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# XRF1061 - Polymetallic sulfide ore analysis with Supermini200

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## Introduction

Polymetallic sulfide deposits are often major sources of copper, zinc, lead, gold and silver ore. Some of these deposits also contain many various metals such as co- or by-products and impurities.

Laboratories in mining sites are often required to analyze large number of samples per day. Therefore, in order to determine multiple elements in ores, simple and fast analysis technique with high accuracy and precision is demanded.

X-ray fluorescence (XRF) analysis is a well-known technique to accurately and quickly determine elements in samples. Pressed powder method in X-ray fluorescence spectrometry is the best solution in terms of simplicity of analysis for powder sample. Since plenty of X-ray lines due to its various metal elements can be detected in polymetallic sulfide, high spectral resolution optics is essential for high quality analysis. Furthermore, appropriate corrections for complex matrix effects owing to its elemental and compositional variations are required in XRF analysis. Conventional correction technique for metals in ores is a method using Compton scattering as internal standard but the results were often not satisfactory for meeting the requirement of the mining industry. Rigaku has developed an improved Compton scattering method by integration of theoretical alpha correction for significantly improved analytical results.

This note demonstrates rapid and accurate determination of multiple elements in polymetallic sulfide ores by pressed powder method using Supermini200.

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## Instrument

The Supermini200 is a unique benchtop sequential wavelength dispersive (WD) XRF spectrometer with high spectral resolution and high sensitivity for light elements. The spectrometer is designed to minimize installation requirements such as cooling water, special power supply and space. For the analysis of light elements, the newly developed sealed proportional detector (S-PC) can be substituted (option) for the conventional flow proportional detector (F-PC). Since the S-PC eliminates the need for an external P10 (detector) gas supply, the Supermini200 can be a truly utility-free WDXRF instrument. It is equipped with an air-cooled 200W X-ray tube and up to three analyzing crystals allowing analysis of element oxygen to uranium.

The functionally versatile software allows easy setup of various types of applications. In particular, the flowbar guides the user step by step for the setting up of quantitative and qualitative analysis conditions.

The user friendly interface of the semi-quantitative analysis program "SQX" allows quick elemental determination of unknown samples without the need for reference materials.

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## Quant Scatter FP function

“Quant Scatter FP” is a unique function integrated into the Rigaku Fundamental Parameter (FP) system. The function is able to generate dedicated theoretical alphas to correct for matrix effects in empirical calibration curves by the internal standard method using Compton scattering X-ray. This method without theoretical alphas has been preferred for heavy element analysis in some ores because of its ease of use. However, this conventional method does not fully correct for matrix effects especially for ore samples with high heavy element content. Rigaku’s correction scheme overcomes this problem by integrating the Compton scattering internal standard correction method with theoretical alpha correction by FP method. This advanced correction method for powder samples provides improved analysis results of ores.

“Quant Scatter FP” function (optional) is available for Supermini200, Rigaku Simultix and ZSX Primus family.

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## Standards and sample preparation

The calibration standards used for this study were commercially available 7 certified reference materials (CRMs). These standards are composed of mainly typical ores from volcanogenic massive sulfide (VMS) deposits.

Careful and consistent pulverization is quite essential to obtain good results because sample inhomogeneity due to complicated mineral assemblage can be a source of error.

The well-dried (2 hours at 105°C) samples were ground with a tungsten carbide container and then pressed under pressure at 200 kN using sample cups without binding agent. Generally, any binding agents introduces some analytical errors in internal standard method using the Compton scattering X-rays. Therefore, it is strongly advisable to press samples without binding agents when this application technique is applied. Pressed pellets were covered by polymer thin film (4 µm Prolene® film) for measurement.

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## Measurement and calibration

Supermini200 was used for measurement of the element lines of Na, Mg, Al, Si, P, S, K, Ca, Ti, Cr, Mn, Fe, Cu, Zn, As, Ag, Cd, Sb and Pb and Pd-Ka Compton scattering line. A primary beam filter was used for Ag, Cd and Sb measurements to reduce background.

Calibration equations for Fe, Cu, Zn, As and Pb are as follows:

$$W_i = (AI_R^2 + BI_R + C) \cdot (1 + \sum \alpha_j W_j)$$

$$I_R = \frac{I_i}{I_{Compton}}$$

$\alpha_j$  : theoretical alpha of element j

$W_j$  : weight fraction of element j

$I_i$  : intensity of element i line

$I_{Compton}$  : intensity of Pd-Ka Compton line

$A, B, C$  : constants

Matrix correction coefficients (alpha) applied to the calibrations are theoretically calculated by the FP software. The theoretical alphas for Fe, Cu, Zn, As and Pb calibration are calculated by taking the Compton scattering internal standard into consideration by the Quant Scattering FP function integrated in the software.

## Results

Calibration accuracies for all the components measured in this study are listed in Table 1.

The accuracy is calculated by the following formula:

$$Accuracy = \sqrt{\frac{\sum_i (C_i - \hat{C}_i)^2}{n-m}}$$

$C_i$ : calculated value of standard sample

$\hat{C}_i$ : reference value of standard sample

n : number of standard samples.

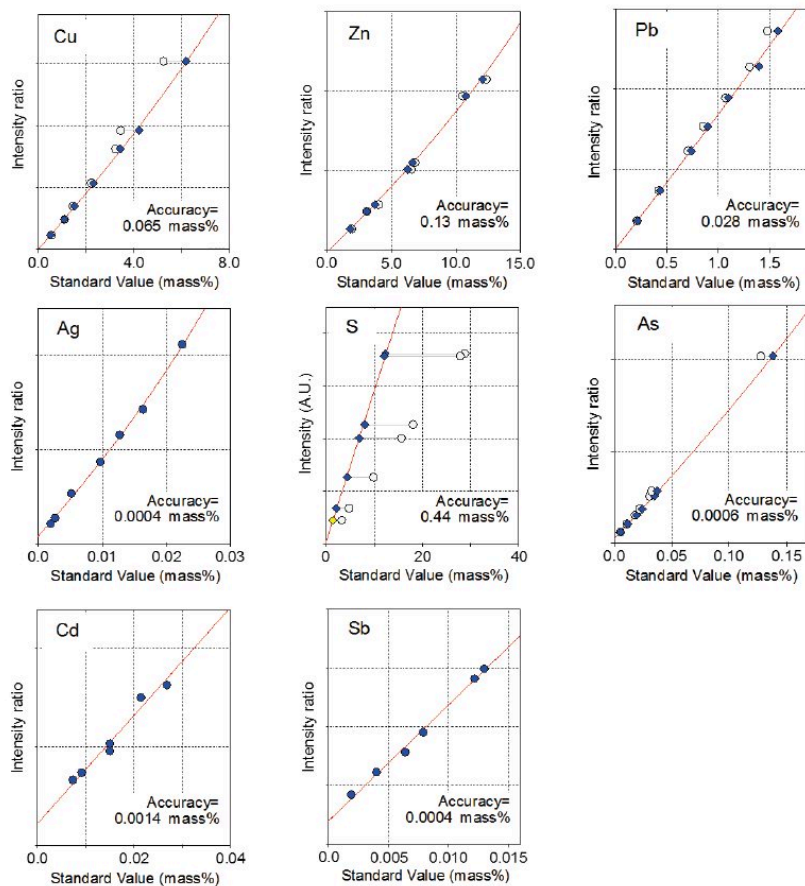
m: degree of freedom (linear 2, quad. 3)

**Table 1:** Accuracy of the calibration curve (unit : mass%)

Element	Concentration range	Typical accuracy of calibration
Na*	0.42 - 2.3	0.028
Mg*	0.76 - 2.9	0.082
Al*	1.7 - 6.5	0.098
Si*	7.4 - 24	0.47
P*	0.025 - 0.078	0.0034
S	3.3 - 29	0.44
K*	0.20 - 1.4	0.010
Ca*	1.8 - 5.8	0.17
Ti	0.22 - 1.0	0.020
Cr	0.0054 - 0.015	0.0020
Mn	0.064 - 0.13	0.0015
Fe	7.1 - 25	0.55
Cu	0.53 - 5.2	0.065
Zn	1.9 - 12	0.13
As	0.0052 - 0.12	0.0006
Ag	0.0019 - 0.023	0.0004
Cd	0.0073 - 0.027	0.0014
Sb	0.0019 - 0.013	0.0004
Pb	0.21 - 1.5	0.028

\*: Reference values were used for calibration

Calibration curves for selected eight components, which are Cu, Zn, Pb, Ag, S, As, Cd and Sb, are shown in Figure 1.



**Figure 1:** Calibration curves of representative components in polymetallic sulfide ore. Cu, Zn, As and Pb are calibrated with scattering X-ray internal standard and theoretical alpha correction. Blue: Corrected (certified), Yellow: Corrected (uncertified), Circle: Uncorrected

## Conclusions

X-ray fluorescence spectrometry is rapid, precise and accurate method that meets the requirements of mining industry. Owing to its high spectral resolution, the wavelength dispersive optics is especially suited for the detection of multi metal elements in polymetallic ores. Supermini200 is a compact WD system but is a highly versatile spectrometer for ore sample analysis. In this application note it is demonstrated that improved correction method using the "Quant Scatter FP" function, which is a combination of Compton scattering ratio method and theoretical alpha correction, is practically applicable for analysis of polymetallic sulfide ores.

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## Related products



### Supermini200

Benchtop tube below sequential WDXRF spectrometer analyzes O through U in solids, liquids and powders