MOBILE FACILITY FOR GAMMA-ACTIVATION ANALYSIS OF GOLD ORES

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Abstract

Results of the development and research of characteristics of a new gamma-activation analysis (GAA) facility, created for quantitative analysis of gold-bearing ores in real conditions of a gold-mining enterprise, are presented. Linear electron accelerator УЭЛР-8-10А (P ≤ 10 kW, E=7-9 MeV), maximally adapted to the tasks of GAA, was used for irradiation. Gamma-radiation was registered by a two-channel precision gamma spectrometer based on hemispherical HPGe detectors with a diameter of 110 mm.

The values of gold detection limit (3σ), measured from the spectra of certified reference samples with an ultra-low background level, were 0.025-0.028 ppm with single irradiation. In this case, the root-mean-square measurement error for a gold concentration of 1 ppm did not exceed 8%, and 4% for a concentration of 10 ppm. The GAA facility provides the analysis of coarse-ground samples (1-3 mm) with a capacity of at least 65 samples per hour.

1. INTRODUCTION

Gamma activation analysis (GAA) is a unique method for analyzing samples at all stages of the search, exploration and production of gold, as well as other precious (noble) metals and related elements [1-6]. The essence of the method consists in irradiating large-mass ore samples with high-energy gamma quanta, generated by a linear electron accelerator (LINAC), and recording the induced activity of excited gold nuclei (isomers) using a gamma spectrometer. The advantages of this method were discussed in detail in [1-6].

These benefits were first realized in three GAA industrial laboratories, commissioned at the Muruntau mine, Uzbekistan in 1977, in Magadan, Russia in 1979 and in Batagay, Russia in 1986 [3]. Currently, only one of the three commissioned laboratories is operating – at the Muruntau mine, Uzbekistan. After modernization of the main systems, the gold detection limit (DL) in this laboratory is 0.04 ppm (3σ) with a measurement error of less than 10% for gold concentrations of 1.08 ppm [7-11]. At the same time, the laboratory demonstrates remarkable productivity – at least 120 measurements per hour. In 2020, about 1 700 000 samples were measured on two measurement lines with two LINACs.

In recent years, due to the intensification of work on the exploration and development of deposits of gold and rare earth elements on all continents, there has been an increase in the interest of both mining enterprises and development companies in the precise and prompt determination of the concentrations of these elements. The Australian company Chrysos Corporation, in partnership with the Chinese company Nuctech, has recently created its first commercial prototype of a facility using GAA technology for gold [6,12,13]. As a result, a gold detection limit of 0.03 ppm (3σ) is declared with a measurement error of 7-8% for a gold concentration of 0.3 ppm and 4.0% for a gold concentration of 1.0 ppm. It should be noted that the analytical productivity of the equipment is 72 samples per hour.

This work is devoted to the results of the development and study of the characteristics of "Au-Isomer", a new industrial GAA facility created for PAVLIK Gold Ore Company, Russia [14]. The entire complex of the

"Au-Isomer" GAA facility being put into operation, is designed as a mobile one; its design allows disassembling the GAA facility and transporting it to another mine in four 40-foot containers. When developing and studying the characteristics of the GAA facility itself, the design, principle of operation and characteristics of which are presented in this work, the main efforts of the developers were aimed at providing high productivity of gold analysis and at the same time ensuring its high sensitivity and accuracy.

2. FACILITY DESIGN

The facility has dimensions of 7.1 x 4.5 m and a height of 4.1 m together with a crane, designed for assembly and disassembly of the radiation shield (Fig. 1). Outside the facility, in separate rooms, a power supply unit for the accelerator is located, and a chiller for cooling the target of the LINAC.

FIG. 1. "Au-Isomer" gamma-activation analysis facility.

The installation works in a fully automatic mode; the algorithm of its operation is presented below (Fig. 2). Containers with ore samples 1, installed on the receiving conveyor 2 of the GAA facility's Sample Transportation System, are fed through conveyor 3 to channel 4, along which the containers are rolled to the target 5 of the LINAC. During irradiation, containers are rotated by drive 6 to ensure uniform distribution of the dose in the container volume.

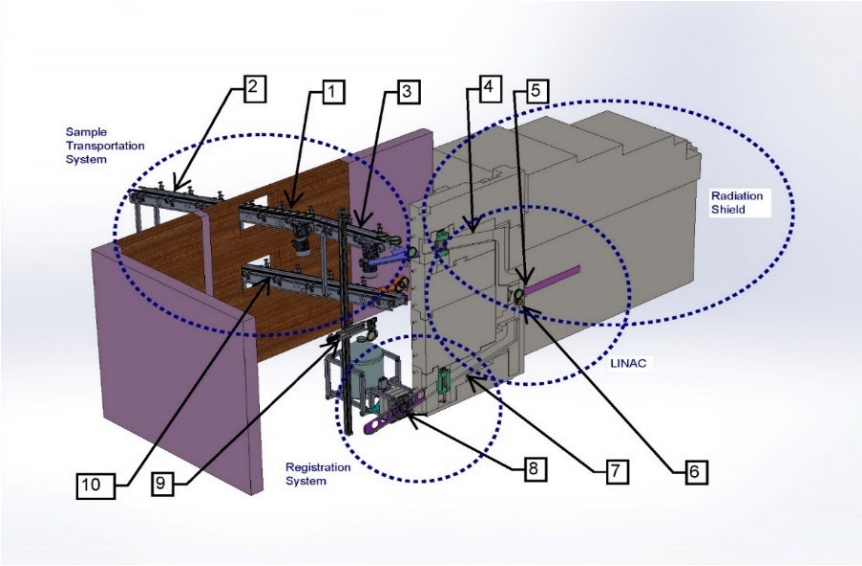

FIG. 2. The GAA facility sketch drawing.

Channels 4 and 7, like the LINAC, are surrounded by Radiation Shield. After irradiation, the containers are rolled down channel 7 to precision spectrometers 8 of the Registration System to measure the induced activity. After measurement, the containers are lifted by a lifting mechanism 9 of the Sample Transportation System for unloading onto a containers' return conveyor 10 or loading into channel 4 for re-irradiation. The decision to re-irradiate is carried out by a computer program at a low signal level in the energy range of radiation of the gold isomer. The containers measured for the second time will alternate in channels 4 and 7 with the containers measured for the first time, and the barcode system of the containers at the entrance to channel 4 will inform the computer control system of which container went to irradiation.

At present, time specifications for the developed GAA facility's operating procedure are as follows: moving the container to the barcode reader area – 10 sec, to the irradiation area after reading the barcode – 2 sec; irradiation – 10 sec; moving the container to the detectors of the registration system after irradiation – 2 sec; measurement – 15 sec; making a decision on re-irradiation – 0.5 sec, unloading – 12 sec. Thus, the total time of one analysis is about 52 seconds, which provides an analysis throughput of at least 60-65 analyzes per hour. It is planned in the near future to reduce the time of samples feed to the reader, starting from the second sample to 3 seconds, and the time of unloading – to 9 seconds. Thus, the total time of one analysis will be about 47 seconds, which will provide an analysis throughput of at least 75 analyzes per hour. This is less than in Muruntau (120 s/h), but comparable to Chrysos (72 s/h) [12].

2.1. Linear Electron Accelerator (LINAC)

In the developed facility, a LINAC УЭЛР-8-10А [15,16] of the Corad company was used, which was maximally adapted to the tasks of GAA (Fig. 3). The accelerator generates an electron beam with a power of up to 10 kW and electron energy adjustable in the range of 7-9 MeV. The ability to adjust the electron energy and maintain it at a stable level provides ample opportunity for optimizing sample irradiation conditions to suppress lines of interfering elements that may overlap with gold lines in the GAA spectrum. In turn, this allows increasing the accuracy of determining the gold in the sample and reducing the limit of its detection.

Circuits for stabilizing the amplitude of the beam current pulse, the coolant temperature and the level of microwave power supplied to the input of the accelerating section ensure the stability of the bremsstrahlung intensity and electron energy in the accelerator within 2%. As a microwave power amplifier in a LINAC, the klystrons КИУ-268 (Torium, Russia), VKS-8262F (CPI, U.S.A.) or TH2173F (Thales Electron Devices, France) can be used. The high-voltage power supply of the klystron and the electron source is provided by two highly efficient semiconductor modulators. The accelerator is powered from a three-phase network 380/220 V, 50 Hz, the maximum power consumption of the accelerator is 80 kW.

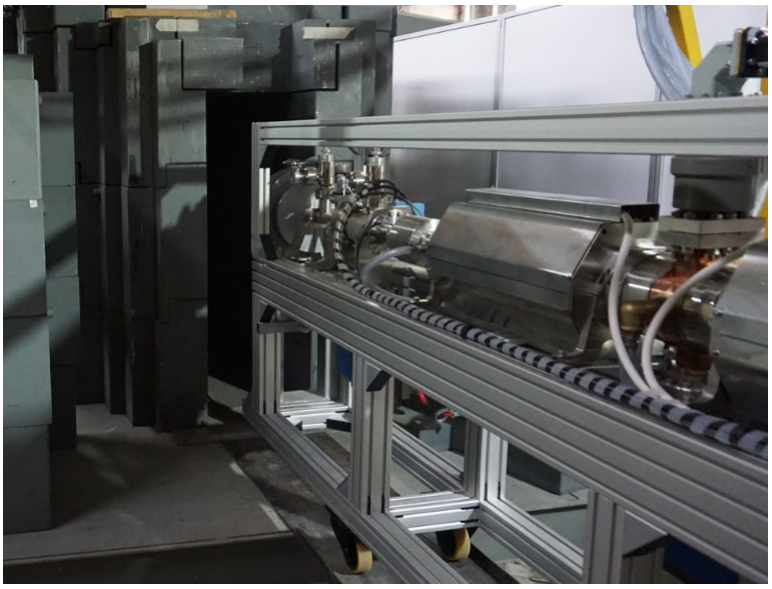

FIG. 3. LINAC in the "maintenance position", outside of the radiation shield.

2.2. Radiation shield

In the developed GAA facility, the LINAC and the system for moving containers with samples are placed in an iron collapsible radiation shield in order to prevent the radiation from affecting personnel and the background level when registering induced activity. The radiation shield provides a level of background radiation at any point around its enclosure of no more than 1 μSv/h. This level of radiation background formally allows the installation of "Au-Isomer" in ordinary industrial premises without the allocation of a sanitary protection zone and a restricted area.

The radiation shield consists of iron blocks; their weight does not exceed 2.4 tons. The total weight of the radiation shield is 118 tons. All assembly work is carried out using a conventional overhead track crane with a lifting capacity of up to 3.5 tons, which is a part of the facility. Dismantling front protection blocks provides access to the sample container transportation system. Disassembling the blocks at the rear of the shield provides the ability to roll the accelerator out of the shield for maintenance and repair. This procedure takes no more than 20 minutes. The mobility of the complex is contingent on the possibility of disassembling the radiation shield and transporting it to another facility (mine), if necessary, in four trucks.

2.3. Radiation registration system

The GAA facility registration system determines practically all of its metrological characteristics. It is a two-channel precision gamma spectrometer based on hemispherical HPGe detectors with a diameter of 110 mm in each channel (Fig. 4) [17]. Detectors of such a large diameter were developed by us specifically for the tasks of detecting gold in ore samples. Unique values of the energy resolution ((702-726) eV at 122 keV and (1337-1375) eV at 662 keV) with a high relative detection efficiency of gamma radiation (80%), allow for high sensitivity and accuracy of the gold concentration analysis. The detectors are cooled with liquid nitrogen. Processing and amplification of signals from detectors are carried out by a Boson multichannel analyzer equipped with analytical software that records, calculates and analyzes the spectra of all samples [18]. The registration system contains only a passive shield of HPGe detectors and does not contain active shielding.

FIG. 4. BSI production of large diameter HPGe detectors for gold analysis.

2.4. Container transportation system

The container transportation system for this project is developed on the basis of standard industrial automation components; the overall control of the system is fully automatic, carried out using the industrial Unitronic controller with a touch-screen display. The container transportation system performs the functions of containers' alternate reception, their transfer to the irradiation zone, then to the measurement zone, return to the irradiation zone or moving them to the storage location. During the container movement, the transportation system reads all information about the analyzed sample from the barcodes.

3. SAMPLE PREPARATION

Preparation of sample material for analysis is the most important technological procedure of any analytical method; it is what the accuracy of the analysis is largely dependent on [1-3,6]. The developed method of sample preparation for GAA of gold does not contradict the basic requirements of the analytical work quality management system for quantitative methods of analysis [19]. The responsibility for the quality of sampling and the representativeness of the initial geological sample lies with the acquirer of the analysis. Taking into account the vast experience in the practical implementation of the GAA method for gold (Muruntau and Batagay laboratories), the following optimal sample analysis scheme has been worked out. For analysis, the GAA laboratory is given an analytical sample weighing 1 kg with <3.0 mm fragmentation size. The GAA receives a sub-sample weighing 0.5 kg (approximately ½ part of the analytical sample). Depending on the bulk density, this is a complete standard container for GAA. It has been experimentally established that the bulk density of a finely ground material with a particle size <0.10 mm can vary over a wide range, while the mass of a sample of a standard GAA container is in the range from 250 to 400 grams.

Prepared samples are packed in containers made of radiation-resistant plastic, securing at least 100 irradiation cycles. The diameter of the container was chosen equal to the diameter of the largest presently possible HPGe detector – 110 mm, and the thickness – 45 mm, which provides a sample weight of 500 ± 50 g (depending on the bulk density) and, therefore, good representativeness of the results (Fig. 5).

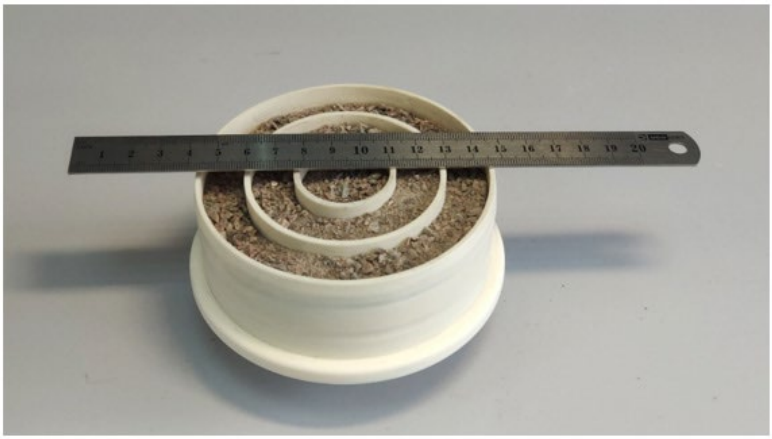

FIG. 5. BSI production of large diameter HPGe detectors for gold analysis.

After carrying out all the sample preparation operations, the necessary identification data of the packed samples (number, weight, date of measurements, etc.) are entered into the computer, which is followed by labelling of the containers with barcodes. The containers, ready for measurements, are moved from the sample preparation room of the GAA Laboratory to the sample loading and unloading room and are placed on the conveyor of the container transportation system, which delivers the samples to the irradiation zone.

4. THE GAA FACILITY'S CHARACTERISTICS RESEARCH AND DISCUSSION

4.1 Calibration

The metrological characteristics of the newly developed GAA facility for gold have been investigated and confirmed during acceptance tests, during which more than 2000 analyzes were carried out. Calibration of the facility before the start of measurements was carried out using reference samples provided by the PAVLIK company, manufactured and certified by VIMS and MST [20,21]. Samples with a content of 1.27 ÷ 24.3 ppm were irradiated once for 10 seconds and, after cooling for 2 seconds, were delivered to the registration area. The spectrum was acquired during 15 seconds. To ensure the precision of the calibration in the range of low gold concentrations, samples of 0.160 and 0.400 ppm were irradiated and tested 4 times, after which the average value of the gold concentration was calculated.

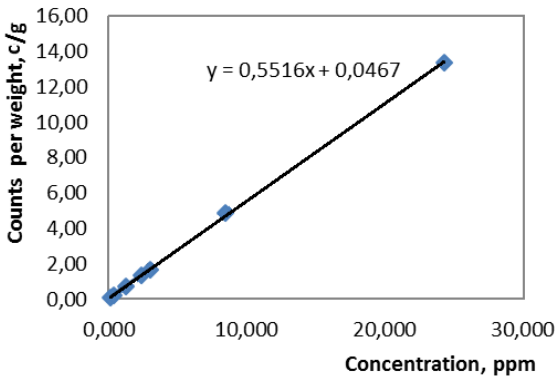

FIG. 6. Dependence of the gold peak of complete absorption area at 279 keV on the gold content in reference samples.

Figure 6 shows a plot of the dependence (based on the results of calibration) of the 279 keV gold peak area of the total absorption, reduced to one gram of the analyzed sample, on the concentration of gold in the reference sample (regression dependence). Subsequently, in the course of measurements, the stability of the calibration of the facility was monitored automatically by feeding a high gold content (24.3 g/t) reference sample for analysis every 3 hours of operation of the facility. To ensure the precision of calibration in the range of low gold concentrations, samples of 0.160 and 0.400 ppm were irradiated and tested 4 times, upon which the average value of gold concentration was calculated.

TABLE 1. PARAMETERS OF REFERENCE SAMPLES AND TESTING RESULTS

Sample №	Reference sample	Au content	Irradiation number	2021.08.31					Average value, ppm	SD, ppм	RMSD, %
				12:10	13:29	14:40	15:57	16:56			
1	OREAS 682	0,074	4	0,087	0,094	0,031	0,105	0,090	0,081	0,030	40,74
2	VIMS211GO	0,160	4	0,303	0,165	0,209	0,109	0,221	0,201	0,085	53,40
3	MST SG147f	0,310	3	0,315	0,316	0,322	0,316	0,361	0,326	0,027	8,60
4	VIMS212GO	0,400	3	0,446	0,359	0,332	0,426	0,400	0,393	0,048	11,92
5	MST Gq157d	0,850	1	0,823	0,916	0,819	0,945	0,888	0,878	0,064	7,56
6	VIMS213GO	1,270	1	1,392	1,245	1,319	1,466	1,479	1,380	0,158	12,45
7	VIMS214GO	2,380	1	2,533	2,463	2,420	2,591	2,420	2,485	0,140	5,87
8	MST 173e	3,000	1	2,987	3,193	3,069	3,162	2,989	3,080	0,131	4,36
10	MST G172f	8,400	1	8,841	8,922	8,275	8,701	8,940	8,736	0,465	5,54
11	VIMS215GO	8,440	1	8,974	8,759	9,590	9,223	8,932	9,096	0,801	9,49
13	MST SGq156i	11,200	1	9,129	8,864	9,003	8,953	9,238	9,037	2,422	21,63
14	VIMS216GO	11,500	1	12,635	12,213	12,706	12,271	12,376	12,440	1,074	9,34
15	VIMS217GO	24,800	1	25,623	25,619	25,507	26,278	25,352	25,676	1,041	4,20
16	VIMS185GO(S)	34,500	1	34,652	34,050	34,235	33,859	34,664	34,292	0,428	1,24

To check the calibration accuracy, multiple analysis of reference samples within a wide range of gold concentrations (0.074 - 34.5 ppm), was performed. The results of the measurements are shown in Table 1. All measurements were carried out 5 times, the values of standard deviation (SD) and root-mean-square deviations (RMSD) of concentration were calculated from the results of the measurements.

4.2 Study of the gold detection limit (DL)

To determine the DL of the developed GAA facility, we used ultra-low background samples manufactured and certified by the MST and Rosdragmet firms [21,22]. Ultra-low background samples were made of purified natural quartz sand with the addition of gold: MST SG147f with a gold content of 0.312 ppm, MST Gq157d –

0.85 ppm, sample 27006101 – 1.908 ppm. All spectra were recorded after single irradiation for 10 s and a spectrum acquisition time of 15 s. For example, the spectrum of the MST Gq157d sample is shown in Fig. 7. To the left of the gold peak, a Hafnium peak is seen in the spectrum of MST Gq157d. The MST SG147f 's spectra contain trace concentrations of Barium and Yttrium, and the spectrum of the 27006101 sample contains Selenium Se-77m (not shown). However, these trace amounts of impurities do not affect the background characteristics of the samples when determining DL for gold.

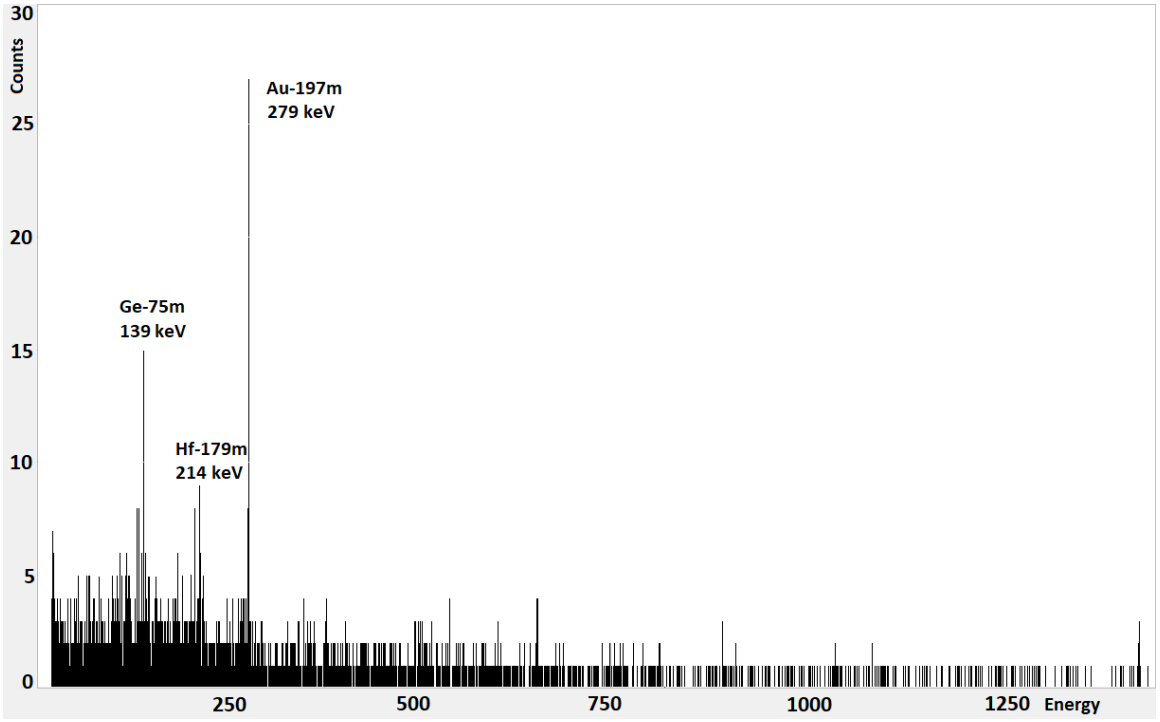

FIG. 7. Spectrum of MST Gq157d sample (single irradiation – 10 sec; spectrum set – 15 sec).

The gold detection limit DL was determined in accordance with the international standard [23]:

$$DL[ppm] = \frac{C * 3 * \sqrt{Nf * \Delta Au/\Delta f}}{\mathrm{NAu}}$$

where, C is the concentration of gold in the sample [ppm], NAu is the number of impulses in the peak of gold, Nf is the number of background pulses in the selected zone, ΔAu is the width of the gold peak in the channels, Δf is the width of the selected background zone in the channels.

According to calculations, the DL values determined from the spectra of certified ultra-low background samples MST SG147f, MST Gq157d, and 27006101 with single irradiation for 10 s and a spectrum acquisition time of 15 s were 0.028, 0.025, and 0.027 ppm (3σ), respectively. The achieved DL level exceeds the DL levels in previous works on the GAA of gold-bearing ores [7-11, 13].

4.3 Accuracy of measurements

An equally important characteristic of GAA is the measurement accuracy [24], which would provide gold concentration values. We conducted a study of statistical errors in the analysis of samples with gold content in the concentration range from 0.312 to 24.3 ppm. Each sample was tested six times with single irradiation. The results obtained were used to determine the average value of gold concentration and a relative standard deviation (SD) from the passport value of the concentration. The test results are graphically presented in Figure 8, and in tabular form, those are given in Table 2. The data show that for the gold concentration of about 1 ppm the root-mean-square deviation does not exceed 8%, and for a concentration of 10 ppm – 4%.

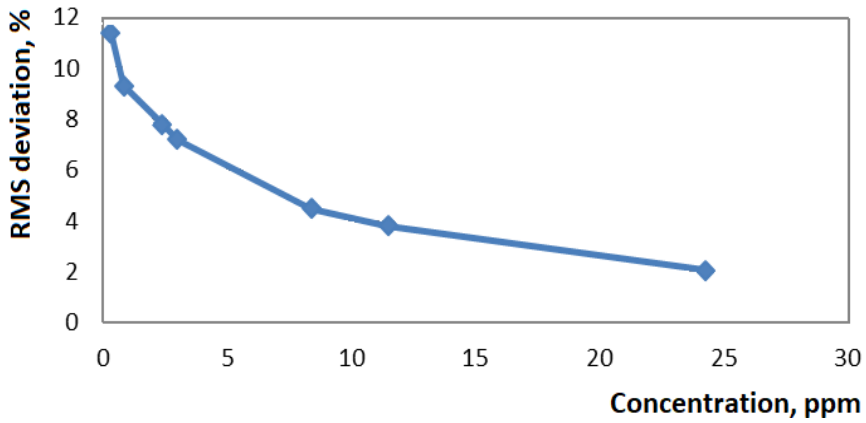

FIG. 8.
Graph of the relative standard deviation dependence on the measured concentration of gold for different concentrations

TABLE 2. STATISTICAL ERRORS IN THE ANALYSIS OF REFERENCE SAMPLES WITH GOLD CONTENT IN THE CONCENTRATION RANGE FROM 0.312 TO 24.3 PPM

Reference sample	Gold concent., ppm	DL, 3σ, ppм	Average concent. value, ppм	SD, ppм	RMSD, %
MST SG147f	0.312	0,034	0.331	0.036	11.390
MST Gq157d	0.850	0,031	0.885	0.079	9.320
VIMS214GO	2.380	0,078	2.334	0.186	7.800
MST G173e	3.000	0,090	2.908	0.216	7.190
MST G172f	8.400	0,085	8.658	0.376	4.480
VIMS216GO	11.500	0,078	11.731	0.439	3,810
MST209	24.300	0,030	24.312	0.501	2.060

4.4 Stability of the results of gold concentration determination

To study the stability of the results of determining the gold concentration in our experiments, we used samples MST209 with a gold concentration of 24.3 ppm, VIMS212GO with a gold concentration of 0.4 ppm and MST SGBlank10 with a gold concentration of less than 0.005 ppm. The measurements were carried out for 6 consecutive days, 5 measurements per day at different times, with constant calibration coefficients and irradiation modes. Thus, MST 209 and MST SGBlank10 samples were tested 30 times with single irradiation. The VIMS212GO sample was analyzed six times. Based on the test results, the mean concentration value and deviation from the mean value were calculated for each sample. The deviation of the measured concentration from the reference for the sample of 0.4 ppm was less than 6.5% and less than 1.0% for the sample of 24.3 ppm. Studies have shown high stability of the results.

In addition, the results of measuring the gold concentration in a sample that does not contain gold were also analyzed for stability. The sample used was MST SGBlank10 with a gold concentration of less than 0.005 ppm. Each measured concentration value for the MST SGBlank10 sample is the average of five measurements with single irradiation of the sample. The test results are shown in Figure 9. Studies have shown that the deviation of the concentration measured in each cycle from the reference does not exceed 0.011 ppm.

4.5 Precision and repeatability of results

Testing of the GAA facility for precision and repeatability [24] of measurement results was carried out on "wild card" samples provided by PAVLIK. In the first stage, the concentration of gold in 30 samples was measured with single irradiation. Based on the measurement results, 17 samples were taken, the gold content of which evenly covers the range of gold concentrations from 0.1 to 20 ppm. The collected samples were tested twice consecutively on the first day and then again on the second day. The measurements were carried out with single irradiation and unchanged settings in the measuring system.

The measurement precision [24] was determined from the obtained values of the concentration of two adjacent measurements on the first and second days. The deviation of each measurement from the average in the concentration range of 0.1-1.0 ppm was 3.78%. The deviation of each measurement from the average in the concentration range of 2.0 ÷ 20.0 ppm was 0.96%.

The repeatability of measurements [24] was determined from the obtained values of the concentration of two successive measurements on the first and second days. The deviation of each measurement from the average in the concentration range of 0.1-1.0 ppm was 1.68%. The deviation of each measurement from the average in the concentration range of 2.0 ÷ 20.0 ppm was 1.4%.

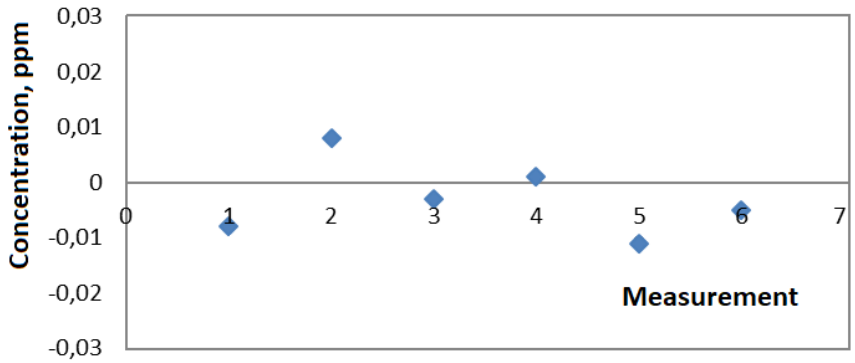

FIG. 9.
Graph of the relative standard deviation dependence on the measured concentration of gold for different concentrations.

4.6 Investigation of real ores

The spectra of the real samples of gold-containing ore with the presence of various associated elements were studied. Our test results, obtained with real ore samples, have confirmed the effectivity of the analysis for the following elements: Au, Ag, As, Ba, Br, Cd, Er, Ge, Hf, Hg, In, Ir, Lu, Pb, Pt, Rh, Se, Sn, Th, U, Y, and W. The concentrations of all these elements can be accurately measured by developing appropriate measurement techniques. In the process of spectra measurements, it was noted that the main input to the background pedestal where the peaks of gold, silver, hafnium and other associated elements are situated, is specified by the products of the photofission of uranium and thorium. The content of uranium in ore samples provided to us was estimated as ~ 40-50 g/t, thorium ~ 100 g/t, barium ~300 g/t, and yttrium ~ 200 g/t.

4.7 Metrological Certification

The Au-Isomer GAA facility has been certified by the State Test and Measurement Instrument Certification Center of the D.I. Mendeleev All-Russian Institute for Metrology (VNIIM), as per the state GOST R 8.568-2017 standard, and recognized as suitable for use in testing samples of rocks, ores and products of their processing for gold content by the method of gamma activation analysis using a UELR-8-10A linear electron accelerator (certificate № 209/06-2021 of 15.10.2021).

A measuring technique "Determination of gold in rocks, ores and products of their analysis by the method of gamma activation using a UELR-8-10A linear electron accelerator" for the facility has passed the metrological certification, as well (certificate No 2121/209 - (RA.RURU10494) -2021, dated 17.12.2021).

5. CONCLUSION

In the course of characteristics studies, more than 2000 analyzes were carried out on the developed "Au-Isomer" GAA facility. The facility demonstrated excellent DL values – (0.025-0.028) ppm with single irradiation for 10 sec and a spectrum acquisition time of 15 sec. In this case, the root-mean-square measurement error for a gold concentration of 1 ppm did not exceed 8%, and for a concentration of 10 ppm – 4%. The "Au-Isomer" provides an analysis capacity of at least 65 samples per hour with the possibility of further increasing the productivity up to 75 samples per hour.

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