Guidelines for the Determination of Mineralogy and Mineralogical Properties

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Geological Components

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Mine Components

↓

Minerals

↓ oxidation, reduction
leaching, hydrolysis

Mine Drainage

↓

Environmental Impact?

Purposes of a Mineralogical Assessment:

- Interpretation of static and kinetic test results typically includes assumptions about the minerals contributing to the test results. These mineralogical assumptions must be checked to avoid major errors in the interpretation of results.
- Mineralogical information also provides a check that static tests are conducted properly.
- Mineralogy used to check the assumption that all the sulfide-S will generate the same acidity as pyrite and the non-acid generating-S.
- Mineralogy needed to determine the silicate mineralogy and its potential as a source of neutralization.

Need to check potential for galvanic effects that may impact sequence of metal leaching or acid generation.

Sequence of weathering expected if there is galvanic interactions: Pyrrhotite > galena-sphalerite(Fe) > pyrite-arsenopyrite > magnetite

- Galvanic effects may delay oxidation of pyrite and thus the generation of acidic drainage (Day, 2003).
- Zn released initially at neutral pH conditions (Kwong, 1995).
- Requires contact between contributing sulfide minerals.

- Need to check the assumption that all the carbonate-C is calcite and estimate the amount of Fe and Mn carbonate, which is <u>not</u> net neutralizing under aerobic conditions.

  Many are solid solutions and may deviate from theoretical formula (e.g. Fe-bearing dolomite and Mg in ankerite).

Carbonate Minerals	**Theoretical Formula**
Calcite	$CaCO_3$
Magnesite	$MgCO_3$
Siderite	$FeCO_3$
Rhodochrosite	$MnCO_3$
Dolomite	$CaMg(CO_3)_2$
Ankerite	$Ca(Fe,Mg)(CO_3)_2$

Purposes in Mineralogical Assessment:

- Check for properties, such as the relative distribution of AP and NP minerals in fractures and veins versus that in the groundmass, that will result in the waste rock fines having a different composition from the whole rock.
- Check for potential sources of contaminants and indications of previous weathering.

A mineralogical assessment is typically required for each kinetic test sample and a ‘representative’ sub-set of the static test samples.

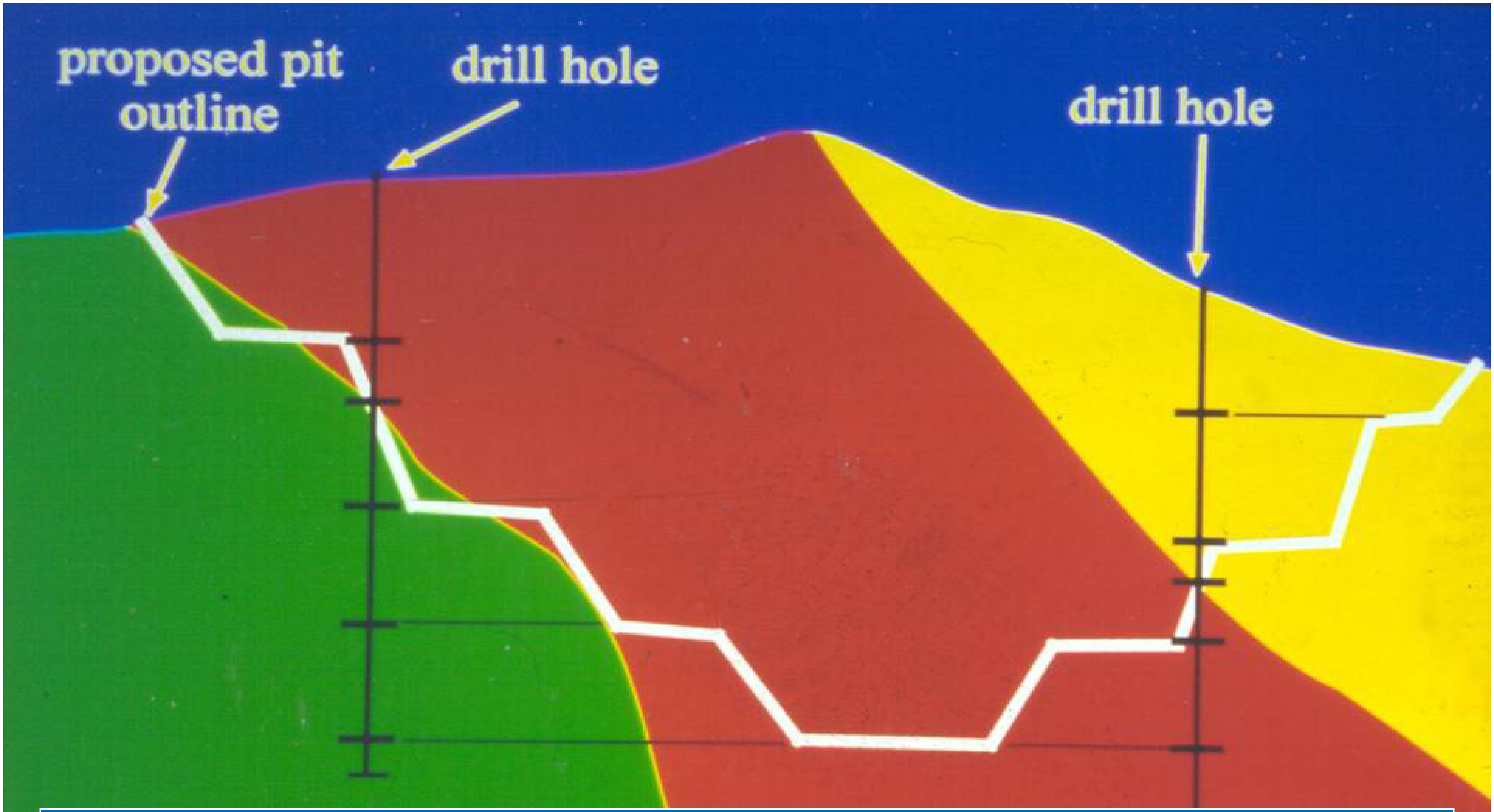
The information should include the spatial distribution and chemical composition of different minerals, in addition to their identity and proportion.

Mineralogical procedures include:

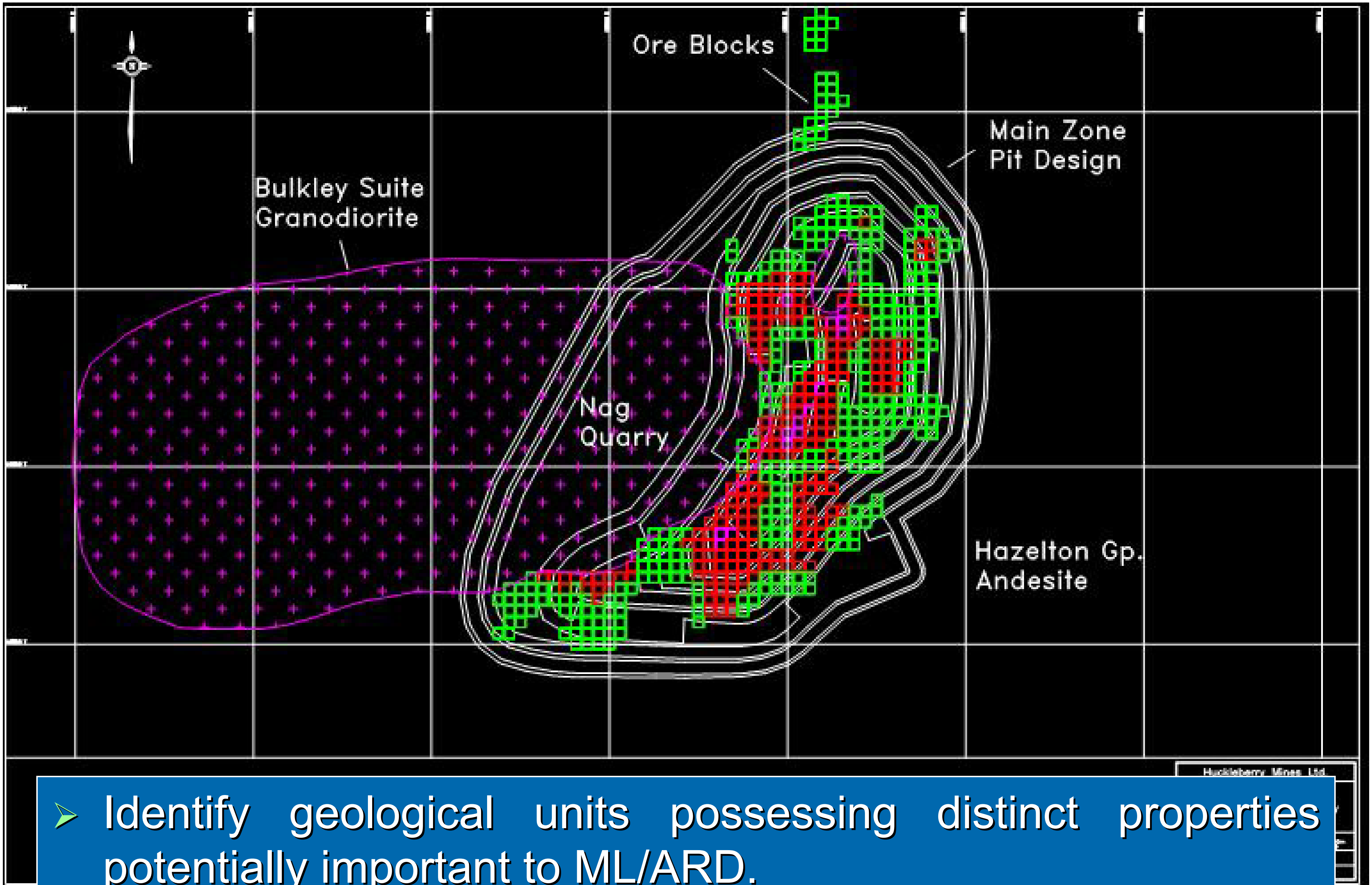
- Visual Description
- Petrographic Analysis
- X-ray Diffraction
- SEM/EDS
- Microprobe
- Laser Ablation

At a minimum should conduct first three. Other methods will be used to answer specific questions.

Visual Description



- Describe and map all geological materials excavated, exposed or otherwise disturbed.
- Describe the central tendency and variability, spatial distribution and alteration.



- Identify geological units possessing distinct properties potentially important to ML/ARD.
- Describe the variability and spatial distribution.

Visual description of drill core provides valuable information.

Logging of drill core should include assessment of mineralogical properties of value in drainage chemistry (ML/ARD) assessment, such as alteration, weathering and presence of organic-C and S (e.g., use hand lens and HCl fizz).

Petrographic Analysis

Petrographic Analysis: Advantages

- Contributes information regarding composition and variability of different geological units
- Identification of minerals present and their distribution, or relative abundance, alteration of silicates, presence of clays
- Particularly useful for identification of rims, oxidation of sulfides
- Can identify grains down to 50 um, all dependent on quality of microscope, may detect sulfides to 5-10 μm

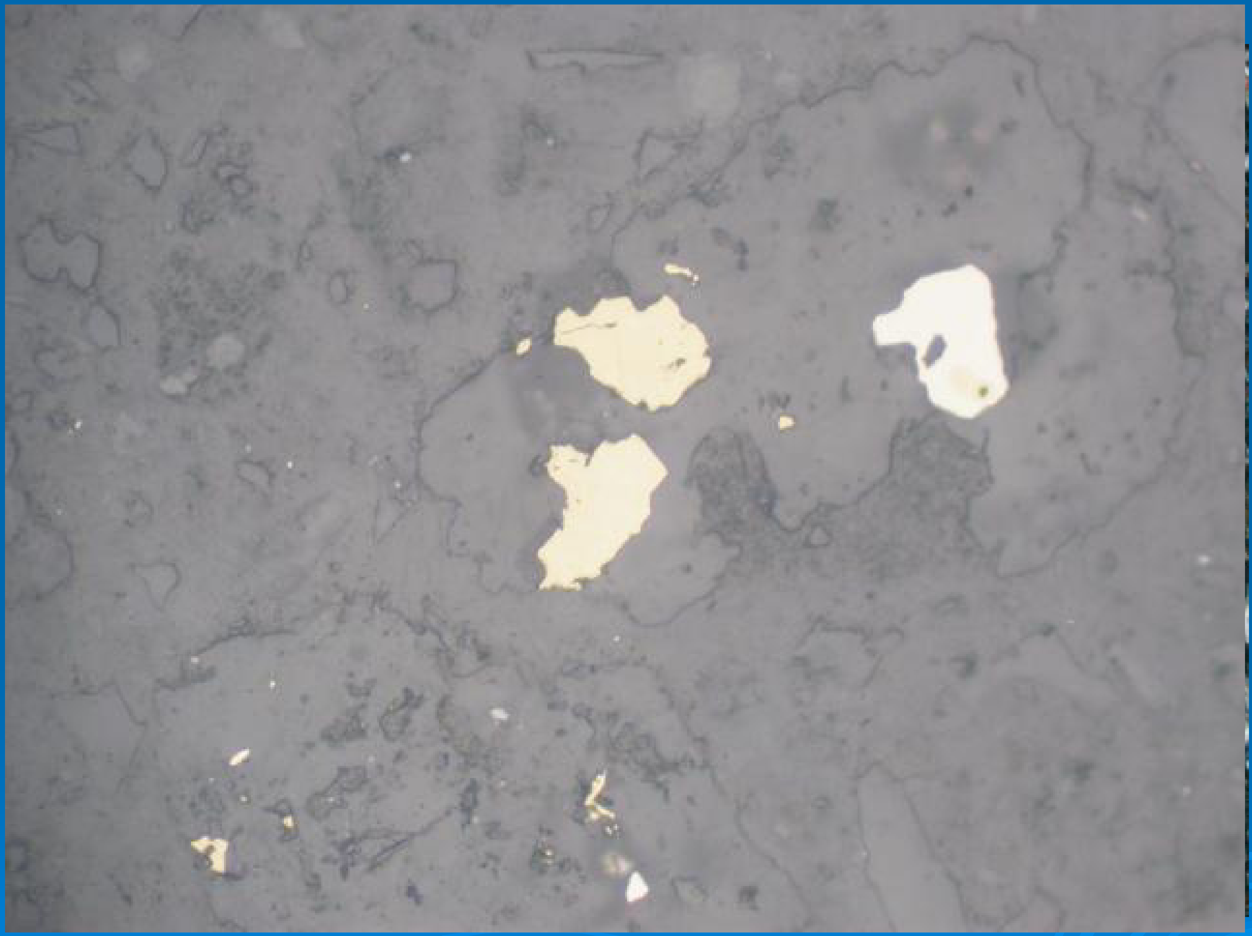
Also very useful in identifying relative percentages of different minerals in fractures and veins versus groundmass and identification of sulfide minerals.

Valuable check on XRD results.

Sample Types

- Rock Samples:
  - Provide textures, best assessment of alteration; weathering; localization of important minerals relative to each other and areas of weakness such as fractures
- Chip Samples:
  - May provide better overview of all the rock types present; but percentages of rock types present is not necessarily representative
- Powders:
  - Possible to extract information on mineralogy; particularly approximate sulfide present – least helpful in petrography
  - Often get mix of chips and powder

Example of powder & chip sample



FOV = 1.25mm

Requirements – before analysis

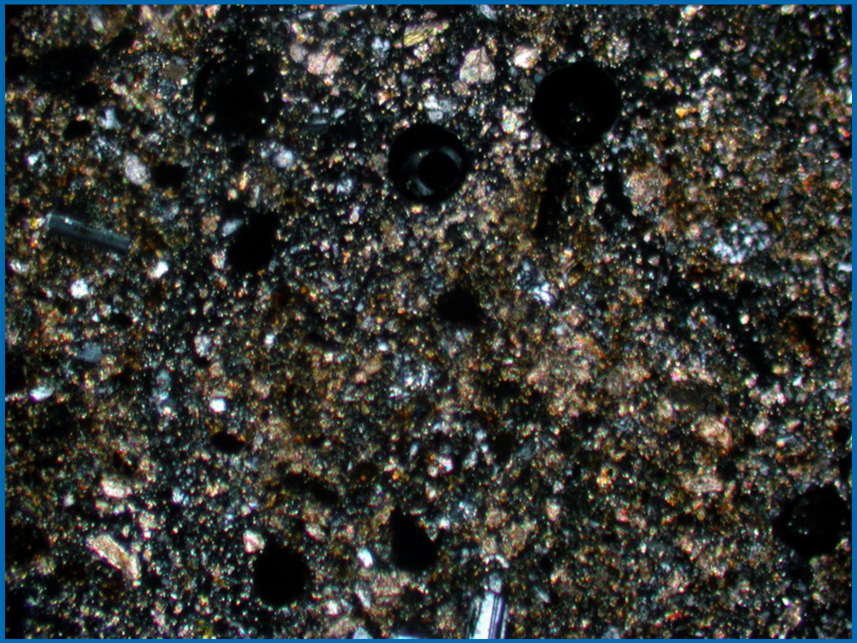
- Good Visual Descriptions
  - need use of hand lens, HCl and scratchers (done on rocks)
- Careful Sampling
  - representative of variability and material of concern – relies on previous work and deposit geology
  - number of samples – statistical representation
  - samples before and after testing, leach columns
  - preservation of oxidation (cells, rims); or limiting oxidation prior to sample preparation
- Sample Preparation
  - objective is 30 µm, polished thin sections; useful for identification of opaque minerals and for SEM-EDS analysis
  - “Best Practices” – game may be won or lost here

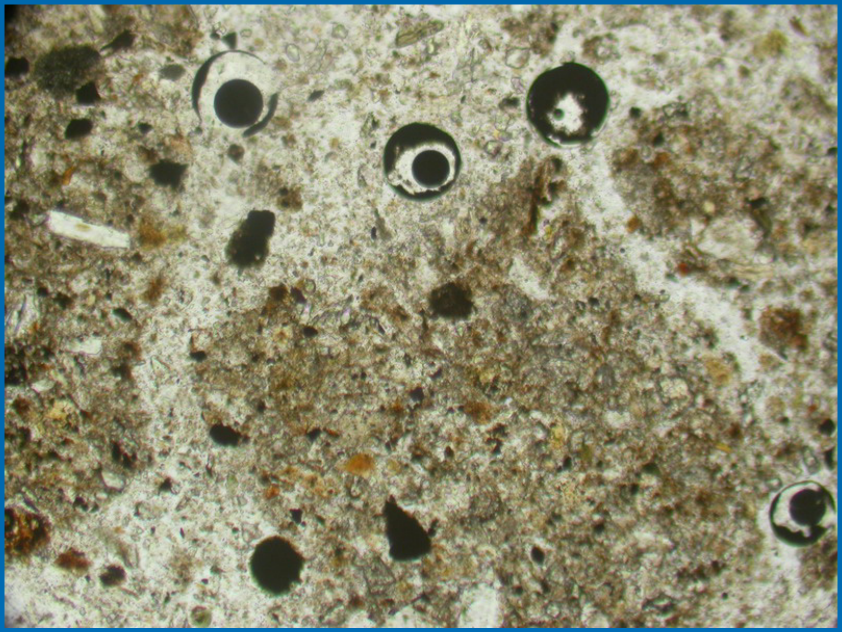
PROBLEMS ENCOUNTERED DURING PREPARATION*

- the materials are friable and fragile and require impregnation prior to sectioning;
- different resins must be used for different materials, e.g. polyester resins are the most suitable for impregnating clay-rich material, however they react with certain minerals (e.g. some sulfates), so are unsuitable for use with ocean floor materials;
- many samples are wet or damp, and must be dried prior to impregnation while minimizing exposure to elevated temperatures (see next point);
- clay-rich materials and certain sulfates (e.g. gypsum, anhydrite) react adversely to heat and to water, so preparation techniques have to minimize contact
- traditional grinding techniques use loose grinding powder, which can become embedded in the resin or soft clay-rich material;
- traditional polishing techniques, using metal laps, can cause plucking and surface deformation and cracking of minerals.

* Based on work by Philip T. McGuire, Department of Earth Sciences, James Cook University

Example of fine abundant carbonate





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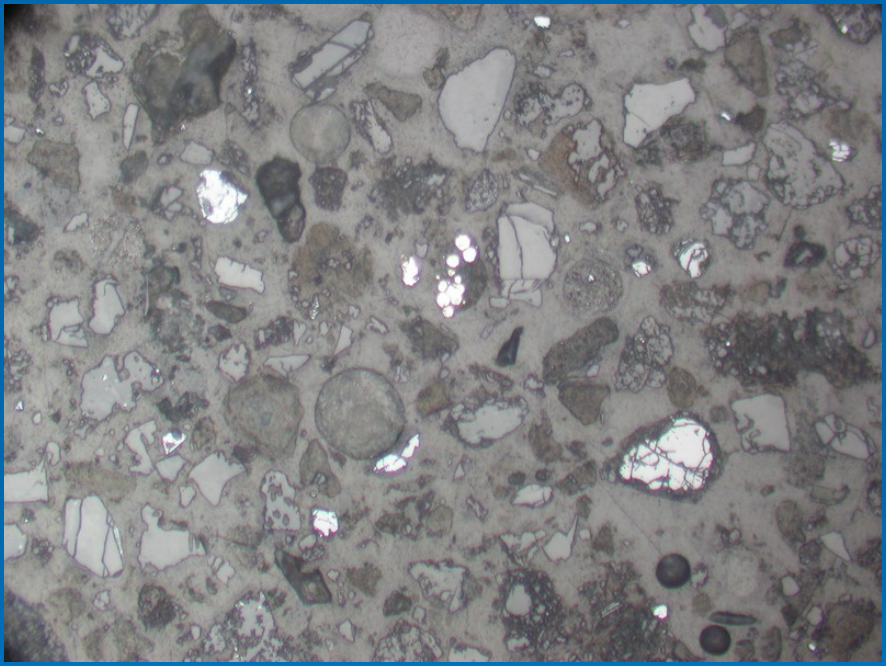
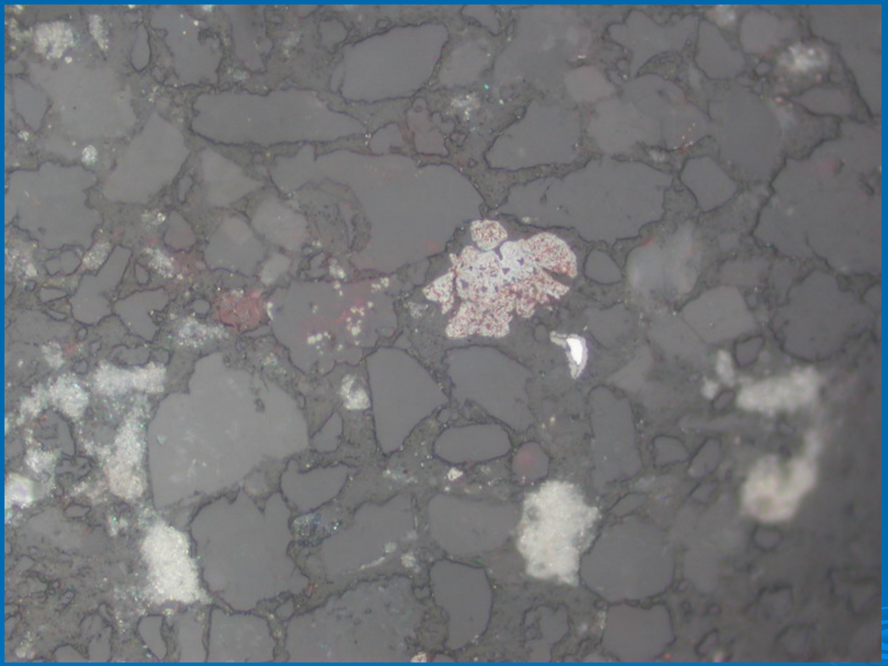
Examples of Sulfide/Oxides





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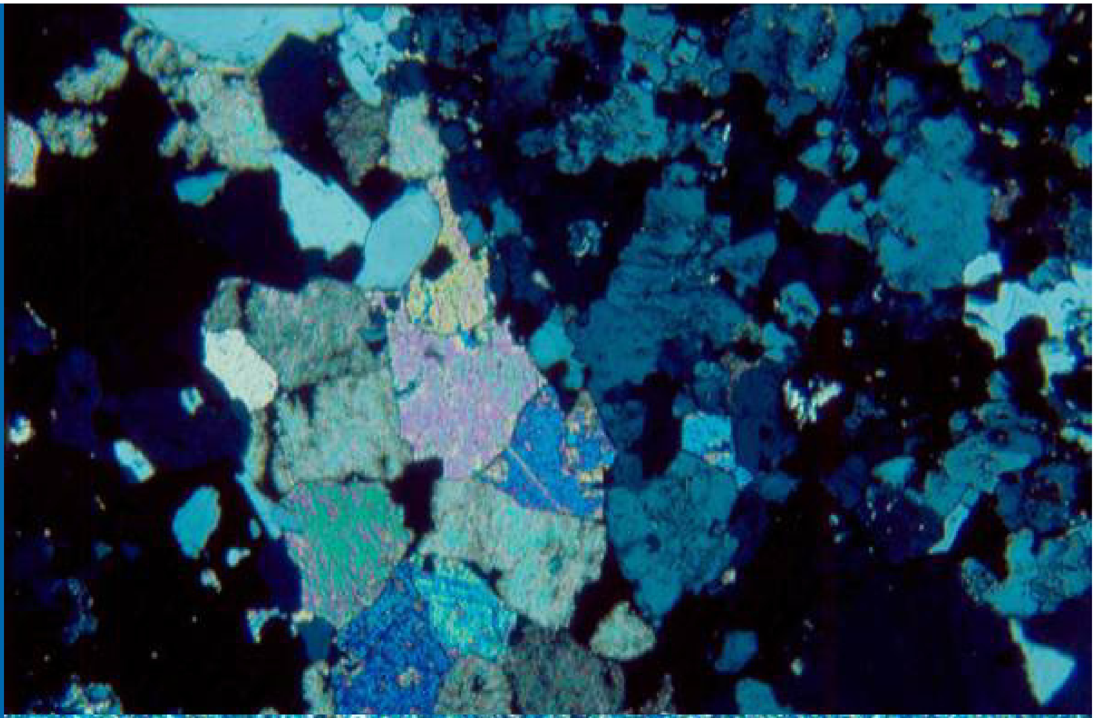
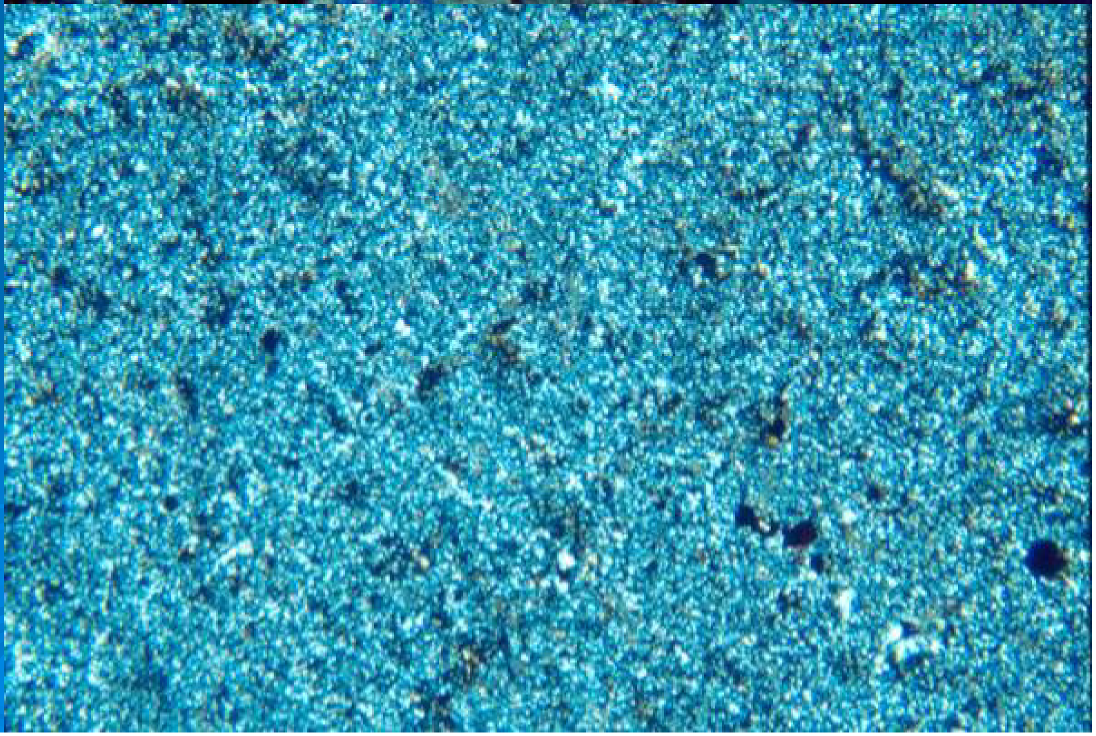
Petrographic Analysis: Disadvantages

- Not a stand alone technique – there is none!
- Subjective skill, dependent on "operator"
- Fracture coatings may be missed
- Heterogeneous samples; distribution, may not be covered in single thin section
- Distinguishing mineral compositions is difficult, needs support by SEM-EDS or microprobe

Petrographic analysis unable to:

- identify silicate mineralogy of small silicate grains < ~ 20 µm (e.g. tailings); or
- distinguish between carbonate species.





Carbonates

- Great range in carbonate compositions; can usually see calcite, not necessarily distinguish others, particularly as small grains
- Carbonates commonly vary within project area (sampling, and identification important)
- "Best" practice would include Rietveld XRD and SEM-EDS or microprobe in order to identify and quantify variations

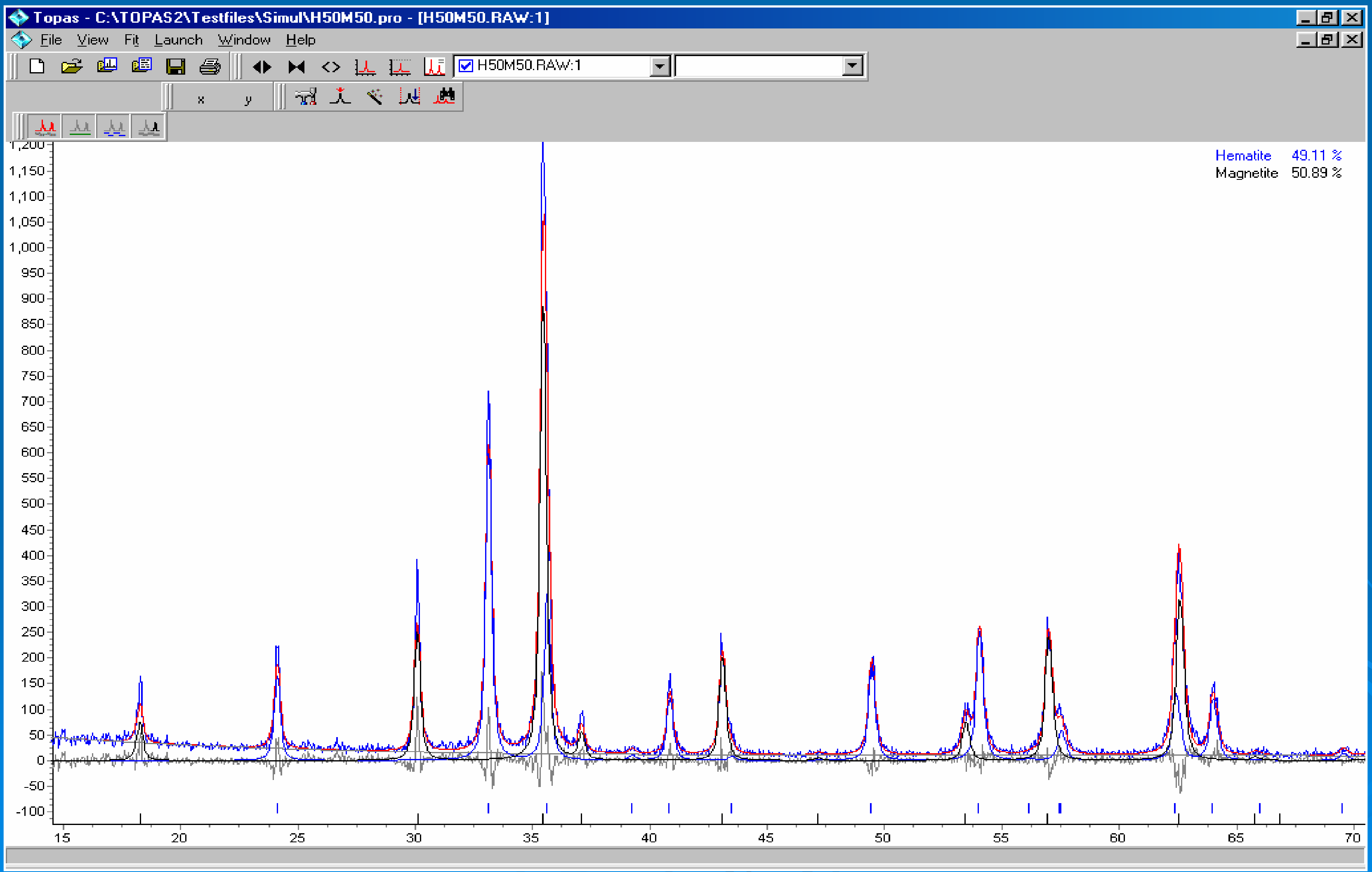
Guidelines for Petrographer

- Provide % estimates for minerals present; use easy to read table format for extraction of data
- Make clear to client that estimates are approximate (dependent on petrographer, sample, grain size, etc)
- Evaluate rims of sulfides carefully – consider loss of material during sectioning
- Consider possibility of man-made (e.g. roaster) products in material
- Make clear recommendations for further work, including alternative of SEM or Rietveld and need for other more specialized techniques such as short wave infrared spectrometry

XRD Analysis

Rietveld procedure is highly recommended

Rietveld XRD analysis provides almost quantitative mineralogy, including % of different carbonate minerals (see Raudsepp & Pani, 2001 & 2003).



An example of Rietveld XRD results for rock containing a number of different carbonate minerals.

Sample	Calcite	Siderite	Ankerite	Qtz	Albite	Ortho.	Kao.	Chl.	Musc.	Py.	Illite	Hem.	Bar.	Total
B23381	0.3	5.9	7.1	42.8	5.9		32.0		5.2	1.0				100
B23820	1.1	5.5	4.3	72.8			4.9		1.7	0.8				100
C17317		5.3	6.0	72.9	1.0		11.9		2.0			0.5	0.5	100
B23224	7.2	4.5	1.5	63.8			16.4		5.4	0.8			0.5	100
B23373	5.3	0.7		37.4	18.3	3.4	3.3	18.1	11.5	1.1	1.0			100
C14822	4.7	0.5		44.4	15.8	5.3		29.3						100
C17310	0.4		9.6	68.6			12.0		7.0	2.1			0.3	100
C17214	5.2		2.9	79.8			5.4		4.9	1.4			0.5	100
C17295	1.2			8.3	1.1	3.5		3.6		1.3				100
B23125	9.6			57.1		2.7	1.8	14.1	12.7	2.0				100
B23934	8.2			54.4	3.6	2.2	1.3	11.5	15.0	1.2	2.6			100
B23883	7.0			8.1				4.3	5.3	2.1			1.2	100

Minerals abbreviated: Quartz, Orthoclase, Kaolinite, Chlorite/Clinochlore, Muscovite, Pyrite, Hematite and Barite

Comments

- Limited number of institutions conducting Rietveld XRD
- Locally, at UBC – Mati Raudsepp
- Mining companies, e.g., Teck Cominco Ltd., Phelps Dodge

Microbeam-type Analysis

- SEM-EDS Analysis
- Electron Microprobe
- Laser Ablation

Microprobe and laser ablation measure chemical composition of minerals

- Estimate composition of carbonate minerals, such as ankerite and Fe-bearing dolomite, with variable composition
- Check assumptions that element comes from certain minerals (e.g., whether Ba and Pb only occur as sulfate minerals)
- Identify mineral sources and thus rate of release of potential contaminants (e.g., Se, see Day paper)

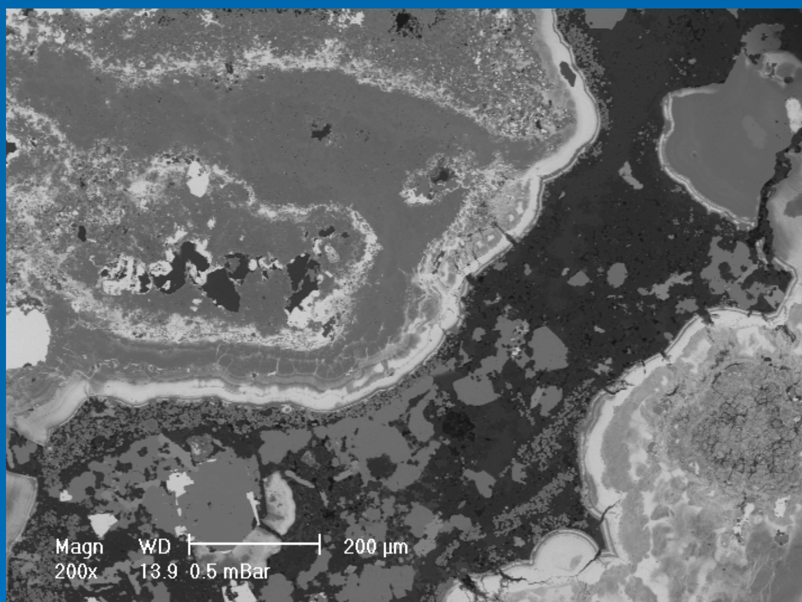
Example of microprobe analysis results indicating the chemical composition of carbonate minerals

	wt.% oxide						# atoms / formula unit			
	MgO	CaO	MnO	FeO	CO_2 *	Total	Mg^{2+}	Ca^{2+}	Mn^{2+}	Fe^{2+}
calcite	0.5	53.2	1.2	0.7	43.5	99.2	0.03	1.92	0.04	0.02
calcite	3.9	42.9	0.7	9.0	43.9	100.4	0.19	1.53	0.02	0.25
ankerite	11.7	20.9	0.4	22.0	42.9	97.8	0.59	0.77	0.01	0.63
ankerite	8.3	19.4	0.4	26.9	41.0	96.0	0.44	0.74	0.01	0.80
siderite	11.2	5.8	0.3	38.1	40.3	95.7	0.61	0.23	0.01	1.16
siderite	11.4	5.7	0.4	38.0	40.4	95.9	0.61	0.22	0.01	1.15
siderite	4.4	3.2	0.0	54.3	40.5	102.3	0.24	0.12	0.00	1.64

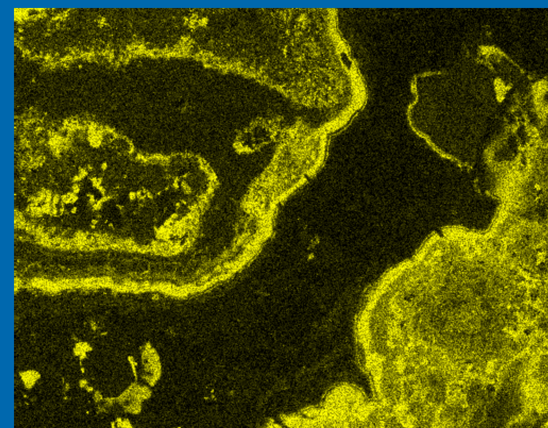
Where the $FeCO_3$ content may be significant, microprobe with Rietveld XRD can be used to estimate the $FeCO_3$ content and interpret TIC-NP and Sobek-NP results.

	Rietveld XRD (wt%)			XRD/microprobe		TIC-NP	Sobek-NP	
				$CaCO_3$	$FeCO_3$			
#	Cal.	Ank.	Sid.	NP	NP		Fizz	NP
S1	0.3	7.1	5.9	47	74	138	Mod	102
S2		6.0	5.3	41	62	125	Slight	68
S3	7.2	1.5	4.5	83	42	130	Slight	86
S4	8.7	5.2	2.5	124	35	178	Mod	154
S5		6.4	2.4	42	40	108	Mod	95
S6	0.1	1.7	1.5	14	16	38	Slight	28
S7	12.9	4.7	1.1	160	21	173	Mod	168
S8	5.3		0.7	53	6	41	Slight	51

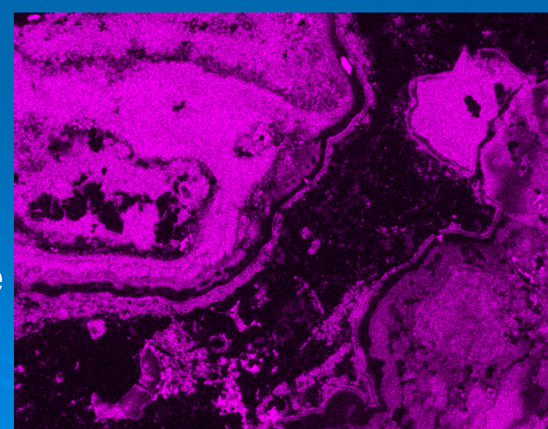
- SEM/EDS (X-ray mapping) also used to estimate proportion of different minerals



Backscattered image of pyrite, sphalerite and galena ore, scale bar 200 µm



Pb



Fe

General Considerations:

- Some information on mineralogy and mineral distribution may already be available in drill logs, exploration reports, metallurgical test work and academic reports; for example, some clients may have information on clays and sulfates from SWIR data.
- Should provide geochemistry and any other information to mineralogist/petrographer – this will help in determining protocol for sample preparation and interpretation of the results.
- Generally speaking, the more lines of evidence available for consideration, the more accurate is the resultant mineral identification.

Conclusions

Mineralogical Information is Invaluable

- Mineralogy will indicate which minerals are likely to contribute to static test measurements, such as AP and NP, and is used to assess the likelihood that they will contribute similar amounts in the field.
  - Rietveld XRD used to determine if Fe and Mn are significant part of CO_3 mineralogy (approx. $200 per sample).
  - Identify elemental composition of CO_3 mineral with microprobe if relative magnitude of Ca and Mg portion of solid-solution minerals is important (approx. $300 for 6 samples with 5 grains each sample, excluding petrog/prep).

- Kinetic Testing: If possible determine mineralogy of test material before and after
  - Identify presence of gypsum and other soluble species;
  - Provides check on assumptions regarding likely source of solutes measured in leachate; and
  - Indicate if some weathering products were not removed in leachate.
- Other properties of interest will depend in part on the questions raised by other test work and the specific conditions of each site, material and disposal option.

Reasons given for inadequate mineralogy:

- Not an ARD specific-test
- Thought to be expensive
- Not part of standard ARD prediction packages

Expect to spend $400-500 total per sample on at least 2 methods of mineralogical analysis. Costs are similar to those of ABA and less than kinetic tests.

Costs of inadequate mineralogical understanding are often prohibitive in terms of delayed approval and environmental risks.

- Given the difficulty in acquiring accurate, quantitative data, careful planning is required to obtain useful mineralogical information at a reasonable cost. As with other analytical procedures, need to make sure you analyze the materials and the fractions of concern.
- Increasingly BC mines are using the Rietveld XRD technique (e.g., Kemess, Mt Polley, Snip).
- SEM/EDS and microprobe are also starting to be used in answering regulatory questions.
- In good drainage chemistry prediction, mineralogy is now a required not an optional analysis.