Study and application of X radiation sampling technique*

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Abstract The physical bases of a X radiation sampling technique are investigated. Three technical problems of unevenness, matrix and heterogeneous mineralization effects, are considered and successfully resolved. This new technique was applied to three sites of Au, Sn and Cu deposits (representing higher, medial and lower atomic number minerals) in different exploration stages in China and satisfactory results are obtained.
Keywords X radiation sampling, Gold deposit, Copper deposit, Tin deposit

1 Introduction

Traditionally, determination of element concentrations in rocks, soil and stream sediments in exploration of mineral resources is based on sampling techniques. Analytical procedures usually include sampling; grinding the bulk sample into powder sample with less than 76 μm in diameter; analysing the element concentration of 3~5 g powder sample from ground sample. A common character of this traditional sampling is that the random relative errors produced in sampling, grinding and abstracting are usually far larger than in chemical and physical analyses. The turn-around time of determination of the elemental concentration of a sample is typically one week for mines or more. Energy-disperse X-ray fluorescence (EDXRF) analysis offers several advantages over traditional samplings. One can directly measure in-situ with a portable X-ray fluorescent spectrometer. The turn-around time per spot of interest is only several minutes. The technique was defined as X radiation sampling.[1]

2 Theory

The physical basis of X radiation sampling is "Moseley law" i.e. the energy of the characteristic X-rays from the atom of an element is directly proportional to the square of the atomic number. So, when samples (e.g. powder, rock or soil) are irradiated with a radioactive isotope source, the types of elements can be quantitatively determined by measuring the energy of the characteristic X-rays from the samples. Quantitatively determining the concentration of an aimed element in samples is based on the following formula[2]:

$$I_x = \frac{KI_0}{\mu_0 + \mu_x} \cdot F(H) \cdot C_A \qquad (1)$$

where I_x is the intensity of the characteristic X-rays emitted from an aimed element; I_0 the intensity of the (X or γ) rays emitted from an isotope source; C_A the concentration of the aimed element in samples; μ_0 and μ_x the mass attenuation coefficients of source (X or γ) rays and characteristic X-rays, respectively; $F(H)$ the unevenness factor relating to the unevenness of measured surface; K a constant. Main factors influencing Eq.(1) will be elucidated in the following.

3 Method

3.1 Unevenness effect

Unevenness effect in X radiation sampling is inevitable. According to the character of unevenness factor ($F(H)$), the following methods can be adopted to minimize unevenness effect: taking the ratio of characteristic X-rays to scattered radiation as a basic parameter, which can effectively reduce the errors caused by the changes of source-sample distance (see Fig.1); the formula of determining an optimal source-sample distance is:

$$\varepsilon_j = \frac{\Sigma_{i=1}^{n}(R(H_j + \triangle H_i) - R(H_j))}{n \cdot R(H_j)} \qquad (2)$$

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where ε_j is the average of the absolute relative errors of the ratio of the elemental characteristic to scattered radiation; R the ratio of the elemental characteristic to scattered radiation, H_j source-sample distance (10, 15, 20, 25, 30, 35, 40 mm) in model experiments, for any given H_j in model experiments, $\triangle H_i$ can be -20, -15, -10, -5, 0, 5, 10, 15, 20 mm and 10 mm< $H_j+\triangle H_i$ < 40 mm; n the number of $\triangle H_i$. Obviously, the minimum of ε_j gives the smallest relative error of the ratio of the characteristic to scattered radiation caused by the changes of source-sample distance. H_j producing the minimum of ε_j has been defined as the optimal source-sample distance(H_0). By experiment on simulating rock walls, the H_0 values are determined as 23 mm for Sn (Fig.2), 16 mm for Pb-Zn and 20 mm for Mo.

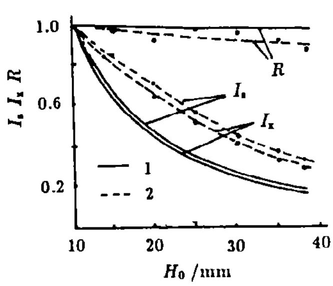


Fig.1 Ix, Is and R vs H
I_s—Scattered radiation intensity; 1—Theoretical; 2—Experimental

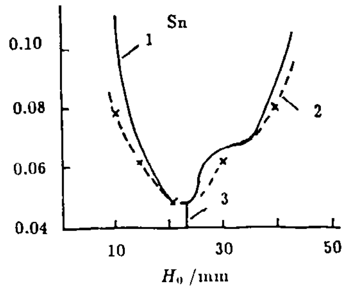


Fig.2 The experiment curve of the optimum source-sample distance for tin deposit
1—Theoretical; 2—Experimental; 3—Optimum source-sample distance

3.2 Heterogeneous mineralization effect

Through a series of experiments, we found that the process of X radiation sampling can be described as normal distribution. Based on this theory, the best way to minimize the heterogeneous mineralization effect is to choose an optimal measuring net on sampling surface of interest, and the net should be as small as possible. The best net is $d \cdot d$, where d is the diameter of effective detection area of a probe in cm.

3.3 Matrix effect

Under the condition of effectively overcoming unevenness effect, we established a model for overcoming matrix effect. That is

$$C_i = \varepsilon_i + \Sigma A_i R_i + \Sigma B_{i,j} R_i R_j + \Sigma C_{i,j,k} R_i R_j R_k + \Sigma D_{i,j,k,h} R_i R_j R_k R_h + \cdots \quad (3)$$

where C_i is the concentration of the i-th element in the samples; R_i the ratio of the i-th element characteristic to the scattered radiation; R_j, R_k, R_h the ratios of the j-th, k-th, h-th element characteristic considered as main interfering elements in the samples to scattered radiation, respectively; ε_i the i-th element zero intercept; A_i, $B_{i,j}$, $C_{i,j,k}$, $D_{i,j,k,h}$ regressive coefficients.

To judge which elements are main interfering elements in samples, the following judgement expression is given

$$T_j = (\mu_{xj} - \mu_{sj}) \cdot \triangle C_j \quad (4)$$

where T_j is the judgement of the j-th elements; $\triangle C_j$ the range of the j-th element concentration in samples; μ_{xj} and μ_{sj} the mass attenuation coefficient of the j-th element for characteristic X-rays and scattered rays of previous radiation, respectively.

4 Instrument

A microprocessor-based portable X-ray flourescence analyser was designed and manufactured at our university. It consists of a probe and a center unit, weighing 6 kg. The probe consists of a X-ray detector and an alternative isotope sources, ^{238}Pu and ^{241}Am; the ^{238}Pu source is used to stimulate lower atomic number element K-shell and higher atomic number element L-shell characteristic X-rays, and ^{241}Am

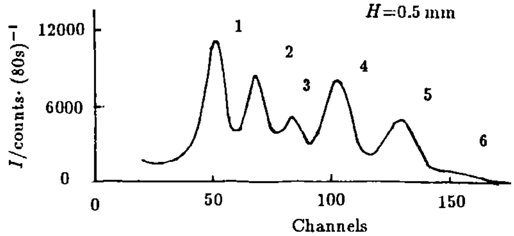


Fig.3 Experimental spectrum of Pb-Zn sample irradiated by ^{238}Pu source with a proportional probe

1. FeK_α; 2. ZnK_α; 3. PbL_α; 4. PbL_β+scattered peak from UL_α; 5. Scattered peak from UL_β; 6. Scattered peak from UL_γ

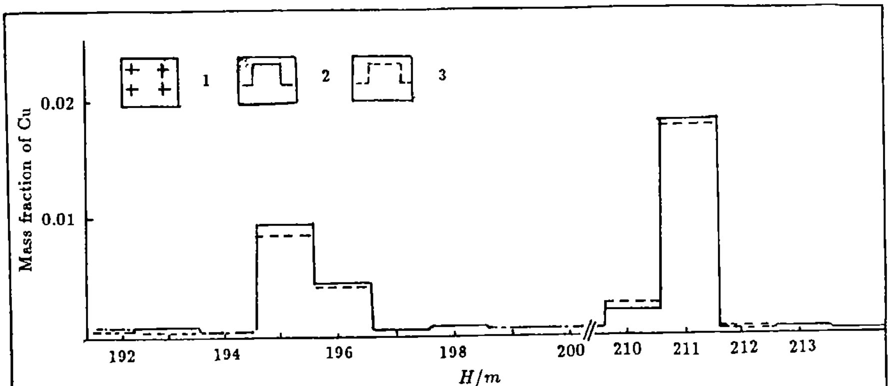
source is for stimulation of medial atomic number element K-shell characteristic X-rays and for measurement of scattered γ rays. Its differential spectrum is shown in Fig.3. The centre unit includes a preamplifier, a linear pulse amplifier and analog–to–digital convertor that produces a 512 channel spectrum for storage in the microprocessor unit with automatic peak searching and curve smoothing function. Its output is a printer.

5 Applications

Since X radiation sampling technique is a non-destructive method of in-situ determining element concentration on the surface of original state rocks, ores, soil or stream sediment. It has been widely applied to mineral reconnaissance survey, mineral exploration and mining. We have successfully applied this technique to near thirty types of mineral deposits, such as Pb, Zn, Cu, Mo, Ba, Sn, Sb, Ag and Au, and obtained vast economical and social benefits.

5.1 Stage of reconnaissance survey

The working area is situated in the south of a large-scale gold deposit. No evidences



I	0.08	0.06	0.02	0.94	0.44	0.02	0.06	0.02	0.02	0.22	1.82	0.02	0.06	0.04
II	0.053	0.018	0.039	0.85	0.42	0.039	0.057	0.048	0.026	0.27	1.86	0.087	0.051	0.035
III	192	193	194	195	196	197	198	199	200	210	211	212	213	214
IV	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +	+ + + +

Fig.4 Isogram of X radiation sampling for surveying gold

1. Granite; 2. The curve of Cu grade determined by X radiation sampling; 3. The curve of Cu grade determined by channel-chemical sampling; I. Cu grade by X radiation sampling; II. Cu grade by channel -chemical sampling; III. The number of samples; IV. Lithology

showed that there was possibility of buried gold veins in this area before X radiation sampling technique was applied. According to strong correlation of As and gold, we determined the distribution of As in soil with X-ray fluorescence analyzer and successfully indicated gold veins (see Fig.4), which has been proved by drill holes and tunnels later. This work gave a precious information about gold veins and took less than 20 d.

5.2 Stage of exploration

In this stage, we can easily use X radiation sampling to determine the border of ore bodies and to estimate ore grades. Fig.5 is an example of applying the technique to determine Cu ore bodies and its grades in an exploratory trench. Two ore bodies were divided and exactly corresponded with those by traditional channel-chemical sampling methods

5.3 Stage of mining

Table 1 is the statistical results of X radiation sampling applied to Dachang tin mine in Guangxi province. In this mine, X radiation sampling technique has partly replaced traditional channel-chemical sampling method for determining tin ore grade and guiding mining. It is important that there is no pollution to environment and no harm to operators for X radiation sampling.

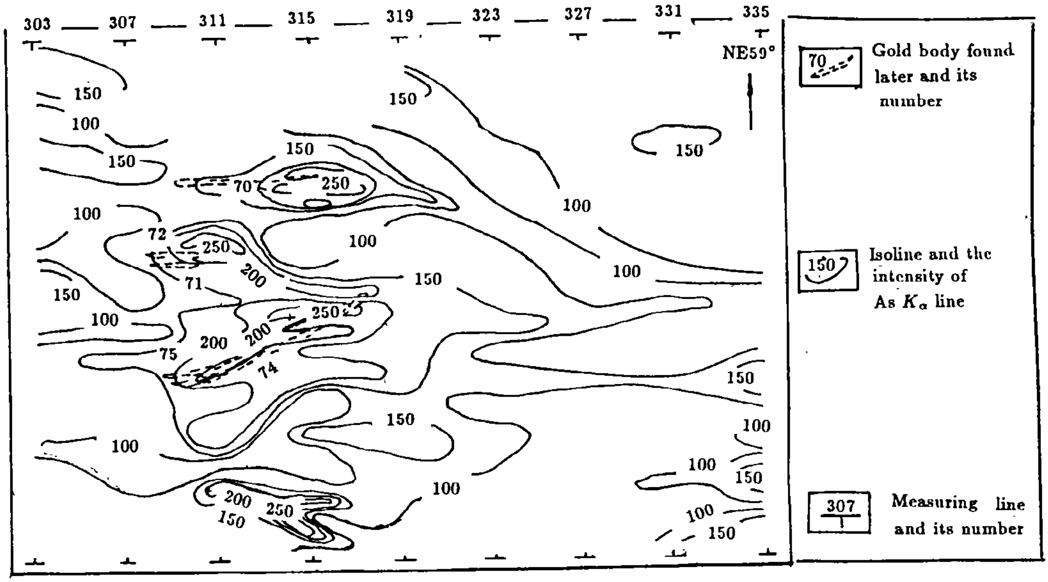


Fig.5 Composite profile of X radiation sampling on an exploration trench wall for determining Cu ore bodies

Table 1 Statistical results of X radiation sampling

Methods	Mass fraction of tin	Number of samples	Relative error	Cost of each sample/RMB	Turn-around time
X radiation sampling	9.710×10^{-3}	105	4.44×10^{-3}	0.7	1 h
Channel-chemical sampling	9.068×10^{-3}	88	–	14	≥7 d

6 Conclusion

X radiation sampling technique can be used to determine element concentration or ore grades on rock surface and in soil. So, it can be applied to outline mineralization anomaly in reconnaissance survey, to block out ore bodies in mineral exploration and to guide mining and estimate ore reserves in mining. Additionally, this technique is a non-destructive methods of directly measuring elements in ores under their original state and can prevent the detriment of powdery stuff to health and lighten the labour intensity of workers.

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