**0.12**	**0.29**	**0.48**	**0.11**	**1.73**
pH, standard units, field	**6.33**	**6.56**	**6.33**	**7.17**	**5.41**

Detections are presented in **bold**.

-- Not analyzed.

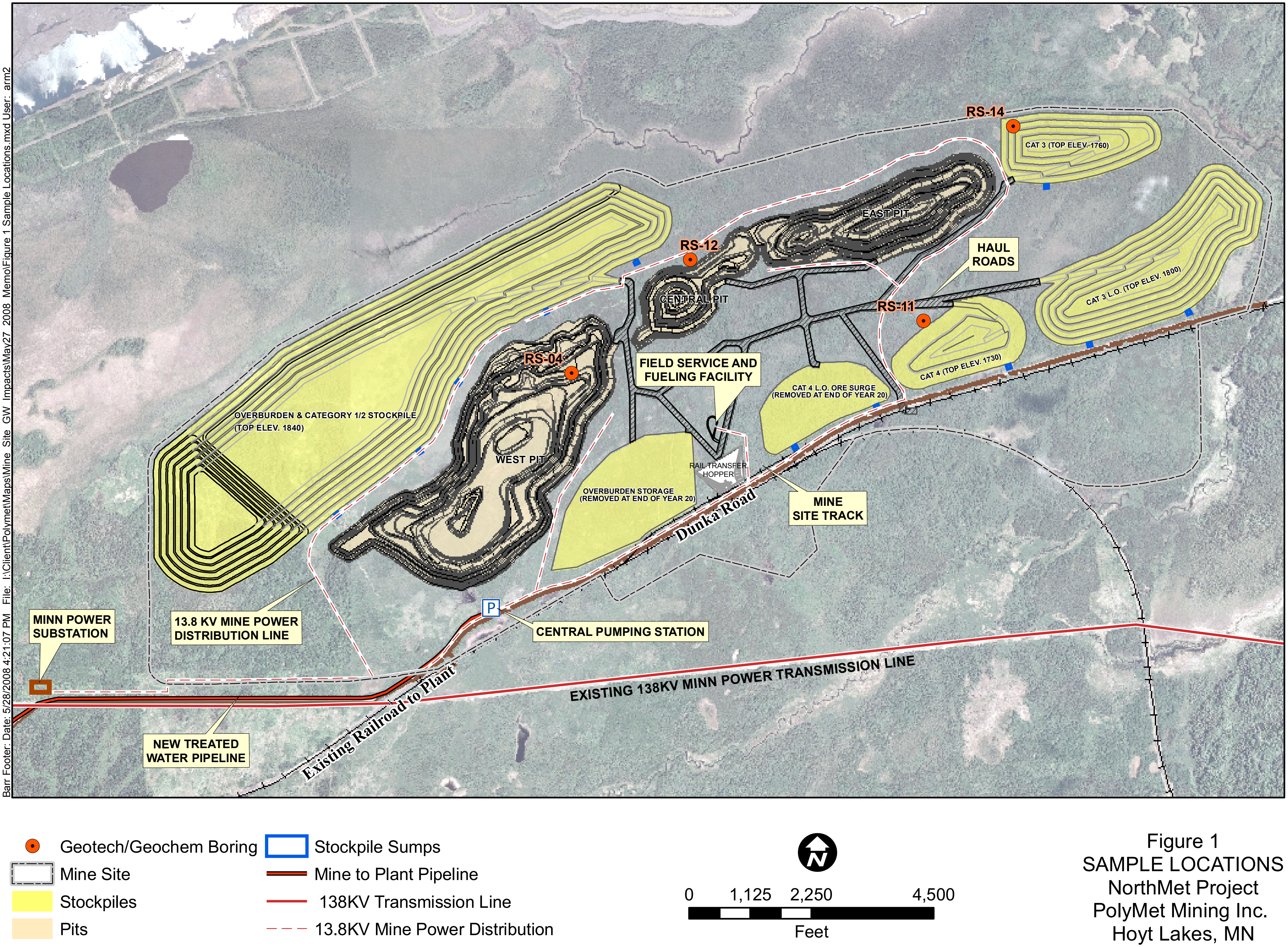

Figure 1
SAMPLE LOCATIONS
NorthMet Project
PolyMet Mining Inc.
Hoyt Lakes, MN

1317 South 13th Avenue P.O. Box 479 Kelso, Washington 98626 (360) 577-7222 (360) 636-1068 fax

Columbia Analytical Services INC.
An Employee - Owned Company

February 29, 2008 Analytical Report for Service Request No: K0801365

Marta Nelson
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

RE: PolyMet Overburden Characterization/23/69-B75INV 004

Dear Marta:

Enclosed are the results of the samples submitted to our laboratory on February 15, 2008. For your reference, these analyses have been assigned our service request number K0801365.

All analyses were performed according to our laboratory's quality assurance program. Where applicable, the methods cited conform to the Methods Update Rule (effective 4/11/2007), which relates to the use of analytical methods for the drinking water and waste water programs. The test results meet requirements of the NELAC standards. Exceptions are noted in the case narrative report where applicable. All results are intended to be considered in their entirety, and Columbia Analytical Services, Inc. (CAS) is not responsible for use of less than the complete report. Results apply only to the items submitted to the laboratory for analysis and individual items (samples) analyzed, as listed in the report.

Please call if you have any questions. My extension is 3358. You may also contact me via Email at LHuckestein@caslab.com.

Respectfully submitted,

Columbia Analytical Services, Inc.

Lynda Huckestein
Client Services Manager

LH/lb

Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons
tr	Trace level is the concentration of an analyte that is less than the PQL but greater than or equal to the MDL.

Inorganic Data Qualifiers

*	The result is an outlier. See case narrative.
#	The control limit criteria is not applicable. See case narrative.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result.
E	The result is an estimate amount because the value exceeded the instrument calibration range.
J	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
U	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.
i	The MRL/MDL has been elevated due to a matrix interference.
X	See case narrative.

Metals Data Qualifiers

#	The control limit criteria is not applicable. See case narrative.
B	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
E	The percent difference for the serial dilution was greater than 10%, indicating a possible matrix interference in the sample.
M	The duplicate injection precision was not met.
N	The Matrix Spike sample recovery is not within control limits. See case narrative.
S	The reported value was determined by the Method of Standard Additions (MSA).
U	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.
W	The post-digestion spike for furnace AA analysis is out of control limits, while sample absorbance is less than 50% of spike absorbance.
i	The MRL/MDL has been elevated due to a matrix interference.
X	See case narrative.
*	The duplicate analysis not within control limits. See case narrative.
+	The correlation coefficient for the MSA is less than 0.995.

Organic Data Qualifiers

*	The result is an outlier. See case narrative.
#	The control limit criteria is not applicable. See case narrative.
A	A tentatively identified compound, a suspected aldol-condensation product.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result.
C	The analyte was qualitatively confirmed using GC/MS techniques, pattern recognition, or by comparing to historical data.
D	The reported result is from a dilution.
E	The result is an estimate amount because the value exceeded the instrument calibration range.
J	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
N	The result is presumptive. The analyte was tentatively identified, but a confirmation analysis was not performed.
P	The GC or HPLC confirmation criteria was exceeded. The relative percent difference is greater than 40% between the two analytical results (25% for CLP Pesticides).
U	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.
i	The MRL/MDL has been elevated due to a chromatographic interference.
X	See case narrative.

Additional Petroleum Hydrocarbon Specific Qualifiers

F	The chromatographic fingerprint of the sample matches the elution pattern of the calibration standard.
L	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter molecular weight constituents than the calibration standard.
H	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of heavier molecular weight constituents than the calibration standard.
O	The chromatographic fingerprint of the sample resembles an oil, but does not match the calibration standard.
Y	The chromatographic fingerprint of the sample resembles a petroleum product eluting in approximately the correct carbon range, but the elution pattern does not match the calibration standard.
Z	The chromatographic fingerprint does not resemble a petroleum product.

Columbia Analytical Services, Inc.
Kelso, WA
State Certifications, Accreditations, and Licenses

Program	Number
Alaska DEC UST	UST-040
Arizona DHS	AZ0339
Arkansas - DEQ	88-0637
California DHS	2286
Colorado DPHE	-
Florida DOH	E87412
Hawaii DOH	-
Idaho DHW	-
Indiana DOH	C-WA-01
Louisiana DEQ	3016
Louisiana DHH	LA050010
Maine DHS	WA0035
Michigan DEQ	9949
Minnesota DOH	053-999-368
Montana DPHHS	CERT0047
Nevada DEP	WA35
New Jersey DEP	WA005
New Mexico ED	-
North Carolina DWQ	605
Oklahoma DEQ	9801
Oregon - DHS	WA200001
South Carolina DHEC	61002
Utah DOH	COLU
Washington DOE	C1203
Wisconsin DNR	998386840
Wyoming (EPA Region 8)	-

Case Narrative

COLUMBIA ANALYTICAL SERVICES, INC.

Client:	Barr Engineering Company	**Service Request No.:**	K0801365
Project:	PolyMet Overburden Characterization	**Date Received:**	2/15/2008
Sample Matrix:	Soil		

CASE NARRATIVE

All analyses were performed consistent with the quality assurance program of Columbia Analytical Services, Inc. (CAS). This report contains analytical results for samples designated for Tier II data deliverables. When appropriate to the method, method blank results have been reported with each analytical test. Additional quality control analyses reported herein include: Laboratory Duplicate (DUP), Matrix Spike (MS), and Laboratory Control Sample (LCS).

Sample Receipt

Five soil samples were received for analysis at Columbia Analytical Services on 2/15/2008. The samples were received in good condition and consistent with the accompanying chain of custody form. The samples were stored in a refrigerator at 4°C upon receipt at the laboratory.

General Chemistry Parameters

No anomalies associated with the analysis of these samples were observed.

Total Iron

No anomalies associated with the analysis of these samples were observed.

Cation Exchange Capacity

The analysis for Cation Exchange Capacity was performed at CAS in Jacksonville Florida. The analytical report is included in its entirety herein.

Approved by________________ **Date** 2/29/08

Chain of Custody Documentation

K080 1365

BARR

Chain of Custody

4700 West 77th Street
Minneapolis, MN 55435-4803
(952) 832-2600

Project Number 23/69-B75INV 004

Project Name PolyMet Overburden Characterization

№ 25407

COC 1 of 1

Project Manager: JAM2

Project Contact: MSH

Sampled by: JAM2/MMB/REE

Laboratory: CAS

	Collection		Matrix		Type			Number of Containers/Preservative: Water												Soil								
Sample Identification	Date	Time	Water	Soil	Grab	Comp.	QC	Volatile Organics (Pres.) *1	Semivolatile Organics *2	Dissolved Metals (HNO_3)	Total Metals (HNO_3)	General (Unpreserved) *3	Cyanide (NaOH)	Nutrients (H_2SO_4) *4	Oil and Grease (H_2SO_4)	Sulfide (Zn Acetate)	Methane	Bacteria ($Na_2S_2O_3$)	DRO (HCl)	VOCs (2-oz tared MeOH) *1	GRO, BTEX (2-oz tared MeOH) *1	DRO (2-oz tared) - 25 grams	Metals (2-oz unpreserved)	SVOCs (2 or 4-oz unpres.) *2	% Moisture (plastic vial, unpres.)	4 oz. unpreserved	Total No. Of Containers	Remarks:
1. RS-04 10-15'	1/17/08			x	x																					2	2	Run: Total Fe, CEC, TOC
2. RS-11 17-25	1/30/08			x	x																					2	2	"
3. RS-11 25-31	1/30/08			x	x																					2	2	"
4. RS-14B 0-5'	1/26/08			x	x																					2	2	"
5. RS-12 5.5-15'	1/26/08			x	x																					2	2	"
6.																												
7.																												Contact Marta Nelson
8.																												at Barr with questions.
9.																												1-952-832-2702
10.																												
11.																												
12.																												

Relinquished By:	On Ice?	Date	Time	Received by:	Date	Time
	[✓ Y] N	2/14/08	1200	CAS	2/15/08	0900
	Y N					

Samples Shipped VIA: ☐ Air Freight ☒ Federal Express ☐ Sampler ☐ Other ______

Air Bill Number: 7914 9970 553B

Common Parameter/Container - Preservation Key
*1 - Volatile Organics = BTEX, GRO, TPH, Full List
*2 - Semivolatile Organics = PAHs, PCP, Dioxins, Full List, Herbicide/Pesticide/PCBs
*3 - General = pH, Chloride, Flouride, Alkalinity, TSS, TDS, TS, Sulfate
*4 - Nutrients = COD, TOC, Phenols, Ammonia Nitrogen, TKN

Distribution: White-Original Accompanies Shipment to Lab; Yellow - Field Copy; Pink - Lab Coordinator

PC LH

Columbia Analytical Services, Inc.
Cooler Receipt and Preservation Form

Client / Project: BARR Service Request K08 01365

Received: 2-15-08 Opened: 2-15-08 By:

1. Samples were received via? *US Mail* [✓ *Fed Ex*] *UPS* *DHL* *GH* *GS* *PDX* *Courier* *Hand Delivered*
2. Samples were received in: (circle) [✓ *Cooler*] *Box* *Envelope* *Other* ______ *NA*
3. Were custody seals on coolers? NA Y [✓ N] If yes, how many and where? ______
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N
4. Is shipper's air-bill filed? If not, record air-bill number: ______ NA [✓ Y] N
5. **Temperature of cooler(s) upon receipt (°C):** -0.4
   Temperature Blank (°C): —
6. If applicable, list Chain of Custody Numbers: ______
7. Were custody papers properly filled out (ink, signed, etc.)? NA [✓ Y] N
8. Packing material used. *Inserts* *Bubble Wrap* *Gel Packs* [✓ *Wet Ice*] *Sleeves* *Other* ______
9. **Did all bottles arrive in good condition (unbroken)?** *Indicate in the table below.* NA [✓ Y] N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? [✓ Y] N
11. Did all sample labels and tags agree with custody papers? *Indicate in the table below* [✓ Y] N
12. **Were the correct types of bottles used for the tests indicated?** NA [✓ Y] N
13. Were all of the preserved bottles received at the lab with the appropriate pH? *Indicate in the table below* [✓ NA] Y N
14. Were VOA vials and 1631 Mercury bottles checked for absence of air bubbles? *Indicate in the table below.* [✓ NA] Y N
15. **Are CWA Microbiology samples received with >1/2 the 24hr. hold time remaining from collection?** [✓ NA] Y N
16. Was C12/Res negative? [✓ NA] Y N

Sample ID on Bottle	Sample ID on COC	Sample ID on Bottle	Sample ID on COC

Sample ID	Bottle Count	Bottle Type	Out of Temp	Head-space	Broken	pH	Reagent	Volume added	Reagent Lot Number	Initials

Additional Notes, Discrepancies, & Resolutions: ______

Page 1 of: 1 2

Total Solids

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Results

lient:	Barr Engineering Company	**Service Request:**	K0801365
roject:	PolyMet Overburden C/23/69-B75INV 004		
ample Matrix:	Soil		

Total Solids

rep Method:	NONE	**Units:**	PERCENT
nalysis Method:	160.3M	**Basis:**	Wet
est Notes:			

ample Name	**Lab Code**	**Date Collected**	**Date Received**	**Date Analyzed**	**Result**	**Result Notes**
S-04 10-15'	K0801365-001	01/17/2008	02/15/2008	02/21/2008	84.6	
S-11 17-25	K0801365-002	01/30/2008	02/15/2008	02/21/2008	88.5	
S-11 25-31	K0801365-003	01/30/2008	02/15/2008	02/21/2008	88.0	
S-14B 0.5'	K0801365-004	01/26/2008	02/15/2008	02/21/2008	83.9	
S-12 5.5-15'	K0801365-005	01/26/2008	02/15/2008	02/21/2008	92.4	

lient:	Barr Engineering Company	**Service Request:**	K0801365
roject:	PolyMet Overburden C/23/69-B75INV 004	**Date Collected:**	01/17/2008
ample Matrix:	Soil	**Date Received:**	02/15/2008
		Date Analyzed:	02/21/2008

Duplicate Sample Summary
Total Solids

rep Method:	NONE	**Units:**	PERCENT
nalysis Method:	160.3M	**Basis:**	Wet
est Notes:			

ample Name	**Lab Code**	**Sample Result**	**Duplicate Sample Result**	**Average**	**Relative Percent Difference**	**Result Notes**
S-04 10-15'	K0801365-001	84.6	86.4	85.5	2	

General Chemistry Parameters

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client :	Barr Engineering Company	**Service Request :**	K0801365
Project Name :	PolyMet Overburden Characterization	**Date Collected :**	01/17-30/08
Project Number :	23/69-B75INV 004	**Date Received :**	02/15/08
Sample Matrix :	SOIL		

Carbon, Total Organic

Prep Method :	Method	Units :	Percent
Analysis Method :	9060M	Basis :	Dry
Test Notes :			

Sample Name	Lab Code	MRL	Dilution Factor	Date Prepared	Date Analyzed	Result	Result Notes
RS-04 10-15'	K0801365-001	0.05	1	2/19/2008	02/25/08	0.12	
RS-11 17-25	K0801365-002	0.05	1	2/19/2008	02/25/08	0.29	
RS-11 25-31	K0801365-003	0.05	1	2/19/2008	02/25/08	0.48	
RS-14B 0.5'	K0801365-004	0.05	1	2/19/2008	02/25/08	1.73	
RS-12 5.5-15'	K0801365-005	0.05	1	2/19/2008	02/25/08	0.11	
Method Blank	K0801365-MB	0.05	1	2/19/2008	02/25/08	ND	

M Modified

Report By: CSKILLERN

Client :	Barr Engineering Company	**Service Request :**	K0801365
Project Name :	PolyMet Overburden Characterization	**Date Collected :**	NA
Project Number :	23/69-B75INV 004	**Date Received :**	NA
Sample Matrix :	SOIL	**Date Prepared :**	02/19/08
		Date Analyzed :	02/25/08

Duplicate Summary
Inorganic Parameters

Sample Name : Batch QC
Lab Code : K0801204-001DUP
Test Notes :

Units : Percent
Basis : Dry

Analyte	**Prep Method**	**Analysis Method**	**MRL**	**Sample Result**	**Duplicate Sample Result**	**Average**	**Relative Percent Difference**	**Result Notes**
Carbon, Total Organic	Method	9060M	0.05	0.60	0.59	0.60	2	

M Modified

COLUMBIA ANALYTICAL SERVICES, INC.

QA/QC Report

Client :	Barr Engineering Company	**Service Request :**	K0801365
Project Name :	PolyMet Overburden Characterization	**Date Collected :**	NA
Project Number :	23/69-B75INV 004	**Date Received :**	NA
Sample Matrix :	SOIL	**Date Prepared :**	02/19/08
		Date Analyzed :	02/25/08

Matrix Spike Summary
Inorganic Parameters

Sample Name :	Batch QC	Units :	Percent
Lab Code :	K0801204-001MS	Basis :	Dry
Test Notes :			

Analyte	**Prep Method**	**Analysis Method**	**MRL**	**Spike Level**	**Sample Result**	**Spiked Sample Result**	**Percent Recovery**	**CAS Percent Recovery Acceptance Limits**	**Result Notes**
Carbon, Total Organic	Method	9060M	0.05	2.52	0.60	2.74	85	75-114	

M Modified

Client :	Barr Engineering Company	**Service Request :**	K0801365
Project Name :	PolyMet Overburden Characterization	**Date Collected :**	NA
Project Number :	23/69-B75INV 004	**Date Received :**	NA
Sample Matrix :	SOIL	**Date Prepared :**	02/19/08
		Date Analyzed :	02/25/08

Laboratory Control Sample Summary
Inorganic Parameters

Sample Name :	Laboratory Control Sample	Units :	Percent
Lab Code :	K0801365-LCS	Basis :	Dry
Test Notes :			

Analyte	**Prep Method**	**Analysis Method**	**True Value**	**Result**	**Percent Recovery**	**CAS Percent Recovery Acceptance Limits**	**Result Notes**
Carbon, Total Organic	Method	9060M	0.89	0.86	97	74-123	

M Modified

COLUMBIA ANALYTICAL SERVICES, INC.

QA/QC Report

Client : Barr Engineering Company
Project : PolyMet Overburden Characterization

Service Request : K0801365
Date Collected : NA
Date Received : NA

Carbon, Total Organic
9060M
Units: Percent

CONTINUING CALIBRATION VERIFICATION (CCV)

	Date Analyzed	True Value	Measured Value	Percent Recovery
CCV1 Result	2/25/2008	20.0	18.3	92
CCV2 Result	2/25/2008	20.0	18.5	93
CCV3 Result	2/25/2008	20.0	18.8	94

COLUMBIA ANALYTICAL SERVICES, INC.

QA/QC Report

Client : Barr Engineering Company
Project : PolyMet Overburden Characterization

Service Request : K0801365
Date Collected : NA
Date Received : NA

Carbon, Total Organic
9060M
Units: Percent

CONTINUING CALIBRATION BLANK (CCB)

	Date Analyzed	MRL	Blank Value
CCB1 Result	2/25/2008	0.05	ND
CCB2 Result	2/25/2008	0.05	ND
CCB3 Result	2/25/2008	0.05	ND

Metals

Columbia Analytical Services

- Cover Page -
INORGANIC ANALYSIS DATA PACKAGE

Client: Barr Engineering Company

Project Name: PolyMet Overburden Characterization

Project No.: 23/69-B75INV 004

Service Request: K0801365

Sample Name:	Lab Code:
RS-04 10-15'	K0801365-001
RS-04 10-15'D	K0801365-001D
RS-04 10-15'S	K0801365-001S
RS-11 17-25	K0801365-002
RS-11 25-31	K0801365-003
Method Blank	K0801365-MB

Comments:

Approved By: EmA **Date:** 2/29/08

Metals

- 1 -

INORGANIC ANALYSIS DATA PACKAGE

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project No.:	23/69-B75INV 004	**Date Collected:**	1/17/08
Project Name:	PolyMet Overburden Characterizat	**Date Received:**	2/15/08
Matrix:	SOIL	**Units:**	mg/Kg
		Basis:	DRY

Sample Name: RS-04 10-15' **Lab Code:** K0801365-001

Analyte	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	C	Q
Iron	6010B	4.5	2.0	02/19/08	02/20/08	19700		

% Solids: 84.6

Comments:

Metals

- 1 -

INORGANIC ANALYSIS DATA PACKAGE

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project No.:	23/69-B75INV 004	**Date Collected:**	1/30/08
Project Name:	PolyMet Overburden Characterizat	**Date Received:**	2/15/08
Matrix:	SOIL	**Units:**	mg/Kg
		Basis:	DRY

Sample Name: RS-11 17-25

Lab Code: K0801365-002

Analyte	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	C	Q
Iron	6010B	4.4	2.0	02/19/08	02/20/08	24900		

% Solids: 88.5

Comments:

Metals

- 1 -
INORGANIC ANALYSIS DATA PACKAGE

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project No.:	23/69-B75INV 004	**Date Collected:**	1/30/08
Project Name:	PolyMet Overburden Characterizat	**Date Received:**	2/15/08
Matrix:	SOIL	**Units:**	mg/Kg
		Basis:	DRY

Sample Name: RS-11 25-31

Lab Code: K0801365-003

Analyte	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	C	Q
Iron	6010B	4.4	2.0	02/19/08	02/20/08	18900		

% Solids: 88.0

Comments:

Metals

- 1 -

INORGANIC ANALYSIS DATA PACKAGE

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project No.:	23/69-B75INV 004	**Date Collected:**	
Project Name:	PolyMet Overburden Characterizat	**Date Received:**	
Matrix:	SOIL	**Units:**	mg/Kg
		Basis:	DRY

Sample Name: Method Blank

Lab Code: K0801365-MB

Analyte	Analysis Method	MRL	Dilution Factor	Date Extracted	Date Analyzed	Result	C	Q
Iron	6010B	2.0	1.0	02/19/08	02/20/08	2.0	U	

% Solids: 100.0

Comments:

Metals

- 5A -

SPIKE SAMPLE RECOVERY

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project No.:	23/69-B75INV 004	**Units:**	MG/KG
Project Name:	PolyMet Overburden Characterizat	**Basis:**	DRY
Matrix:	SOIL	**% Solids:**	84.6

Sample Name: RS-04 10-15'S **Lab Code:** K0801365-001S

Analyte	Control Limit %R	Spike Result	C	Sample Result	C	Spike Added	%R	Q	Method
Iron		22000		19700		236.41	972.9		6010B

An empty field in the Control Limit column indicates the control limit is not applicable

Metals

- 6 -

DUPLICATES

Client: Barr Engineering Company

Service Request: K0801365

Project No.: 23/69-B75INV 004

Units: MG/KG

Project Name: PolyMet Overburden Characterizat

Basis: DRY

Matrix: SOIL

% Solids: 84.6

Sample Name: RS-04 10-15'D **Lab Code:** K0801365-001D

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	Method
Iron	30	19700		20500		4		6010B

An empty field in the Control Limit column indicates the control limit is not applicable.

Metals

-7-

LABORATORY CONTROL SAMPLE

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project No.:	23/69-B75INV 004		
Project Name:	PolyMet Overburden Characterizat		

Aqueous LCS Source: **Solid LCS Source:** ERA D045540

Analyte	Aqueous (ug/L) True	Aqueous (ug/L) Found	Aqueous (ug/L) %R	Solid (mg/kg) True	Solid (mg/kg) Found	Solid (mg/kg) C	Solid (mg/kg) Limits		Solid (mg/kg) %R
Iron				13900	15900		64	154	114.4

9143 Philips Highway, Suite 200 Jacksonville, FL 32256 (904) 739-2277 (904) 739-2011 fax

Columbia Analytical Services INC.
An Employee - Owned Company

March 6, 2008 Service Request No: K0801365

Marta Nelson
Barr Engineering
4700 West 77th Street
Minneapolis, MN 55435

Re: PolyMet Overburden Characterization/23/69-B75INV 004

Dear Marta:

Enclosed are the results of the sample(s) submitted to our laboratory on February 15, 2008. For your reference, these analyses have been assigned our service request number **K0801365**.

All analyses were performed according to our laboratory's quality assurance program. The test results meet requirements of the NELAP standards except as noted in the case narrative report. All results are intended to be considered in their entirety, and Columbia Analytical Services, Inc. (CAS) is not responsible for use of less than the complete report. Results apply only to the items submitted to the laboratory for analysis and individual items (samples) analyzed, as listed in the report. In accordance to the NELAC 2003 Standard, a statement on the estimated uncertainty of measurement of any quantitative analysis will be supplied upon request.

Please call if you have any questions. My extension is 4408. You may also contact me via email at Tkissinger@caslab.com.

Respectfully submitted,

Columbia Analytical Services, Inc.

Tom Kissinger

Tom Kissinger
Project Manager

Page 1 of 11

Laboratory Manager: Greg Jordan
Quality Assurance Officer: Kathy Brungard

CAS Jacksonville is NELAC-accredited by the State of Florida, #E82502 valid through 6/30/08. Other state accreditations include: Arkansas, #88-0600 valid through 1/12/06; Georgia, #958 valid through 6/30/08; Louisiana, #02086 valid through 6/30/08; Texas, #T104704197-06-TX valid through 5/31/08; North Carolina, #527 valid through 12/31/07; South Carolina, #96021001 valid through 6/30/07.

COLUMBIA ANALYTICAL SERVICES, INC.

Client:	Barr Engineering	**Service Request No.:**	K0801365
Project:	PolyMet Overburden Characterization	**Date Received:**	2/21/08
Sample Matrix:	soil		

CASE NARRATIVE

All analyses were performed consistent with the quality assurance program of Columbia Analytical Services, Inc. (CAS). This report contains analytical results for samples designated for Tier II data deliverables, including results of QC samples analyzed from this delivery group. When appropriate to the procedure, method blank results have been reported with each analytical test. Analytical procedures performed by the lab are validated in accordance with NELAC standards. Parameters that are included in the NELAC Fields of Testing but are not included in the lab's NELAC accreditation are identified in the discussion of each analytical procedure.

Sample Receipt

5 soil samples were received for analysis at Columbia Analytical Services on 2/21/08. The samples were received in good condition and consistent with the accompanying chain of custody form. Samples are refrigerated at 4±2°C upon receipt at the lab.

CEC Metals by ICP-OES

Elevated Method Reporting Limits

All samples were analyzed with a 10 fold dilution due to sample matrix and Sodium concentration.

Approved by __________ Date 3/6/08

Data Qualifiers

Inorganic Data

*	The result is an outlier. See case narrative.
#	The control limit criteria are not applicable. See case narrative.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result.
E	The result is an estimated amount because the value exceeded the instrument calibration range.
J	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
U	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.
Z	Too many colonies were present (TNTC). The numeric value represents the filtration volume.
i	The MRL/MDL has been elevated due to matrix interference.
X	See case narrative.

Metals Data

*	The result is an outlier. See case narrative.
#	The control limit criteria are not applicable. See case narrative.
B	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
E	The reported value is estimated because of the presence of matrix interference.
M	The duplicate injection precision was not met.
N	The Matrix Spike sample recovery is not within control limits. See case narrative.
S	The result was determined by Method of Standard Additions (MSA).
U	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.
W	The post-digestion spike for furnace AA analysis is out of control limits, while sample absorbance is less than 50% of spike absorbance.
i	The MRL/MDL has been elevated due to matrix interference.
X	See case narrative.
+	The correlation coefficient for the MSA is less than 0.995.

Organic Data

*	The result is an outlier. See case narrative.
#	The control limit criteria are not applicable. See case narrative.
A	The tentatively identified compound is a suspected aldol-condensation product.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result.
C	The analyte was qualitatively confirmed using GC/MS techniques, pattern recognition, or by comparing to historical data.
D	The reported result is from a dilution.
E	The result is an estimated amount because the value exceeded the instrument calibration range.
J	The result is an estimated concentration that is less than the MRL but greater than or equal to the MDL.
N	The result is presumptive. The analyte was tentatively identified, but a confirmation analysis was not performed.
P	The GC or HPLC confirmation criteria were exceeded. The relative percent difference is greater than 40% between the two analytical results (25% for CLP Pesticides)
U	The compound was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL.
i	The MRL/MDL has been elevated due to a chromatographic interference.
X	See case narrative.

Petroleum Hydrocarbon Specific

F	The chromatographic fingerprint of the sample matches the elution pattern of the calibration standard.
L	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter molecular weight constituents than the calibration standard.
H	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of heavier molecular weight constituents than the calibration standard.
O	The chromatographic fingerprint of the sample resembles an oil, but does not match the calibration standard.
Y	The chromatographic fingerprint of the sample resembles a petroleum product eluting in approximately the correct carbon range, but the elution pattern does not match the calibration standard.
Z	The chromatographic fingerprint does not resemble a petroleum product.

Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons
tr	Trace level is the concentration of an analyte that is less than the PQL but greater than or equal to the MDL.

Client: Barr Engineering
Project: PolyMet Overburden Characterization/23/69-B75INV 004

Service Request: K0801365

SAMPLE CROSS-REFERENCE

SAMPLE #	CLIENT SAMPLE ID	DATE	TIME
K0801365-001	RS-04 10-15'	01/17/08	
K0801365-002	RS-11 17-25	01/30/08	
K0801365-003	RS-11 25-31	01/30/08	
K0801365-004	RS-14B 0.5'	01/26/08	
K0801365-005	RS-12 5.5-15'	01/26/08	

COLUMBIA ANALYTICAL SERVICES, INC.

Analytical Report

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project:	PolyMet Overburden Characterization/23/69-B75INV 004	**Date Collected:**	
Sample Matrix:	Soil	**Date Received:**	2/15/2008
		Date Extracted:	2/28/2008
		Date Analyzed:	3/3/2008

Cation Exchange Capacity
EPA Method 9081
Units: mEq/100g
Dry Weight Basis

Sample Name	**Lab Code**	**MRL**	**Result**
RS-04 10-15'	K0801365-001	0.4	10
RS-11 17-25	K0801365-002	0.4	6.9
RS-11 25-31	K0801365-003	0.4	4.0
RS-14B 0.5'	K0801365-004	0.4	27
RS-12 5.5-15'	K0801365-005	0.4	3.1
Method Blank	K080228-MB	0.04	U

COLUMBIA ANALYTICAL SERVICES, INC.

QA/QC Report

Client: Barr Engineering Company
Project: PolyMet Overburden Characterization/23/69-B751NV 004
Sample Matrix: Soil

Service Request: K0801365
Date Collected: NA
Date Received: NA
Date Extracted: NA
Date Analyzed: 2/28/2008

Duplicate Summary
Inorganic Parameters

Sample Name: RS-04 10-15'
Lab Code: K0801365-001

Analyte	**Units**	**EPA Method**	**MRL**	**Sample Result**	**Duplicate Sample Result**	**Average**	**Relative Percent Difference**
Sodium	mEQ/100g	6010B	0.4	10.3	8.81	9.6	16

QA/QC Report

Client:	Barr Engineering Company	**Service Request:**	K0801365
Project:	PolyMet Overburden Characterization/23/69-B751NV 004	**Date Collected:**	NA
Sample Matrix:	Water	**Date Received:**	NA
		Date Analyzed:	2/28/2008

Laboratory Control Sample Summary
Inorganic Parameters

Analyte	Units	EPA Method	TRUE Value	Result	Percent Recovery	CAS Percent Recovery Acceptance Limits
Sodium	mg/L	6010B	10	9.91	99	80-120

Columbia Analytical Services, Inc.
Cooler Receipt and Preservation Form

Client: Barr Engineering Service Request # K0801365

Project: PolyMet Overburden Characterization

Cooler received on 2/21/08 and opened on 2/21/08 by TDK

COURIER: CAS [✓ UPS] FEDEX DHL CLIENT Tracking #

1	Were custody seals on outside of cooler?	[✓ Yes]	No	N/A
2	Were seals intact, signed and dated?	[✓ Yes]	No	N/A
3	Were custody papers properly filled out?	[✓ Yes]	No	N/A
4	Temperature of cooler(s) upon receipt (Should be 4 +/- 2 degrees C) 3.7			
5	Correct Temperature?	[✓ Yes]	No	N/A
6	Were Ice or Ice Packs present	[✓ Yes]	No	N/A
7	Did all bottles arrive in good condition (unbroken, etc....)?	[✓ Yes]	No	N/A
8	Were all bottle labels complete (sample ID, preservation, etc....)?	[✓ Yes]	No	N/A
9	Did all bottle labels and tags agree with custody papers?	[✓ Yes]	No	N/A
10	Were the correct bottles used for the tests indicated?	[✓ Yes]	No	N/A
11	Were all of the preserved bottles received with the appropriate preservative?	[✓ Yes]	No	N/A

HNO3 pH<2 H2SO4 pH<2 ZnAc2/NaOH pH>9 NaOH pH>12 HCl pH<2

Preservative additions noted below

12	Were all samples received within analysis holding times?	[✓ Yes]	No	N/A
13	Were VOA vials checked for absence of air bubbles? If present, note below	Yes	No	[✓ N/A]
14	Where did the bottles originate?	[✓ CAS]	Client	

Sample ID	Reagent	Manuf. Lot # or CAS Chem ID	ml added	Initials

Additional comments and/or explanation of all discrepancies noted above:

Client approval to run samples if discrepancies noted: Date: 9

SR # : K0801365　　Date: 2/21/08　　Initials: TDK

Note that pH is checked and meets the required pH criterion listed in the column heading unless otherwise noted on cooler receipt form.

	Bottle Code																													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30
	40mL	40mL	40mL	40mL	125mL	125mL	125mL	125mL	250mL	250mL	250mL	250mL	250mL	250mL	250mL	500mL	500mL	500mL	1L	1L	1L	1L	1L	2oz	4oz	8oz	16oz	5g	100mL	Misc.
Container	G	G	G	G	P	P	P	P	P	P	P	P	P	G	G	P	P	P	P	P	G	G	G	G	G	G	G	ENC	P	Misc.
Pres.		HCl	Sodium Thiosulfate	H2SO4		HCl	H2SO4	HNO3		H2SO4	HNO3	ZnAcetate NaOH	NaOH		HNO3		H2SO4	HNO3		HNO3		HCl	H2SO4						Sodium Thiosulfate	
Req. pH	N/A	<2	N/A	<2	N/A	<2	<2	<2	N/A	<2	<2	>9	>12	N/A	<2	N/A	<2	<2	N/A	<2	N/A	<2	<2	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Sample #	-	-	-	-	-	-	-	-	-		-	-	-	-	-	-	-	-	-	-	-		-	-	-				-	-
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-038																														
-039																														
-040																														

Intra-Network Chain of Custody

1317 South 13th Avenue • Kelso, WA 98626 • 360-577-7222 • FAX 360-636-1068

CAS Contact: Lynda Huckestein

11

Project Name: PolyMet Overburden Characterization
Project Number: 23/69-B75INV 004
Project Manager: Marta Nelson
Company: Barr Engineering

Lab Code	Client Sample ID	# of Cont.	Matrix	Sample Date	Sample Time	Date Received	Send To	CEC 6010B
K0801365-001	RS-04 10-15'	1	Soil	01/17/08		02/15/08	JAX	II V
K0801365-002	RS-11 17-25	1	Soil	01/30/08		02/15/08	JAX	V 2/19/08
K0801365-003	RS-11 25-31	1	Soil	01/30/08		02/15/08	JAX	V
K0801365-004	RS-14B 0.5'	1	Soil	01/26/08		02/15/08	JAX	V
K0801365-005	RS-12 5.5-15'	1	Soil	01/26/08		02/15/08	JAX	V

Folder Comments:
HOLD ALL SAMPLES FOR 6 MONTHS. CHECK WITH PC

Special Instructions/Comments

Turnaround Requirements

___ RUSH (Surcharges Apply)

PLEASE CIRCLE WORK DAYS

1 2 3 4 5

✓ STANDARD

Requested FAX Date: ________

Requested Report Date: 03/03/08

Report Requirements

___ I. Results Only

✓ II. Results + QC Summaries

___ III. Results + QC and Calibration Summaries

___ IV. Data Validation Report with Raw Data

PQL/MDL/J N

EDD Y

Invoice Information

PO# K0801365

Bill to

Relinquished By: 2/20/08 0900 CAS/Kelso

Received By: 2-21-08 14:20 CAS/JAX

Airbill Number:

IOWA STATE UNIVERSITY
OF SCIENCE AND TECHNOLOGY

College of Engineering
Materials Analysis and Research Laboratory
23 Town Engineering
Ames, Iowa 50011-3232
515 294-8752 or 8761
FAX 515 294-4563
www.marl.iastate.edu

Report of X-ray Analysis

23/69-862004014

Client: Marta Nelson, Barr Engineering

Samples: 5 overburden samples **Date Received: 02/15/2008**

Contract No.: 1000 **Report Date:** 03/07/2008

Instrument set up: PHILIPS PW 2404 X-ray spectrometer **(XRF)**
Rh X-ray tube operated at 3600 watts
Fused disk standards, NIST grade
Analyzed in vacuum mode
SIEMENS D 500 X-ray diffractometer (**XRD**)
Cu X-ray tube operated at 50kV and 27mA
Medium resolution slits

Sample Preparation: The samples arrived at the lab in a wet condition. The samples had been packed in ice for transport to ISU; however, the ice had melted by the time the samples were delivered. The samples were sealed in jars and plastic bags so it was assumed that the moisture had been present in the samples prior to shipping. The samples were allowed to dry at laboratory temperature and relative humidity until they could be processed for X-ray analysis. The air-dry samples were homogenized and then a 100-gram sub sample was taken from each bulk sample. The sub samples were then ground to a fine particle size using a SPEX shatterbox.

X-ray diffraction specimens were back-packed to avoid preferred orientation. X-ray fluorescence specimens were prepared by igniting the samples to a constant mass at 940°C and then fusing them into glass disks.

Results: The results of the studies are summarized below.
The samples were received at the lab in a wet condition (bulk moisture contents ranged from 8 to 20%). The bulk moisture content, air-dry moisture content and overall appearance are summarized in Table 1. All moisture content determinations were conducted at 105 to 110°C.

The chemical assays obtained via XRF are summarized in Table 2. Test results are expressed on an oven-dry basis. The iron content of the samples varied from about 5 to 9%.

The results of XRD analysis are shown in Figures 1 through 8. The minerals identified in the diffractograms are listed at the bottom of each figure. It is important to note that many of the minerals that were identified exhibit a wide range of solid solution and the PDF database entry may only provide a general ID of the phase. Typically the ID would allow for the identification of the mineral group that the phase belongs to.

Comments: The majority of the samples appeared to be primarily composed of sand with some gravel particles; however, the samples denoted as RS-04 and RS-14B also contained finer-sized particles.

The mineralogy of the bulk samples appeared to be roughly similar. Major components tended to be α-quartz and feldspar minerals (both plagioclase and alkali feldspars). Minor components appeared to be amphibole minerals, spinel minerals (magnetic fractions had diffractograms close to magnetite, see Figure 7) and some clay minerals (the 10-angstrom peak suggests the presence of illite (or mica), the 14-angstrom peak suggests the presence of chlorite or a smectite mineral, and the 7-angstrom peak could be kaolinite). The identification of the clay mineral fraction could be significantly enhanced via particle size separation techniques and oriented aggregate XRD specimens.

Manual searches were conducted to inspect for the presence of goethite, lepidocrocite, hematite and magnetite. These four minerals are overlaid on a composite diffractogram for all five of the samples (see Figure 8). The only probable match that was obtained was for magnetite. The other three iron minerals failed to match the strongest lines for the various phases given in the PDF database. This does not mean that the minerals are absent from the samples, it simply means that they were below the detection limit of the XRD method. However, many of the amphibole minerals and some of the clay minerals (especially chlorite) can contain significant amounts of iron (both in divalent and trivalent oxidation states); and hence, the iron may simply be concentrated into those minerals (plus the spinel minerals (e.g., magnetite) as noted above).

Approved: ____________ Date: 3/10/2008

Table 1. Bulk properties of the samples.

Sample	Moisture, % (as received)	Moisture, % (air-dry)	LOI, % (at 940°C)	Visual after drying
RS-04 (10-15')	12.6	0.30	1.23	gray color
RS-11 (17-25')	12.3	0.29	1.39	gray-black color
RS-11 (25-31')	12.6	0.22	1.37	gray-black color
RS-12 (5.5-15')	8.2	0.24	0.87	tan-brown color
RS-14B (0-5')	20.0	1.73	5.80	brown color

Table 2. Results of XRF analysis of the samples (dry basis).

Element expressed as an Oxide	RS-04 (10-15')	RS-11 (17-25')	RS-11 (25-31')	RS-12 (5.5-15')	RS-14B (0-5')
Na_2O	3.45	3.17	2.92	3.96	2.70
MgO	2.05	2.90	2.91	1.46	2.03
Al_2O_3	13.7	15.9	14.8	13.6	14.9
SiO_2	65.9	61.3	60.8	67.3	60.8
P_2O_5	0.17	0.15	0.16	0.15	0.13
SO_3	0.20	0.05	0.04	0.03	0.01
K_2O	2.50	2.85	2.81	2.47	2.06
CaO	3.10	3.04	3.02	2.82	2.80
TiO_2	0.55	0.59	0.68	0.42	0.87
Fe_2O_3 (total)	5.77	7.61	8.80	5.71	7.19
SrO	0.05	0.05	0.05	0.07	0.04
Mn_2O_3	0.09	0.10	0.16	0.10	0.12
BaO	0.05	0.07	0.06	0.06	0.05
LOI (at 940°C)	1.23	1.39	1.37	0.87	5.80

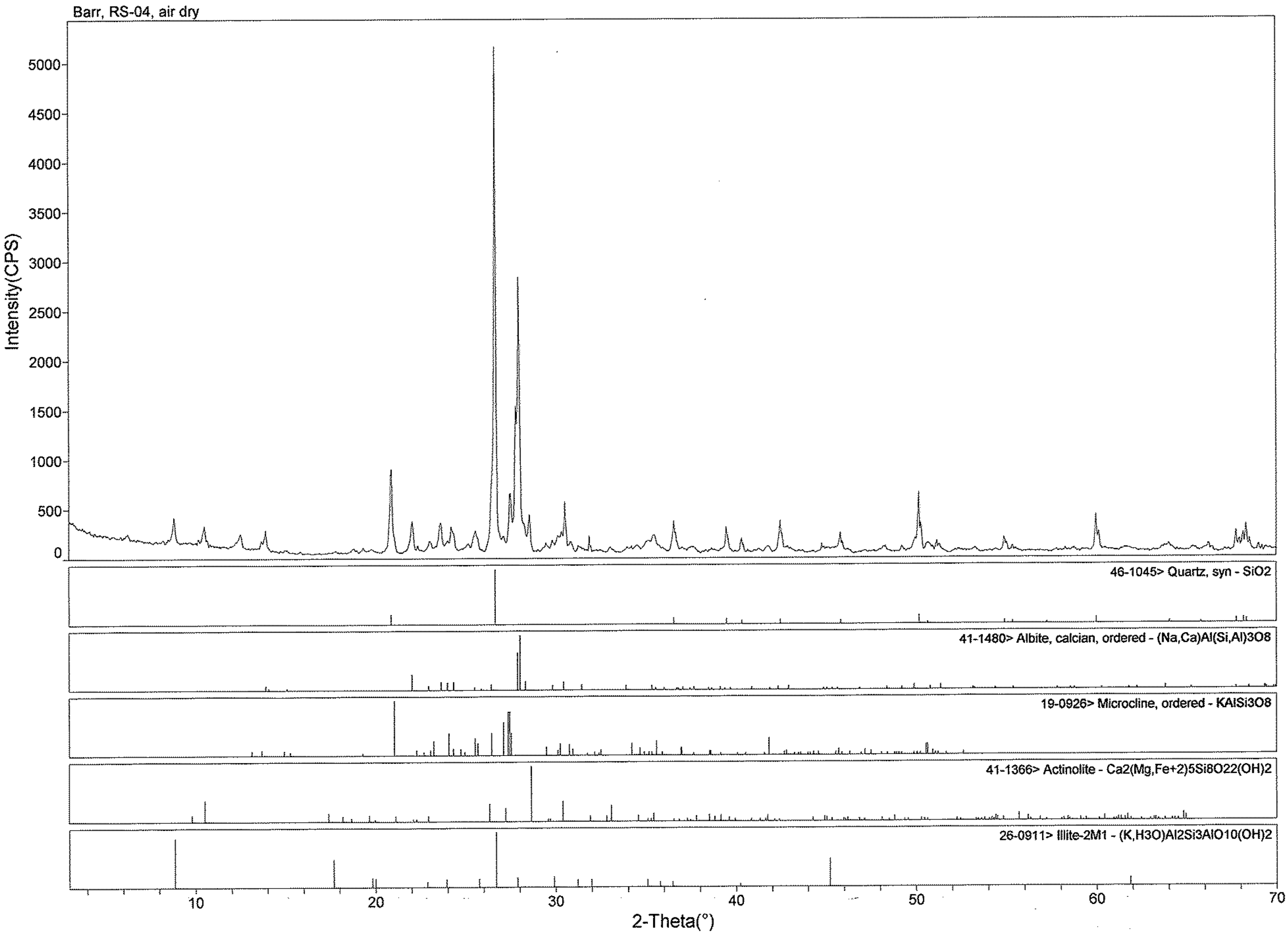

Figure 1. X-ray diffractogram of RS-04, 10-15′ (air-dry).

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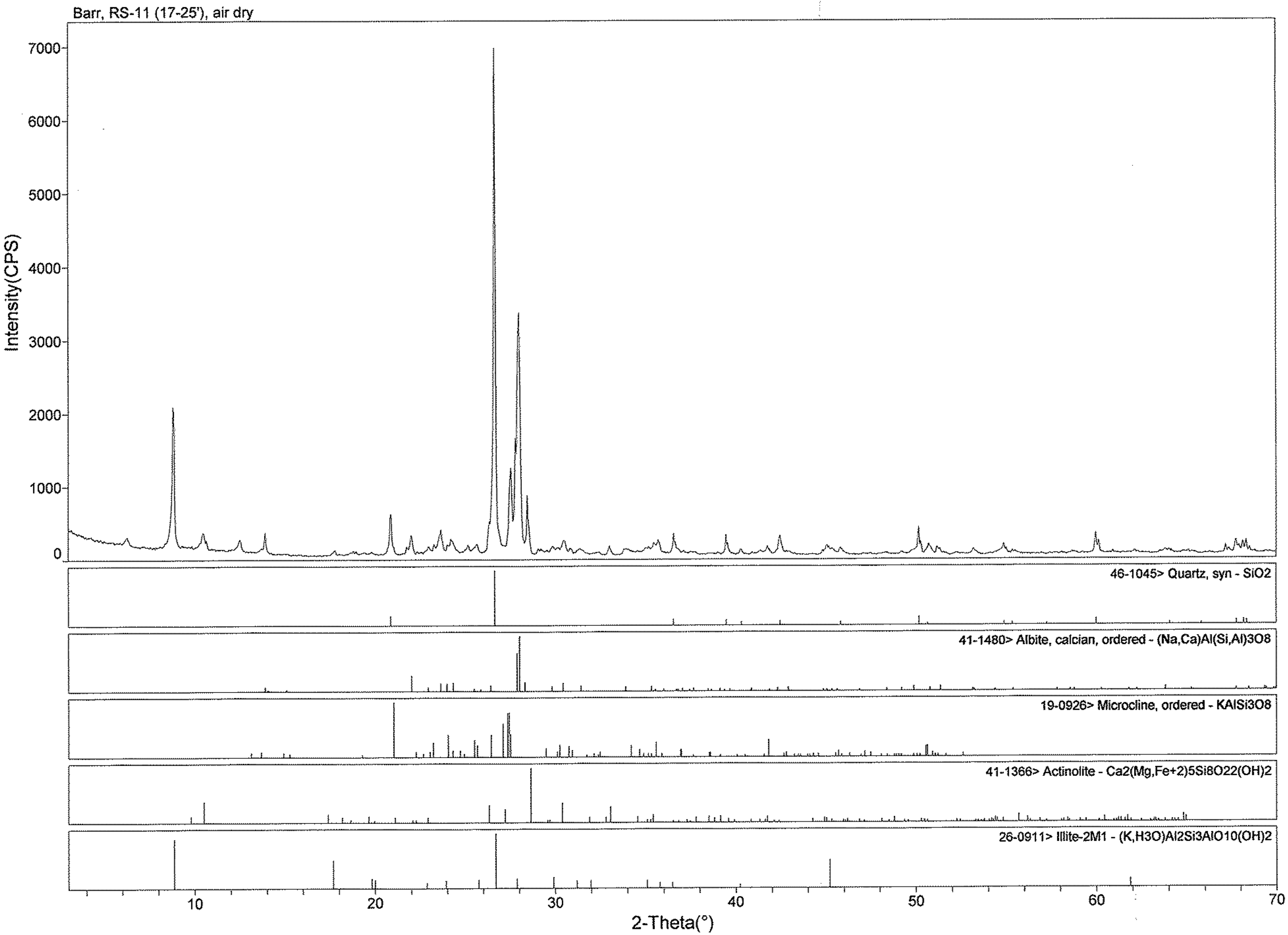

Figure 2. X-ray diffractogram of RS-11, 17-25′ (air-dry).

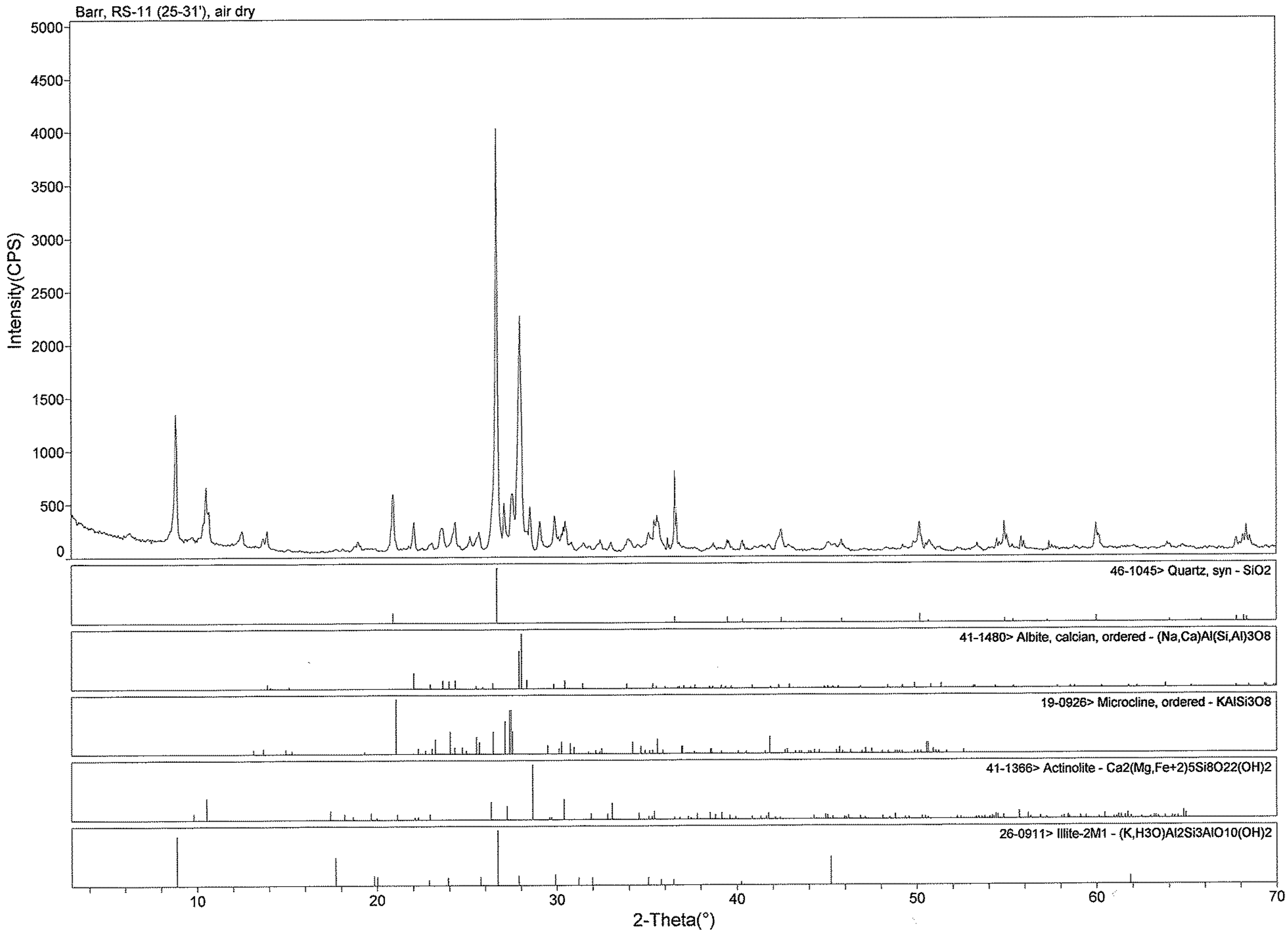

Figure 3. X-ray diffractogram of RS-11, 25-31′ (air-dry).

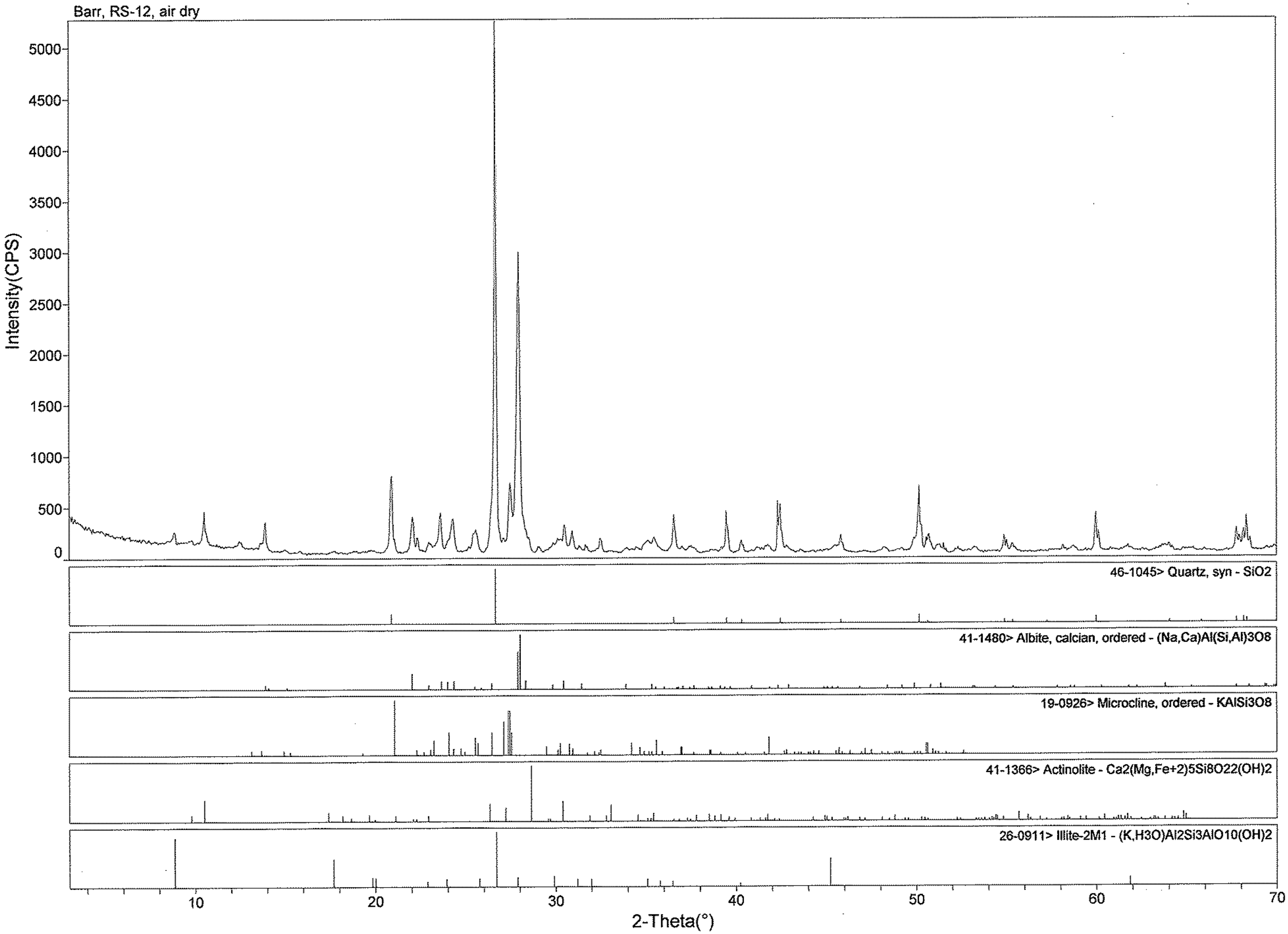

Figure 4. X-ray diffractogram of RS-12, 5.5-15′ (air-dry).

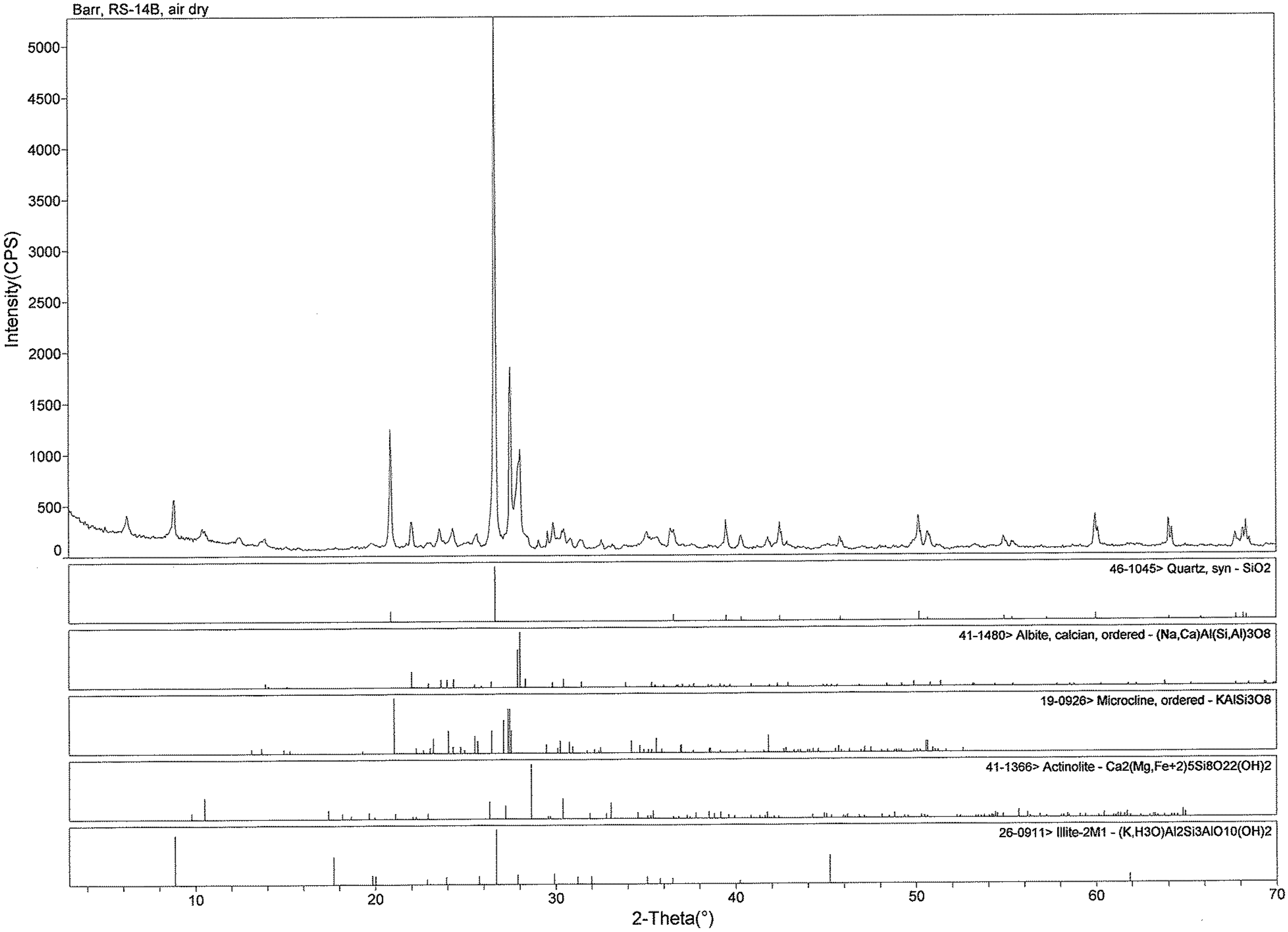

Figure 5. X-ray diffractogram of RS-14B, 0-5′ (air-dry).

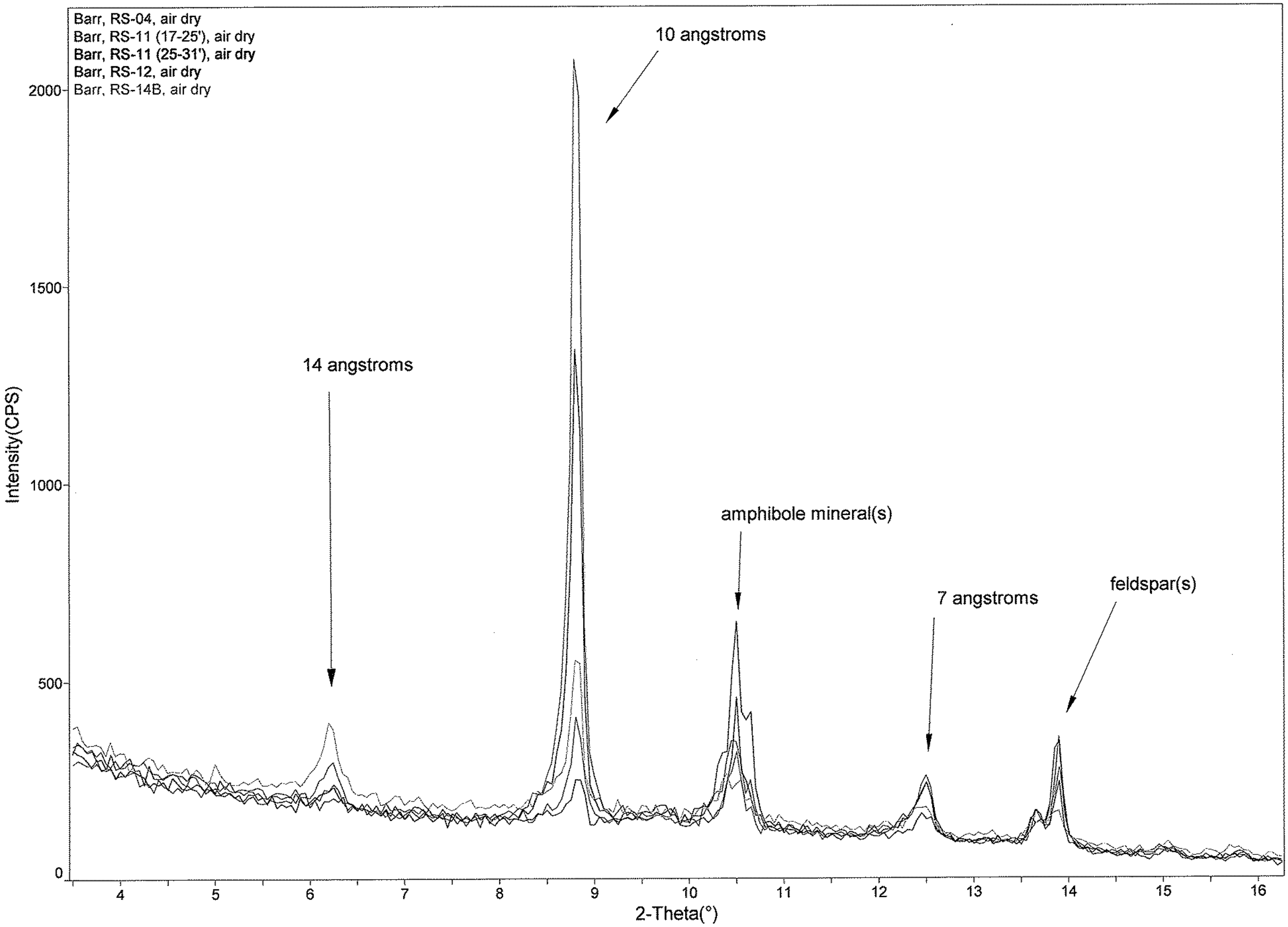

Figure 6. Overlay of X-ray diffractograms illustrating clays and other minerals.

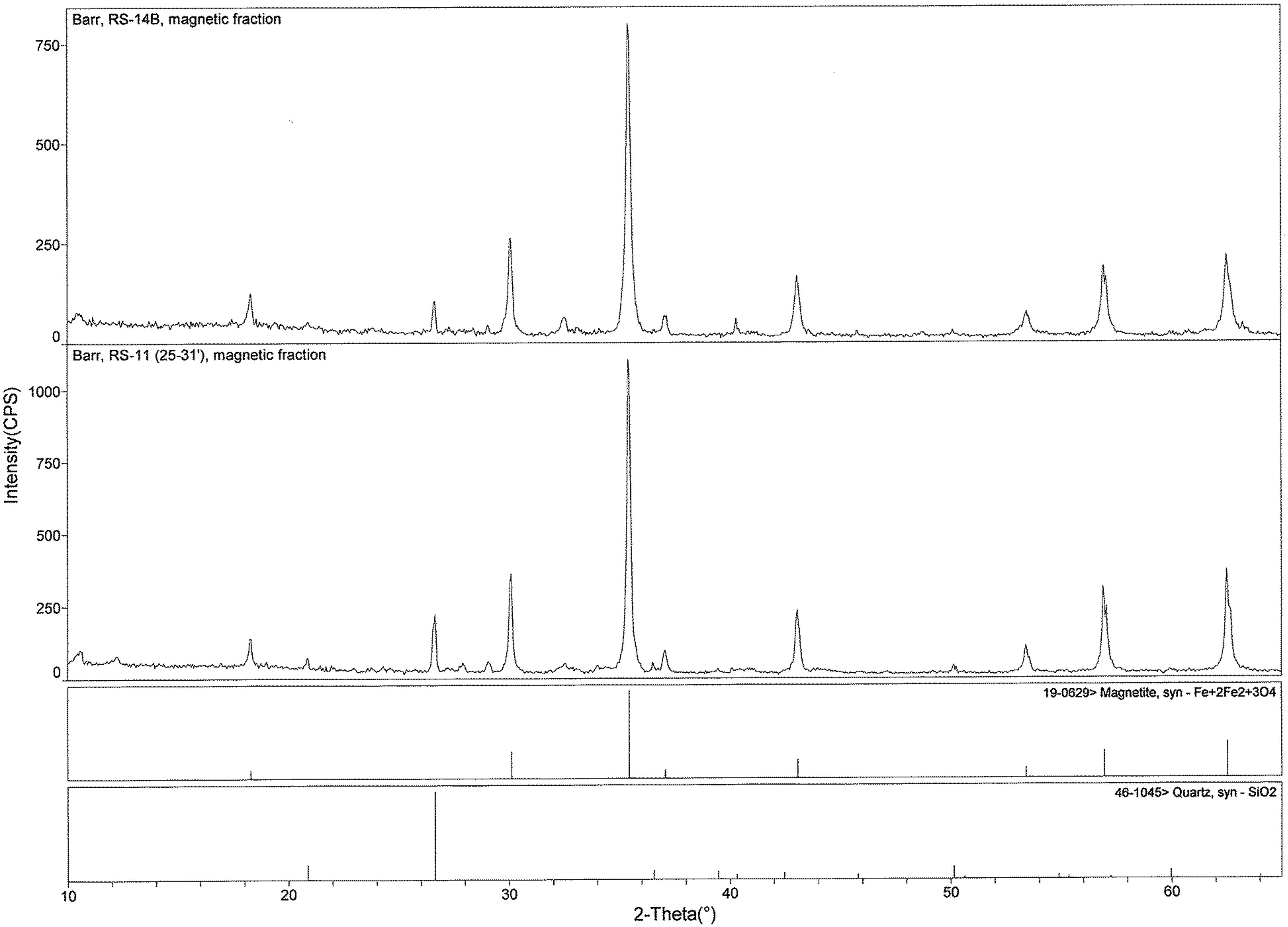

Figure 7. Overlay of X-ray diffractograms illustrating magnetite (from magnetic separation).

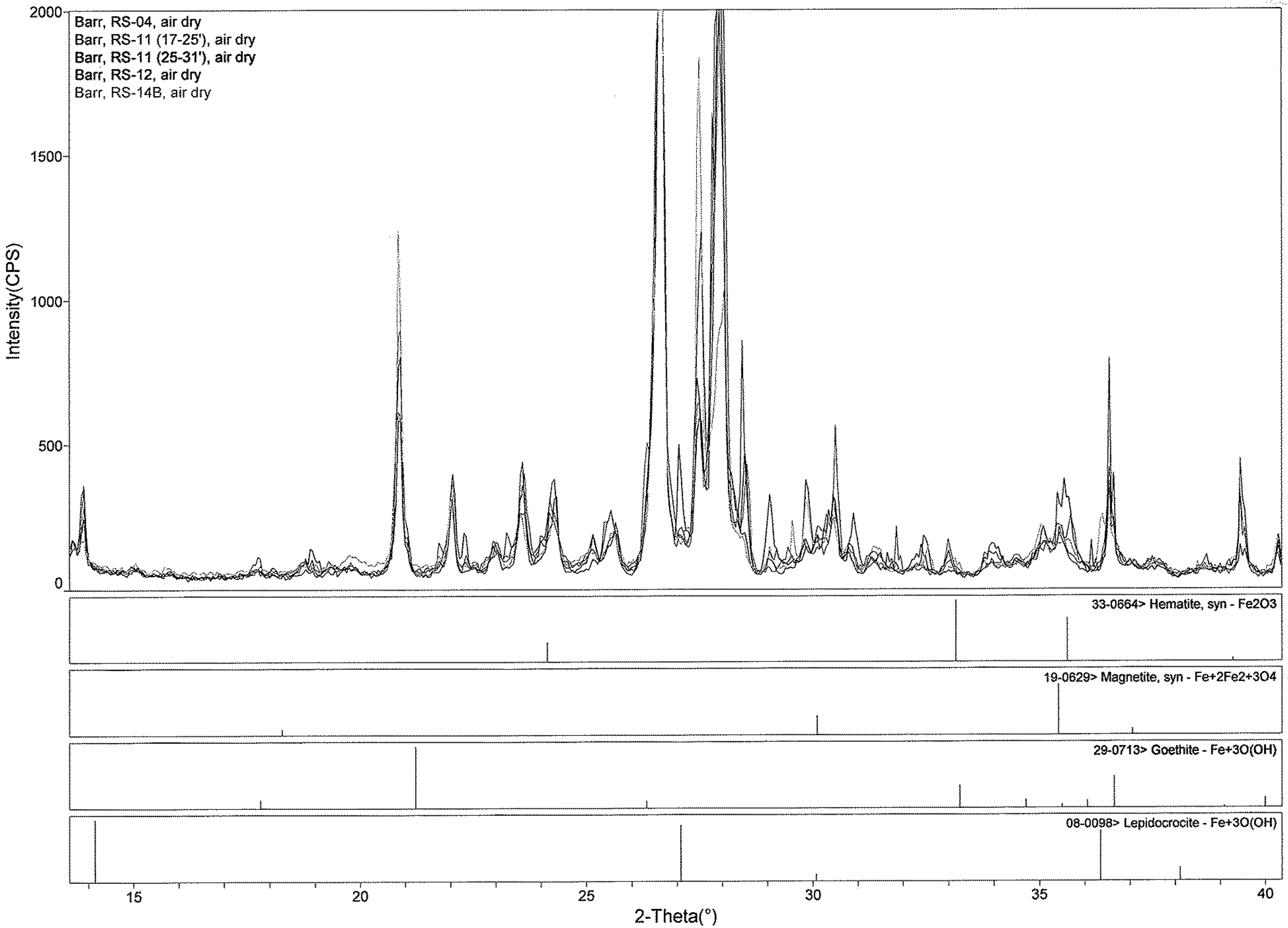

Figure 8. Overlay of X-ray diffractograms showing strong peaks for typical iron minerals.

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