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

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Preparation Laboratory

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

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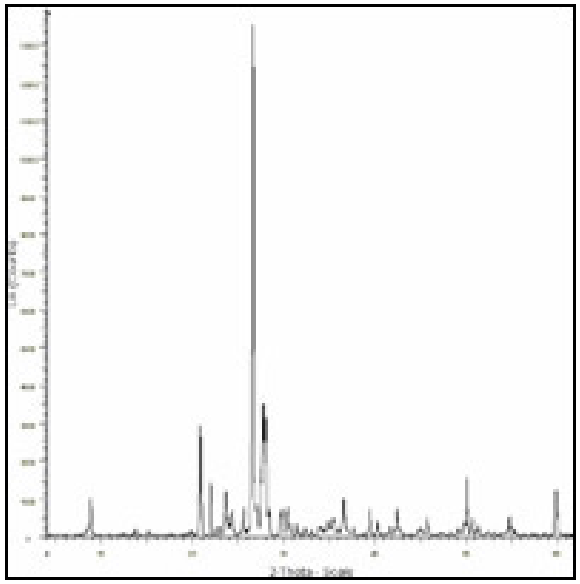
Technology Development: From Concept Through Commercialization

Quantitative XRD



XRD Analysis of Exploration Drill Core

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

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.

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Acid Mine Drainage Chemistry

- Not all forms of Sulfur Produce Acid Mine Drainage
- Formed via Oxidation of Pyrite:

  $$FeS_2 + {}^{15}/_4\ O_2 + {}^{7}/_2\ H_2O \rightarrow Fe(OH)_3 + 2\ H_2SO_4$$

- 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	**FeS_2**	**0.10%**	**0.00%**
Anhydrite	**$CaSO_4$**	0.2%	0.1%
Jarosite	**$KFe^{+3}(SO_4)_2(OH)_6$**	0.1%	0.3%
Sulfur Partitioning			
Total Sulfur as FeS_2		**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