

Analysis of Mining Ore

ARL QUANT'X with Si(Li) Technology Energy-Dispersive X-Ray Fluorescence Spectrometer

Key Words

- ARL QUANT'X
- Si(Li) Detector
- Ore
- Mining
- X-Ray Fluorescence

Introduction

Elemental analysis of mined ore, its concentrates and refined products is an important part of any mining and metal refining operation. X-ray Fluorescence (XRF) spectrometry has been proven a very cost-effective and reliable elemental analysis technique with thousands of units installed around the world. Both ore and metal samples can be analyzed in solid-state, and most measurements can be completed by unskilled operators in a few minutes with excellent precision. Thus XRF avoids the difficulty, expense and delays associated with traditional wet chemical analysis. This publication presents the procedure and typical results obtained with the Thermo Scientific ARL QUANT'X XRF spectrometer for measuring Zn, Pb, Ba, Fe and Si in a variety of sulfide ores.



Instrumentation

The ARL QUANT'X is based on the Energy-Dispersive XRF (EDXRF) principle that allows a single highly-sensitive detector to measure the emission lines of all elements from Sodium (Na, Z=11) to Uranium (U, Z=92). While EDXRF has increasingly limited sensitivity to lighter elements and is rarely used to measure Sodium (Na) or Magnesium (Mg) at concentrations below 1 %, advances in technology now extend its practical range into heavier-metal applications traditionally served by the more sensitive and larger Wavelength-Dispersive (WDXRF) spectrometers.

The full vacuum chamber, large active area and the lowest operating temperature and noise characteristics of any energy-dispersive detector allow the ARL QUANT'X to achieve remarkable sensitivity and repeatability. Unlike most laboratory-grade instruments, the durable mechanical design, compact footprint and minimal site requirements of the ARL QUANT'X permit placement in rough industrial environments. The hardware is controlled solely by software to assure data integrity and reduce operational complications due to mechanical buttons or controls.

Excitation Conditions

In EDXRF, sensitivity and precision are achieved by targeted excitation of the sample to fluoresce only the elements of interest. An instrument with more flexibility and control over the excitation efficiency and background typically shows better performance. The ARL QUANT'X offers a virtually unlimited combination of excitation voltages 4-50 kV and multiple primary beam filters for optimal background control. As shown in Table 1, three spectra were collected off each ore sample, for a total counting time of 3 minutes in a low vacuum atmosphere.

Spectrum	kV	Filter	Time (s)	Analytes
1	4	0	60	Si, S
2	8	1	60	Ba
3	28	5	60	Fe, Zn, Pb

Table 1: Analytical settings

Figure 1 shows an example of spectra 1 and 3 collected from three very different ore samples. The composition of these samples is given in Table 3.

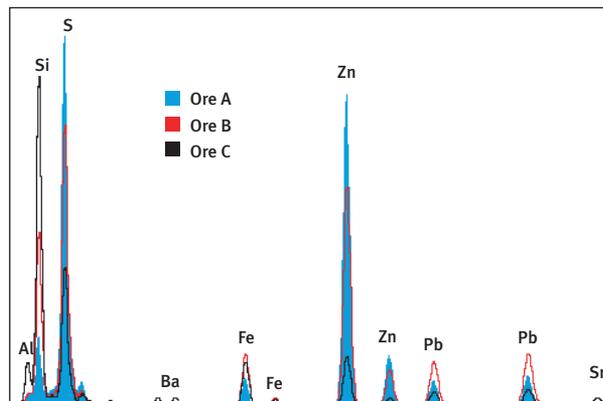


Figure 1: Spectra of three three ore samples under two excitation conditions

Sample Preparation and Presentation

For qualitative analysis, XRF requires no sample preparation at all, regardless of sample shape, size or amount. However, for meaningful quantitative analysis, ore samples are typically crushed and ground in a mill to produce particle size of less than 50 microns (325 mesh), which helps minimize particle-size and mineralogical effects. Although the powder can be analyzed directly, accuracy and sample-to-sample repeatability are improved if the powder is pressed into what's called a "pellet" using a manual or automatic press at 15-20 tons.

Calibration

Most applications of XRF in industrial process control are calibrated using empirical calibrations on the basis of primary or secondary standards. A variety of calibration types and functions, including curve display and fit evaluation, are included in the standard software package supplied with the ARL QUANT[®]X.

The results reported below were obtained using multi-variable regression analysis based on 14 secondary standards with nominal values obtained by Inductively-Coupled Plasma spectroscopy (ICP). Table 2 shows the Standard Error of Estimate (SEE) for the five analytes of interest. The SEE is the average difference between the nominal and calculated concentrations of a given analyte.

Analyte	Spectrum	Calibration Range (%)		SEE %
		Min	Max	
SiO ₂	1	7.7	65	2.2
Fe	3	3.1	15	0.2
Zn	3	0.3	39	0.2
Ba	2	0.3	16	0.1
Pb	3	0.1	18	0.1

Table 2: Summary of Calibration Accuracy

Repeatability

Repeatability tests were performed to show the precision achieved by the ARL QUANT[®]X for the 3-minute total counting time. Table 3 shows the statistical summary for several ore samples representing a wide range of concentrations.

In some cases, the difference between the nominal (given) composition and the average measured result was larger than 3-sigma of variation due to the instrument itself. Here, the discrepancy is caused by the sample (pellet) preparation and the large variety of ores used in this test. For this reason, optimal accuracy for measurement of geologically-derived materials is achieved by fusing the samples with lithium tetraborate, which removes grain size and mineralogical effects. However, due to the dilution and signal reduction that happens as a result of fusion, this type of sample preparation is only suitable for the WDXRF technique.

	Nominal	Ore A	
		Average	1-Sigma
SiO ₂	12.2	13.6	± 0.2
Fe	8.5	8.6	± 0.1
Zn	38.7	38.5	± 0.3
Ba	0.60	0.55	± 0.02
Pb	6.96	6.77	± 0.09

	Nominal	Ore B	
		Average	1-Sigma
SiO ₂	37.3	37.4	± 0.2
Fe	12.0	12.1	± 0.1
Zn	16.4	16.1	± 0.3
Ba	1.56	1.47	± 0.02
Pb	5.51	5.38	± 0.03

	Nominal	Ore C	
		Average	1-Sigma
SiO ₂	53.1	53.6	± 0.1
Fe	4.97	4.90	± 0.02
Zn	1.45	1.38	± 0.01
Ba	9.59	9.90	± 0.02
Pb	0.500	0.409	± 0.005

Table 3: Measurement Precision

Conclusion

The ARL QUANT[®]X EDXRF spectrometer has been used to successfully analyze a variety of elements in mining ore. A simple three-minute analysis under vacuum achieved precision <1 % relative standard deviation (rsd) for all major components. The speed, sensitivity, mechanical simplicity, durability and low operational costs of the ARL QUANT[®]X make it an excellent analytical value for process control applications in the mining industry.

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