

Why Quantitative XRD is a Valuable Tool

- XRF reports all Elements as Oxides
 - For example, all calcium in sample is reported as CaO
- XRD Reports Compounds per Crystalline Structures
 - For example, reports calcium as:
 - Calcite: CaCO_3
 - Lime or Quick Lime: CaO
 - Hydrated Lime or Portlandite: Ca(OH)_2
 - Amorphous (no structure) Calcium: Ca
 - Amorphous sulfur can be organic sulfur as S or fine precipitated sulfates that have not crystallized

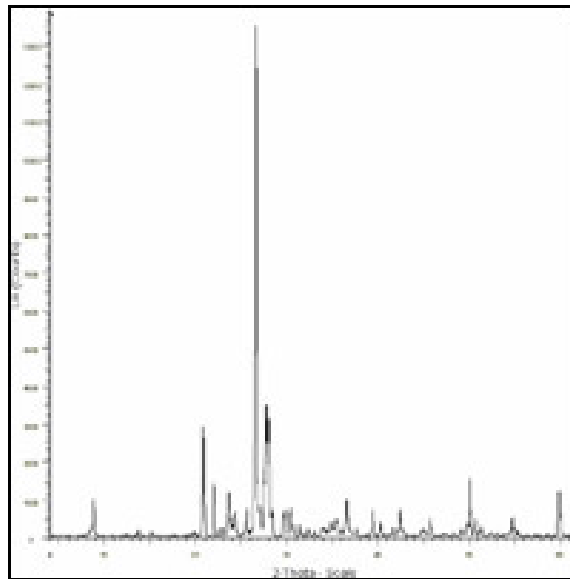
Preparation Laboratory



PMET maintains a complete lab for analysis supporting the ash technology development and preparation of samples for SEM and XRD analysis.

Quantitative XRD

Typical Results of XRD Analysis (Wt%)



Exploration Drill Core	
quartz	33.6
k-feldspar	8.2
plagioclase	23.4
muscovite	14.4
biotite	3.4
chlorite	8.0
kaolinite	5.0
hematite	0.3
pyrite	1.6
chalcopyrite	2.1

XRD Analysis of Exploration Drill Core

Our XRD lab uses a Siemens D500 diffractometer with a Solex solid-state scintillation detector and state of the art data acquisition and processing software.

Acid Mine Drainage Chemistry

- Not all forms of Sulfur Produce Acid Mine Drainage
- Formed via Oxidation of Pyrite:
$$\text{FeS}_2 + \frac{15}{4} \text{O}_2 + \frac{7}{2} \text{H}_2\text{O} \rightarrow \text{Fe}(\text{OH})_3 + 2 \text{H}_2\text{SO}_4$$
- Quantification of FeS_2 is Key Factor
- How to determine quantity of sulfur present as FeS_2 ?

Answer:

Quantitative XRD by Rietveld Refinement

- The Rietveld method is a whole pattern refinement based on instrument parameters, phase chemistry, and crystalline structure.
- A standard spike is used to calibrate system parameters and to calculate the concentration of non-crystalline material.

Example Analysis of Major Brick Manufacturer Sample

- First Step, Measure Total Sulfur of the Samples

Total Sulfur Analysis	
Sample A	Sample B
0.120%	0.122%

- Based on Sulfur Content Alone, Samples Appear Similar

Example Analysis of Major Brick Manufacturer Sample

Compound	Sample A %	Sample B %
pyrite	0.10	0.00
anhydrite	0.2	0.1
jarosite	0.1	0.3
hematite	0.1	0.7
quartz	33.9	27.5
K-feldspar	2.3	2.8
plagioclase	2.4	4.0
muscovite	8.7	11.2
clinochlore	0.1	1.4
calcite	1.0	9.3
dolomite	0.2	0.6
goethite	0.0	0.3
kaolinite	28.3	7.2
Amorphous	22.6	34.6

- Second step is analysis by XRD
- XRF analysis of this sample would report:
 - Pyrite as SO_3 & Fe_2O_3
 - Anhydrite as CaO , SO_3
 - Jarosite as K_2O , Fe_2O_3 , & SO_3
 - Hematite as Fe_2O_3
- XRD directly measures the amount of Pyrite

Example Analysis of Major Brick Manufacturer Sample

		Sample A	Sample B
Total Sulfur		0.120%	0.122%
Quantitative XRD			
Pyrite	FeS₂	0.10%	0.00%
Anhydrite	CaSO₄	0.2%	0.1%
Jarosite	KFe⁺³(SO₄)₂(OH)₆	0.1%	0.3%
Sulfur Partitioning			
Total Sulfur as FeS₂		44.2%	0%
Total Sulfur as Anhydrite		45.1%	19.3%
Total Sulfur as Jarosite		10.7%	31.5%
Total Sulfur in Amorphous		0%	49.2%

The quantity of sulfur in the form of pyrite is determined