ESTIMATION OF APPLICABILITY OF SCATTERED RADIATION FOR XRF

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The applicability of coherently and incoherently scattered radiation was investigated on the example of quantifying the Nb content in wolframite. The calculated and experimental intensities of NbK_α-line for binary and multicomponent mixtures of Nb_2O_5 with various matrices have been compared. The major oxides MgO, SiO_2, CaO, Fe_2O_3, and a typical component of tungsten ore $(Fe, Mn)WO_3$ were used as the main matrices. Measurements were carried out using the energy dispersive X-ray spectrometer with Si(Li)-detector and radioactive isotope source [109]Cd. The analysis of data shows that when using the ratio of the intensities of coherently and incoherently scattered X-radiation of the excitation source the dependence of the analytical line intensity of niobium on the content of W in the samples is taken into account. The experimental results confirm the conclusions of the theoretical consideration. This technique was successfully used to study wolframite samples from the deposits of the Eastern and Western Mongolia.

A procedure for the determination of Cu, Mo and Fe concentrations in ores, copper and molybdenum concentrates, and enrichment waste was developed to control the process of enrichment at the mining company "Erdenet", Mongolia. This procedure was based on using regression equations. The form of regression equations was chosen based on the theoretically calculated intensities. The comparison results of measurements using the newly developed technique and the standard-background method are presented.

Keywords: X-ray spectral fluorescence analysis of ores and enrichment products, method of standard-background, theoretical simulation of matrix effects.

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1. INTRODUCTION

The study of the distribution of impurity elements in the ore minerals allows to reveal a number of regularities of mineralization. For wolframite deposits tantalum, niobium, and scandium are of particular interest from this point of view. It was found that in some cases the wolframites can be an additional source of niobium, tantalum, and scandium [1, 2]. It was also found that the content of these impurity elements and their ratios can inform about the conditions of formation of wolframite deposits. On the other hand, the chemical methods for the separation and determination of Ta and Nb in wolframites are laborious.

X-ray fluorescence method is now widely used for the determination of many trace elements, as well high content elements in rocks. However, this method as the most other methods is under the influence of interfering elements on the accuracy when determining an analyte. There are some ways to eliminate the matrix effects [3, 4]. In spite of several advantages, none of these methods is universal.

We have investigated the applicability of different versions of the background standard method to determine contents of some elements in the geological samples, in particular, niobium in wolframites and molybdenum in Mo-concentrates.

2. RESULTS

Determination of Nb in wolframite

The application of standard-background method allows to significantly reduce the influence of matrix
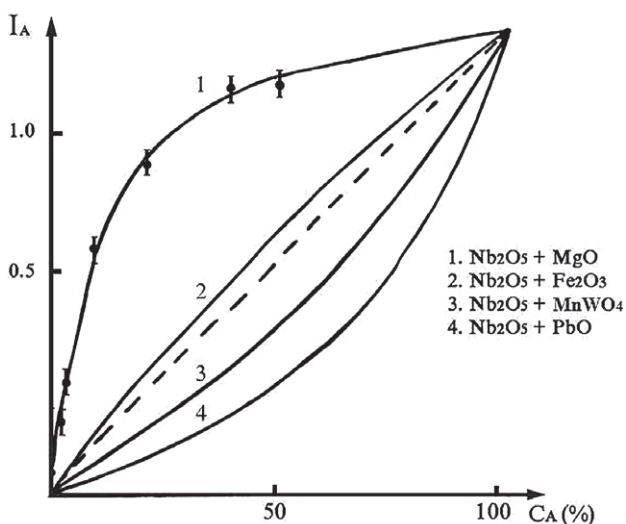

Fig. 1. Dependence of NbK_α intensities on Nb concentration in the different matrices: (1) curve for the case where the absorption of the matrix is less than the absorption of Nb, (2) and (3) curves correspond to the similar absorption of the matrix and Nb, (4) curve for the case where the absorption of the matrix is greater than Nb

effects on analytical results [3, 5-7]. To reduce or eliminate the influence of matrix effects on results of X-ray fluorescence analysis (**XRF**) the applicability of coherently and incoherently scattered radiation was investigated on the example of quantifying the Nb content in wolframite. We have compared the calculated and experimental intensities of NbK_α-line (16.7 keV) for binary and multicomponent mixtures of Nb_2O_5 with various matrices. The major oxides MgO, SiO_2, CaO, Fe_2O_3, and a typical component of tungsten ore (Fe, Mn)WO_3 were used as the main matrices.

The intensities of radiation incoherently scattered from the sample were calculated from the eq. (1):

$$I_{incoh} = K_p I_0 + \frac{\sigma_{incoh}}{\mu_1 + n \cdot \mu_{m\sigma_{incoh}}}, \qquad (1)$$

the intensities of coherently scattered radiation were calculated according to the formula:

$$I_{coh} = K_p I_0 \frac{\sigma_{coh}}{2\mu_1}, \qquad (2)$$

where I_0 is intensity of primary radiation, σ_{incoh} and σ_{coh} are mass scattering coefficients of radiation coherently and incoherently scattered from sample, respectively, E_0 is energy of primary radiation, μ_{m1} and μ_{mincoh} are mass absorption coefficients of incident radiation and radiation scattered from sample, respectively, K_p is constant. The mass absorption coefficients were calculated using the data from [8], and mass scattering coefficients were taken from [9].

The intensities of radiation source coherently and incoherently scattered from sample, and their ratio were taken as analytical parameters. The calculated values of the fluorescence intensities I_A, as well I_{coh} and I_{incoh} for $AgK\alpha$ line and their ratios for binary samples are shown in Table 1. You can see from the considered example of the obtained data that alterations of I_{incoh} and I_A are similar and alteration of I_{coh} is much less.

The mass absorption coefficients of the considering oxides for $NbK\alpha$ – line have changed in the wide range. As a result, the dependence between intensity and concentration is significantly different for separate binary

Table 1

Calculated value of I_A, I_{coh}, I_{incoh} and their ratio of NbK_α-line for the binary samples

Nb_2O_5, %	Matrix	I_A	I_{coh}	I_{incoh}	I_A / I_{coh}	I_A / I_{incoh}	$R_A = I_{incoh} / I_{coh}$	I_A / R_A
0.1	MgO	1.04	5.91	31.8	0.176	0.033	5.38	0.19
	SiO_2	0.84	5.38	26.5	0.16	0.032	4.92	0.17
	CaO	0.24	2.67	6.62	0.09	0.036	2.48	0.10
	Fe_2O_3	0.127	1.98	3.32	0.06	0.038	1.68	0.08
	$MnWO_3$	0.052	2.41	0.99	0.022	0.053	0.41	0.13
10	MgO	53.6	2.32	9.30	23.1	5.76	4.01	13.4
	SiO_2	48.4	2.34	8.82	20.7	5.49	3.77	12.8
	CaO	21.1	2.04	4.55	10.3	4.63	2.23	9.4
	Fe_2O_3	12.4	1.73	2.76	7.14	4.47	1.60	7.7
	$MnWO_3$	5.5	2.26	1.00	2.42	5.47	0.44	12.4

Table 2

Calculated and experimental values of I_{Nb}, I_{coh}, I_{incoh} and their ratios for NbK_α-line in multicomponent samples

Oxide concentrations, %					I_{Nb}		I_{incoh}		I_{coh}		$R = I_i / I_{coh}$		I_{Nb} / R		I_{Nb} / I_{incoh}	
Nb_2O_5	MnO_2	Fe_2O_3	WO_3	SiO_2	1	2	1	2	1	2	1	2	1	2	1	2
1.0	15.0	8.0	30	46	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000	1.000
			40	36	0.798	0.791	0.763	0.786	0.970	0.989	0.787	0.795	1.014	0.995	1.049	1.006
			50	26	0.644	0.691	0.604	0.681	0.950	0.975	0.636	0.699	1.013	0.989	1.066	1.015
			60	16	0.573	0.545	0.492	0.530	0.937	0.967	0.526	0.548	1.089	0.995	1.165	1.028
			70	6	0.497	0.485	0.408	0.467	0.927	0.957	0.441	0.488	1.127	0.994	1.218	1.039

Comment: 1 – calculated and 2 – experimental values.

systems. The relationship between $NbK\alpha$ intensity and Nb concentration is shown in Fig. 1. The solid line corresponds to calculated data and points are experimental values. Measurements were carried out using the energy dispersive X-ray spectrometer with Si(Li) detector and radioactive isotope source Cd-109 during 200 s. The Figure shows good agreement between calculated and experimental data.

Table 2 presents the calculated and experimental data for multicomponent mixture samples which simulate the composition of the tungsten ore. The samples studied contain Nb_2O_5 (1 %), MnO (15 %), Fe_2O_3 (8 %), and the tungsten concentration changes depending on the content of SiO_2.

As Table 2 shows, the specific intensity of the analytical line of Nb greatly varies with change in the tungsten concentration at a constant total concentration of Mn and Fe. When changing the tungsten concentration from 30 to 70 % the niobium intensity varies more than twice. As can be seen from Table 1 the account for the matrix effects using the coherently and incoherently scattered radiation allows to significantly reduce the uncertainty in determining the niobium concentration. The analysis of data presented in Table 2 shows that the ratio of the intensities of coherently and incoherently scattered X-radiation of the excitation source takes well into account the dependence of the intensity of the analytical niobium line on the content of W in the samples. The authors of a number of papers have attempted to explain the applicability of this approach in different analytical situations. A review of these papers can be found in [10]. The experimental results generally confirm the conclusions of the theoretical consideration.

The results of calculation and experiment showed:

The intensity of coherently scattered radiation is less sensitive to the change in a chemical composition of sample than the fluorescence intensity. In case of significant changes in the chemical composition of sample the application of ratio of coherently scattered radiation and the intensity of fluorescence does not provide the acceptable analytical precision when determining Nb concentration;

However, the intensity of incoherently scattered radiation changes similarly to the fluorescence radiation for elements whose absorption edge energies do not exceed the energy peaks of analytical lines. The difference between normalized intensities significantly decreases

when sample consists of elements with low Z and elements with high Z for different matrices;

The use of dividing the fluorescence intensities by the ratio of coherently and incoherently scattered radiation intensities $R = I_{incoh} / I_{coh}$ provides the satisfactory accuracy of analysis. In this case the concentration has to be calculated by simple formula the intensities of coherently scattered radiation were calculated according to the formula:

$$C_A = \frac{I_A}{I_{st}} \cdot \frac{R_{st}}{R_A} \cdot C_{st},$$ (3)

where I_A and I_{st} are intensities of analytical lines for the determining elements in samples analysed and certified reference materials, respectively; R_{st} and R_A are ratios of coherently and incoherently scattered radiation for analysed samples and certified reference materials; C_{st} is concentration of element in CRM.

The study has shown that an uncertainty of Nb determination in wolframite samples by this method is less than 5 %. In order to obtain the better precision of the determination of Nb concentration it is necessary to take account of the dependence of analytical parameters on the concentration of W in sample.

The experimental test was performed for the set of wolframite samples of known chemical composition and certified reference materials SVT [11]. The concentration of Nb determined by standard-background method (ratio of coherently and incoherently scattered radiation) is shown in Table 3. XRF results give good agreement with certified data, although the chemical composition of the certified reference materials SVT varies within the wide

Table 3

Results of XRF determination of Nb_2O_5 in standard reference materials, %

CRM	Certified value*	Concentration obtained by formula (3)
SVT -5	0.0134 ± 0.0005	0.0130 ± 0.0007
SVT -6	0.110 ± 0.005	0.108 ± 0.004
SVT -8	1.06 ± 0.06	1.00 ± 0.0052
SVT -12	0.100 ± 0.004	0.100 ± 0.004
SVT -13	0.013 ± 0.0005	0.014 ± 0.001
SVT -7	0.0070 ± 0.0005	0.0078 ± 0.0007
SVT -9	2.03 ± 0.04	1.95 ± 0.06

Comment: * – the confidence intervals are calculated using the conventional formula from [13].

Table 4

The variation in chemical composition of the industry products

Product	Concentration, %			
	Cu	Mo	Fe	Density of pulp (flow)
Ore	0.3-1.7	0.006-0.08	1-6	18-35
Waste	0.04-0.3	0.002-0.04	0.5-5	7-35
Concentrate of Cu	20-40	0.04-0.8	10-40	15-50
Concentrate of Mo	0.4-3.0	40-55	0.5-5	-
Joint concentrate	5-18	0.05-0.5	15-35	15-40

range and is very different from the composition of the certified reference materials used. We have successfully used this technique to study wolframite samples from the deposits of the Eastern and Western Mongolia.

Determination of Mo in molybdenum concentrate

To develop new XRF techniques for any substance it is necessary to obtain the information about the ranges of the chemical sample compositions and choose a certain means to eliminate their influence on the analysis results. In addition, a mathematical correction could be used for accounting matrix effects. Nowadays this approach is widely used in practice.

The calculation of matrix effects is the important step in the development of the X-ray fluorescence methods. To control the process of enrichment at the mining company "Erdenet", Mongolia, it is necessary to determine Cu, Mo and Fe. The effect of chemical composition on the intensities of the CuK_α-, MoK_α-, and FeK_α-lines was studied for ores, copper and molybdenum concentrates, and enrichment waste [12].

As Table 4 shows, concentrations of elements vary over a wide range for each product. In particular, there are the most significant changes in the Cu contents in copper concentrate and Mo in molybdenum concentrate (up to 10 % abs. or more). All products contain iron (from 1 to 40 % abs.). The concentration of Mo in tailing

dump is close to the detection limit ($n \cdot 0.001$ % abs). The detection limit was calculated according to the common used formula from [5]. As a result, the matrix effects can occur in determining Cu, Mo and Fe in ore and Cu and Mo in concentrates.

The calculation of matrix effects was performed using the theoretical intensity. These effects were estimated using the intensities of analytical lines excited by the polychromatic primary beam by the formula, which takes into account the effects of enhancement and absorption. The excitation effect of the third order has not taken into account because its contribution is negligible for the considered case.

For these calculations the contents of Cu, Mo and Fe were taken according to the chemical analysis, and concentrations of S and other elements were given based on contents of reference samples of the same type of products. The following conditions of excitation were used for the calculations: potential is 40 kV, Rh anode, thickness of Be window of X-ray tube is 200 microns and the angles of incidence of the primary and take-off of fluorescence radiation are 90° and 45°, respectively.

The absorption effects were mostly occurred when analysing enrichment waste and ore liquid mix. The change in the interfering element concentrations by 1 % in these products leads to the significant change in the intensity of analytical lines of the detemining elements. Table 5 presents the values of the influence coefficients of major rock-forming elements, as well S, Cu and Mo for FeK_α, CuK_α, and MoK_α analytical lines. The coefficients characterize the change in fluorescence intensity (% rel.) for the particular analytical line when changing the concentration of the influencing element by 1 % abs. [5]. This change was due to the change in SiO$_2$ content in the sample. The calculation is performed for the reference samples of Cu concentrate and Cu-Mo ores.

The contents of S, TiO$_2$, Fe$_2$O$_3$, and Mo have a great influence on the intensity of CuK_α-line. For example, in the ore with increasing the CaO content by 1 % the intensity of CuK_α decreases by 1.78 %, while at a similar change in the concentration of Fe$_2$O$_3$ the intensity of CuK_α decreases by 3.49 %. For K_α of Mo and Fe the same situation takes place.

To estimate the effect of the chemical composition of the samples on the XRF results using the external standard method we have plotted the theoretically calculated

Table 5

Influence factors of major rock-forming elements on intensities of analytical lines of the determining elements

Influence component	Copper concentrate			Ore		
	Fe	Cu	Mo	Fe	Cu	Mo
Al$_2$O$_3$	0.07	0.04	0.01	0.11	0.09	0.09
S	-0.9	-0.44	-0.22	-1.29	-1.78	-1.05
CaO	-1.34	-0.7	-0.38	-1.97	-1.78	-1.76
TiO$_2$	-1.31	-0.7	-0.4	-2.01	-1.86	-1.96
Fe$_2$O$_3$	-	-1.4	-0.8	-	-3.49	-3.86
Cu	0	-	-1.68	0.56	-	-7.45
Mo	-1.58	-0.59	-	-1.86	-1.13	-

intensities of analytical lines I_{2i} versus the element concentration in the samples (Fig. 2). These dependencies were linear, except for Mo (Fig. 2, *d*). However, the ratio of the measured Mo intensity to the intensity of scattered radiation has a linear dependence on the Mo concentration in molybdenum concentrate (Fig. 3). The dispersion of points around the calibration line is due to the influence of chemical composition of sample matrices on the fluorescence intensity and characterizes the uncertainty in results of the analysis when using the method of external standard. The value of influencing factors depends on excitation conditions (anode material, Be window thickness of X-ray tube and geometry of spectrometer) and the chemical composition of the sample analysed.

When significant varying the sample composition (if there are several CRMs) the concentration of the element determined can be obtained with the sufficient accuracy using regression equations. The form of regression equations was chosen based on the theoretically calculated intensities.

To select optimal regression equations and to determine calibration characteristics, experimental data of intensities obtained from measurements of over the twenty samples of each product were used. The following equations were considered for all products:

$$C_i = a_{0i} + a_{1i}R_i + R_i \sum_{j=4}^{6} a_{ji}R_j \quad , \quad (4)$$

$$C_i = a_{0i} + \sum_{j=1}^{n} a_{ji}R_j \quad \text{and} \quad (5)$$

$$C_i = a_{0i} + a_{1i}R_i \, , \quad (6)$$

where $R_i = J_i \dfrac{J_{2i}^0}{J_{2i}}$ and $R_j = J_j \dfrac{J_{2j}^0}{J_{2j}}$ are corrected intensities of analysed and intefering elements, respectively. Samples with known elemental contents are used for the determination of equation coefficients. The analytical results of specialized chemical laboratory at the Erdenet Corporation were used too. The results calculated from these equations show that the residual uncertainties do not exceed the acceptable values. The best accuracy is provided by equation (4) and (5) when contents of elements in analyzed samples change within a wide range. The application of equation (6) also gives results with good accuracy if the chemical composition varies over relatively narrow ranges.

Table 6 presents the comparative results of measurements using the new developed technique,
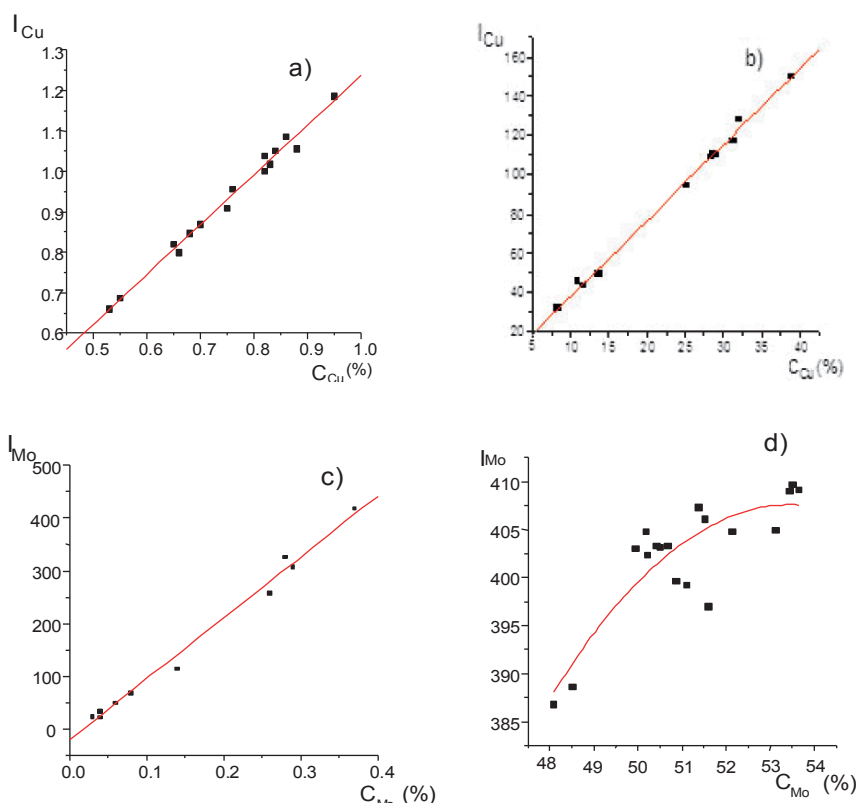

Fig. 2. Dependence of intensities of analytical lines on the concentrations of determining elements: Cu in ore; b) Cu in copper concentrate; c) Mo in ore; d) Mo in molybdenum concentrate
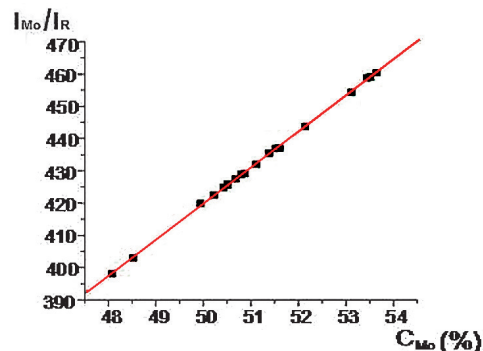

Fig. 3. Dependence of corrected analytical line intensities of molybdenum on its concentrations

calculations by equation (5) and the method of standard-background. Hence it can be seen that the residual uncertainty in determination of those contents by selected equations do not exceed the tolerances.

3. SUMMARY

The applicability of intensities of coherently and incoherently scattered radiation from the source as an analytical parameter, as well the ratios of these intensities are considered. It is shown that the ratio of the intensities of the coherent and Compton scattered radiation takes into fuller account the influence of the chemical composition of samples in a number of cases. The combined method is considered for determining element contents in samples with a wide range of concentrations, based on the application of the standard background

Table 6

Comparative results of measurements used developed new technique and a method of standard-background, %

Ele-ment	Copper concentrate			Molibdenum concentrate			Ore			Tailings dump		
	A	B	tol	A	B	tol	A	B	tol	A	B	tol
Cu	0.44	0.71	0.6	0.065	0.065	0.12	0.032	0.051	0.04	0.009	0.011	0.02
Mo	0.017	0 0.015	0.02	0.57	0.85	0.7	0.002	0.003	0.003	0.002	0.002	0.002
Fe	0.70	0.84	0.7	0.29	0.29	0.3	0.11	0.26	0.3	0.15	0.17	0.2

Comments: A – new method, B – old method, tol – tolerances.

method and the regression equations. The conducted investigations have allowed to develop a method for the determination of niobium in wolframites, as well molybdenum, copper, and iron in the industrial products, namely products of ore processing (when determining contents of low *Z* elements in the matrices with high *Z* or high contents of elements).

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