

NEUTRON ACTIVATION DETERMINATION OF GOLD IN TECHNOGENIC RAW MATERIALS WITH DIFFERENT MINERAL COMPOSITION

by

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The methods used to determine the gold content in the technogenic objects of gold mining were analyzed regarding their non-homogeneity and complexity of chemical and mineral compositions. A possible application of the neutron activation analysis with the use of the californium source of neutrons for determining the content of fine-grained and extra-fine-grained gold in the technogenic objects, including the bottom-ash waste of energy providers, is considered. It was demonstrated that the chemical composition of the sample affects the neutron flux distribution in the sample, which can essentially distort the results of the neutron activation analysis. In order to eliminate possible systematic errors investigations of the effect of the sample mineral composition on the results of the gold determination using the neutron activation analysis were carried out. Namely, a large mass of rock (3-5 kg) was loaded into an activation zone using four matrix types such as silicate, carbon-containing, iron-containing, and titanium magnetite. It was shown that there were no significant difference between the dispersal of the fluxes of thermal and resonance neutrons emitted from ²⁵²Cf during activation of the gold-containing technogenic samples with different mineral compositions.

Key words: neutron activation analysis, fine-grained gold, technogenic object, californium, activation zone, thermal neutron flux

INTRODUCTION

With a decrease in the number of rich gold-bearing placers containing coarse free gold, an interest in processing of dumps of the old deposits [1, 2] as well as in other technogenic sources including the bottom-ash waste of the energy providers grows steadily worldwide including Russia. A considerable content of gold is present as fine-grained and extra-fine-grained fractions in technogenic objects such as concentration tailings, silt-detention basins, partially overburden rocks, mines technical facilities, barriers, and curtain walls. Approximately 75 % of all gold reserves are associated with grain sizes of the order of 0.9-0.001 mm [3]. The wastes of energy providers are of the utmost interest as they increase the environmental load and generate a need to involve such sources in the processing with recovery of valuable components, including fine-grained gold. Despite the fact that the fine-grained gold is unevenly distributed

in slag its content can reach 0.015-1.76 g/t. Such elevated gold content (0.14-0.546 g/t) [4] is present in the products of magnetic fraction.

A high content of the fine fraction in the technogenic objects of gold mining is because of a less-efficient mechanical cleaning of traditional methods. At present, the maximum size of efficiently extracted particles of gold is 0.5-0.25 mm; gold with grain size of <0.1 mm is practically not extracted on sluices, whereas that with grain size of <0.03 mm is not detected at all by mechanical cleaning [5].

Considering the special features of technogenic objects of gold mining, characterized by both high non-homogeneity of gold content [6] and non-homogeneity and complexity of chemical and mineral composition [7], the best method for determining the gold content should be selected.

The assay test is one of the most commonly used chemical methods for determining gold content. This method is based on the capability of molten metallic lead to actively dissolve the precious metals thereby producing the low-melting alloys [8]. A cupellation

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process was employed to separate precious metals from lead. Although this method has high sensitivity (up to 0.01 g/t) and proven procedure, it has not always proved optimal when handling objects such as fine-grained gold.

When determining the content of the fine-grained and extra-fine-grained gold in cases where the small particles of metal are closely associated with minerals having different compositions, the classical methods of gold analysis, in which the acid or assay decomposition is assumed, can result in systematic errors because of incomplete metal recovery [9].

Khanchuk investigated the significant effect of such systematic errors on the results of gold determination [10]. This study showed that methods determining the acid or assay decomposition of samples yield low contents of precious metals, whereas nondestructive methods provide values that are higher by an order of magnitude.

At present, studies are performed aiming at both improving the methods of assay test of gold at reduced energy consumption and labor intensity [11, 12] and integrating cupellation test with other physical analysis techniques, such as atomic adsorption [13]. However, data of the modified assay test of gold do not resolve the incomplete recovery of fine-grained gold, which results in a systematic error in the initial stage.

Atomic adsorption spectrometry analysis has wide-spread application in the determination of precious metals in the tails remaining after leaching of ores and ore concentrates [11, 14]. A physical base of this method is radiation transmission within the range of 190-850 nm through a layer of atomic vapors of the sample. As a result of absorption of light quanta, the atoms pass into the excited energy states. With these transitions in the atomic spectra the resonance lines, characteristic of a given element, comply and the optical density serves as a measure of element concentration.

In general, this method is unique because of its high absolute and relative sensitivities, simplicity, high selectivity, and little influence of the sample composition upon the analysis results. However, the major problem is the need of sample transfer into solution, which complicates the preliminary sample preparation and reduces the speed of analysis. In order to eliminate the analysis errors related to insufficient sample representativity when working with the technogenic objects of gold mining, which are non-uniform in respect of both gold content and mineral composition, it is necessary to transform gold from a large sample volume to the solution, which can considerably reduce the speed of analysis and increase its cost.

The emission spectrum analysis that effectively determines the composition of platinum-group metals and gold in the samples of sulfide copper-nickel ores with high contents of copper, nickel, and iron has attracted much attention in the recent years [15].

In the emission spectrometry, the units based on different types of discharges such as direct-current

arcs, arc discharges of power current, spark discharges and high-frequency discharges with inductively coupled plasma, and capacitive discharge are widely used [16].

In addition to several advantages of this method such as high sensitivity and possibility to determine a large number of elements there are a number of limitations. One of the most popular methods of discharge based on inductively connected plasma requires a long-time sample preparation and expensive equipment [17]. An application of the discharge on graphite arc is characterized by a high level of background signal, which, together with a poor reproducibility of results and the presence of the cyanate ion bands in the spectrum, and does not allow us to use the visible region rich in line to the maximum extent [18].

Although particular disadvantages of some sources of discharge can be minimized by selecting an optimum source for specific analytical task, it is not easy to eliminate a number of general limitations of the method such as the high labor intensity of the analysis and difficulties in identifying multi-element spectra [19] which can become a critical problem when treating the complex in the composition of technogenic objects of gold mining.

As an alternative, one can use the nuclear-physical analysis methods to avoid the systematic errors in fine-grained gold analysis [20-22]. In particular, the use of instrumental neutron activation analysis (NAA), in which the californium source of neutrons is applied, is promising [23]. This method helps determine the gold content in the samples with masses of up to 300 g, thereby eliminating sample representativity [24].

However, it should be noted that the technogenic gold deposits are highly complex and heterogeneous from the chemical viewpoint, and the nature of interaction of neutrons with different elements is essentially different. Depending upon the chemical composition of the sample, a significant change in the neutron flux distribution in the sample can distort the results of NAA. In order to avoid possible systematic errors, the effect of matrix composition of the sample on the NAA results was investigated in the determination of gold in the technogenic objects with the use of NAA.

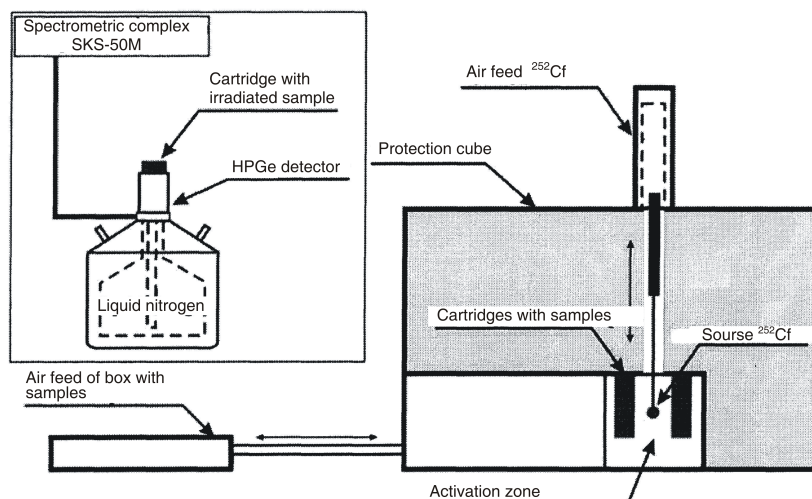
MATERIALS AND METHODS

The experiments were conducted on the NAA unit with the californium source of neutrons characterized by an activity of $1 \cdot 10^8$ per cm^2s .

The experimental set-up and procedure of the unit are depicted in fig. 1, and a detailed description is provided in [23].

The unit includes an activation zone and spectrometric complex. Irradiation of the samples under con-

Figure 1. Experimental set-up and procedure of the neutron activation analysis unit



sideration takes place in the activation zone, and the spectrometric complex measures the induced activity. The activation zone consists of a central channel with the source based on ^{252}Cf and six vertical irradiation channels of up to 16 cm in height and 6.7 cm in diameter, which simultaneously irradiate up to 36 cartridges with the height of 2.7 cm and the maximum sample mass of up to 300 g. In the activation zone, maximum density of thermal neutrons, $F_{\text{th}} = 9.3 \cdot 10^6 \text{ cm}^{-2}\text{s}^{-1}$ as well as resonance neutrons $F_{\text{epi}} = 0.43 \cdot 10^6 \text{ cm}^{-2}\text{s}^{-1}$ can be achieved. Depending upon the expected content of gold, the time taken for activation of samples was 24-64 h, while the curing time was 1 day.

The spectrometry block is based on the complex SKS-50M including the germanium-lithium solid-state detector with an energy resolution of 2.2 keV in the gamma line of $E = 411.8 \text{ keV}$ of the radionuclide ^{198}Au , and the absolute efficiency of registration of this line is 0.25% at the detector volume of 80 cm^3 . In addition, the spectrometry block includes the impulse signal processor SBS-75.

With an apparatus containing the aforementioned parameters, a gold detection limit of 0.5 g/t was achieved.

In order to investigate the effect of the sample mineral composition on gold determination using the NAA method when a large mass of rock (3-5 kg) is introduced in the activation zone, four types of matrix (silicate, carbon-containing, iron-containing, and titanium magnetite) were used. The contents of major macroelements of the studied matrices, according to the X-ray fluorescence analysis (XFA) data, are presented in tab. 1.

Studies on neutron fluxes were conducted using activation detectors (gold foil having an area of 1 cm^2 and thickness of 0.5 m), which were placed in the cartridges filled with rock under consideration. A zone of neutron field contained from 3 to 5 kg of the rock under consideration (depending upon its density) to fulfill the activation channels. The spatial distribution of

Table 1. Chemical composition of matrices (mass percent)

Matrix type	Silicate		Carbon-containing		Iron-containing		Titanium magnetite	
Fe	0.432	0.022	1.003	0.055	31.19	0.39	45.31	0.25
Ti	0.128	0.020	0.009	0.003	3.432	0.052	4.337	0.056
Al	6.760	0.064	11.02	0.088	–	–	1.855	0.049
Mn	0.018	0.004	0.021	0.003	1.524	0.064	0.552	0.006
Si	33.54	0.29	18.40	0.152	0.710	0.018	2.349	0.077
Ca	2.198	0.049	2.224	0.069	5.121	0.218	0.602	0.022
C	2.107	0.052	28.08	0.320	–	–	–	–
O	47.23	0.317	34.30	0.295	53.69	0.520	43.62	0.419

the fluxes of thermal and resonance neutrons was examined by the cadmium difference method; therefore, irradiation of the activation detectors was performed in two ways: with and without cadmium filter. A 0.7-mm-thick cadmium filter was used to exclude the flux of thermal neutrons and to identify the resonant part of the flux. The measurements with each type of matrices were made 6 times as in case of the activation with and without the use of the cadmium filter.

Data of chemical composition obtained using XFA (tab. 1) and reference data on neutron absorption cross-sections for elements were used to calculate the average cross-section of neutron absorption by the matrix elements of the sample as follows

$$\sum_{i=1}^n C_i \sigma_i \quad (1)$$

where C_i (%) is the content of element in the sample, σ_i – the neutron absorption cross-section for this element, and n – the number of elements the contents of which were determined using XFA

RESULTS AND DISCUSSION

Gold determination using NAA was certified to be used with silicate and aluminosilicate samples [25]. Because the technogenic objects of gold mining differ in their chemical composition, there is a need to deter-

mine gold content in rocks with different chemical compositions.

In order to determine the effect of mineral composition of the sample on NAA results, four types of matrix were used, whose chemical compositions are provided in tab. 1. This is explained by the fact that these types of matrix appear frequently in the technogenic objects, while the characteristics of interaction of neutrons with these matrices are different. Lighter elements (*e. g.*, carbon and silicon) have relatively high neutron-scattering cross-sections, while heavier elements (*e. g.*, titanium and iron) are characterized by their relatively large neutron absorption cross-sections, which may significantly affect the average cross section of neutron scattering and absorption for the sample with macro contents of the elements under study.

The following possible sources of the systematic errors of gold determination using NAA based on ^{252}Cf were considered when a large amount of samples, widely differing in their compositions, was introduced in a neutron field formation zone:

- change of neutron spectrum due to different neutron-scattering cross sections, in particular, active neutron scattering by lighter elements, and
- change of neutron flux with respect to neutron absorption by heavier elements present in the samples.

The neutrons of two energy groups (resonance and thermal) are involved in gold activation, but their interaction cross sections with the substance are essentially different. The effect of matrix composition of the rock on distribution of fluxes of thermal and resonance neutrons have been investigated. The obtained results of the spatial neutron flux distribution for different matrices are presented in figs. 2 and 3.

On the basis of the data obtained on irradiation of the activation detectors in the samples with different matrix compositions, the following conclusions can be made.

- Despite the considerable amount (3-5 kg) of rock introduced in the zone of neutron field formation, the flux of resonance neutrons, depending on ma-

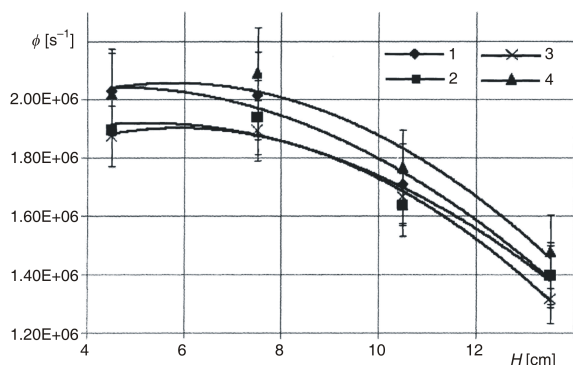


Figure 2. Spatial distribution of the resonance neutron fluxes in different matrices: 1 – silicate, 2 – carbon-containing, 3 – titanium-magnetite, and 4 – iron-containing

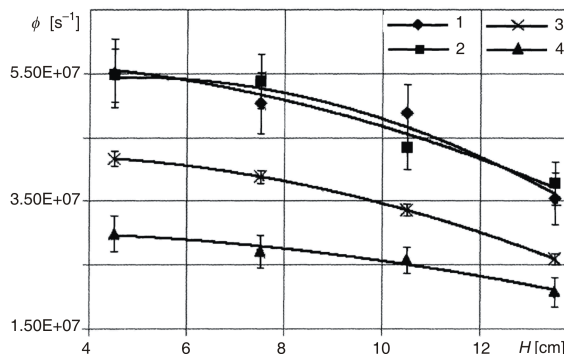


Figure 3. Spatial distribution of the thermal neutron fluxes in different matrices: 1 – silicate, 2 – carbon-containing, 3 – titanium-magnetite, and 4 – iron-containing

trix type, does not change, which would exceed the statistical error (fig. 2). Consequently, during activation of gold by resonance neutrons, the nature of matrix of irradiated rock has no effect on the results of gold determination.

- The thermal neutron flux does not practically change when passing from silicate to carbon-containing matrix (fig. 3), but decreases almost twice at the center of cuvette with a volume of 100 ml when passing to the titanium magnetite matrix (fig. 3). Because the maximum decrease in the thermal neutron flux will occur at the center of cuvette with a sample, for activation of the bulk sample, the detected effect will manifest in a lesser extent. When the bulk sample was irradiated 20 % of gold activation was performed by the resonance neutrons, on which, as noted earlier, the absorption has no significant effect. Therefore, the measurements carried out with two samples (silicate and titanium magnetite) with masses of 50 g each and the same quantity of introduced gold being at the center of the activation channel at a height of 6 cm from the base, for activation in the midst of silicate rock, showed a difference of 10 % when determining the induced activity of gold (fig. 4).

It was assumed that, when passing from silicate to titanium magnetite matrix, a change in neutron flux is related to thermal neutron absorption by the nuclei of iron and titanium present in the sample. The dependence of thermal neutron flux measured at the center of cuvette on the average matrix absorption cross-section is presented in fig. 5.

The silicate and carbon-containing matrices have insignificant average absorption cross-sections of thermal neutrons in contrast to the “heavy” iron-containing and titanium magnetite matrices. These higher values of the average absorption cross-section of heavy matrices correlate with their decrease of thermal neutron flux. The calculation results showed that two elements – iron and titanium – played the primary role in the absorption of neutron flux in the matrices under study. These elements have

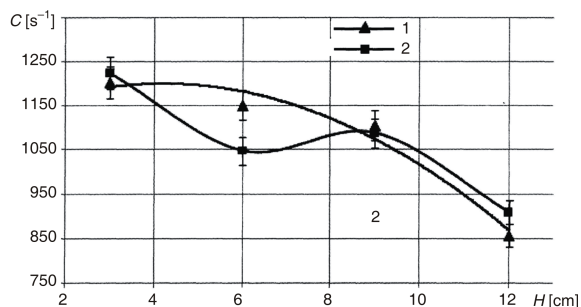


Figure 4. Differences in the induced activities during activation of the bulk gold-containing samples: 1 – silicate, and 2 – titanium magnetite

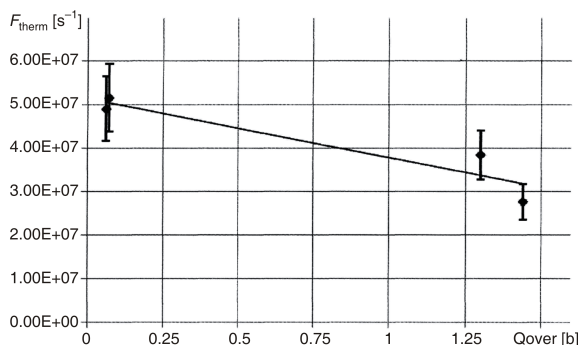


Figure 5. Dependence of thermal neutron flux, F_{therm} , on the averaged absorption cross-section, σ_{aver}

high neutron absorption, and their content in the sample mainly contributes to the decrease of thermal neutron flux. Consequently, it results in underestimation of gold content when using the NAA method.

A systematic error of about 10 % is possible in gold determination by activation of the titanium magnetite and iron-containing samples. In order to eliminate such error, it is necessary to refrain from joint activation of the “light” (silicate and carbon-containing) and “heavy” (titanium magnetite and iron-containing) samples. When using the samples containing significant amount of iron and titanium, it is required to enter the corrective factors based on preliminary chemical analysis of the sample using the XFA method. Abundant arsenic present in the arseno-pyritic samples and rare earth elements can also substantially contribute to the absorption of thermal neutrons due to their higher neutron absorption cross sections. Therefore, the most accurate correction for absorption should be calculated for all elements of the sample determined using the XFA method.

A linear relationship exists between the thermal neutron flux and average cross-section for absorption of the sample matrix (fig. 5), which, in accordance with the results of experimental measurements, is expressed as

$$F_{\text{therm}} = 1.11 \cdot 10^7 \cdot \sum_{el} (\sigma_{el} \cdot C_{el}) - 4.71 \cdot 10^7 \quad (2)$$

where F_{therm} is the thermal neutron flux at the centre of cuvette with the rock, σ_{el} – the absorption cross-section of thermal neutrons by an element, C_{el} – the percentage of element in the sample, and $\sum_{el} (\sigma_{el} \cdot C_{el})$ – the average absorption cross-section of thermal neutrons by the matrix composition of the sample.

For $\sum_{el} (\sigma_{el} \cdot C_{el}) = 1.44 \text{ b}$, ($1 \text{ b} = 10^{-28} \text{ m}^2$) an underestimation of 10 % results in the gold determination using the NAA method, which is only attributed to the absorption of thermal neutrons by the matrix elements of the sample. After determining the chemical composition of the sample by the XFA method and calculating the average absorption cross-section of thermal neutrons, the corrective factor for absorption of neutrons by the sample elements can be determined as follows

$$K_{\text{absor}} = \frac{0.10 \cdot \sum_{el} (\sigma_{el} \cdot C_{el})}{1.44 \cdot \sum_{el} (\sigma_{el} \cdot C_{el})} = 0.069 \cdot \sum_{el} (\sigma_{el} \cdot C_{el}) \quad (3)$$

The gold content in the sample with due account for the corrective factor for absorption of neutrons by matrix elements of the sample is determined as follows

$$C_{\text{Au}} = \frac{c_{\text{det}}}{1 - K_{\text{absor}}} \quad (4)$$

where C_{Au} is the true content of gold in the sample, c_{det} – the gold content determined by the NAA method, using the procedure described [25], and K_{absor} – the corrective factor for absorption of neutrons by matrix elements of the sample.

CONCLUSIONS

The investigations of the effect of matrix composition of the sample on the results of the NAA of gold showed:

- lack of significant difference in the scattering of fluxes of thermal and resonance neutrons emitted from ^{252}Cf when activating the gold-containing technogenic samples with different mineral compositions, and

- decrease in the quantity of generated atoms of radionuclide ^{198}Au in gold-containing technogenic samples due to absorption of the thermal neutron flux from ^{252}Cf by macro scales of iron and titanium contained in the sample results in a systematic error of 10 % in the neutron activation determination of gold.

In order to eliminate the systematic errors in gold determination resulting from the absorption of neutrons with the change in the composition of samples, the technique of introduction of corrections using eqs. 3 and 4 was developed and tested.

The obtained results improved the NAA method for control of the fine-grained gold content at all stages of its recovery, including that in the presence of highly activated elements, thereby increasing its effectiveness and optimizing the schemes of the fine-grained

gold recovery using different methods with respect to the technogenic objects, including those in the Far Eastern region.

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AUTHORS' CONTRIBUTIONS

Theoretical analysis was carried out by V. V. Zheleznov and S. I. Ivannikov; experiments were carried out by S. I. Ivannikov and A. A. Yudakov. All authors analysed and discussed the results. The manuscript was written by O. N. Tsybul'skaya, V. V. Zheleznov, S. I. Ivannikov, A. V. Taskin, and the figures were prepared by S. I. Ivannikov and O. N. Tsybul'skaya.

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**УТВРЂИВАЊЕ ПРИСУСТВА ЗЛАТА НЕУТРОНСКОМ
АКТИВАЦИЈОМ У ТЕХНОГЕНИМ СИРОВИМ МАТЕРИЈАЛИМА
РАЗЛИЧИТОГ МИНЕРАЛНОГ САСТАВА**

Анализиране су методе за утврђивање садржаја злата у техногеним објектима при ископавању злата, у погледу њихове нехомогености и сложености хемијског и минералног састава. Разматрана је могућа примена неутронске активационе анализе са употребом калифорнијума као извора неутрона за одређивање састава фино-зрнастог и екстра фино-зрнастог злата у техногеним објектима, укључујући и пепео снабдевача енергије. Показано је да хемијски састав узорка утиче на расподелу флукса неутрона у узорку што у основи може нарушити резултат неутронске активационе анализе. Да би се елиминисале могуће системске грешке спроведено је истраживање утицаја састава узорка минерала на резултате одређивања присуства злата применом неутронске активационе анализе. Наиме, стена велике масе (3-5 kg) смештена је у активациону зону применом четири врсте матрица, као што су силикатна, са угљеником, са гвожђем и титанијум магнетитом. Показано је да нема значајне разлике у дисперзији флукса термичких и резонантних неутрона из ^{252}Cf током активације техногених узорака различитог минералног састава који садрже злато.

Кључне речи: неутронска активациона анализа, фино-зрнасто злато, техногени објекти, калифорнијум, активациона зона, флукс термичких неутрона