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Determination of Gold by Stripping Voltammetry in Platinum Gold Ore Mineral Raw Materials on Grafite Electrode Modified by Bismuth

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Abstract

The paper considers the possibility of determining the gold (III) by stripping voltammetry on graphite electrode modified by bismuth. It is shown the modification of the graphite electrode by bismuth increases the sensitivity of detection of gold in 2 times. The comparison of the results of gold determination by stripping voltammetry and by atomic absorption is presented. The advantages of stripping voltammetry on a graphite electrode modified with bismuth for gold determination are given.

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Keywords: stripping voltammetry, graphite electrode, modification of the graphite electrode.

1. Introduction

Special feature of the raw material base of the mining industry is a trend to the widespread use of objects with poorer and more complex composition of ores¹, gold content is much lower than in the feedstock. Such objects are poor, finely disseminated, arsenical ores and ores of small deposits, stale tails to gold enrichment plants. For determination of gold trace in such ores the stripping voltammetry has been used for a long time. It has a high sensitivity, low cost hardware and allows to define a wide range of elements in the determined contents.

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Previously, it was shown that for gold (III) determination by a stripping voltammetry the graphite electrode was used². From the literature it is known that the sensitivity of the elements determination by stripping voltamperometry can be significantly improved by using graphite electrodes modified with metals³.

The aim of this work is to consider the possibility of gold determination using a modified bismuth graphite electrode and to develop a technique of gold determination in mineral raw materials and industrial waste.

2. Materials and Methods

To conduct the research and to record the current-voltage curves voltammetric analyzer TA-4 ("OOO" Tomanalyt", Tomsk) with a personal computer was used.

The electrochemical cell consisted of 3-electrodes, wherein the indicator electrode is a graphite electrode (GE) impregnated by polyethylene. Electrochemical cleaning of the electrode surface from gold precipitation was carried out for 1 minute with potential +1.0V. Saturate silver-chloride electrodes, filled with a 1 M solution of KCl, were used as the subsidiary electrode and the reference electrode.

Stirring the solution during electrolysis was carried out automatically by the vibration of the indicator electrode, which is provided by the analyzer used.

Electroconcentration of gold and bismuth on the surface GE was carried out at a minus 1V potential within sixty seconds from a 1M solution of HCl, containing ions of bismuth (III) and gold (III) ions. Electrooxidation of the precipitation was carried out at a linear potential variation ($V = 80 \text{ mV/s}$) in the potential range from minus 0.2 V to 1V. Electrooxidation of gold was observed at a potential of +0.8 V.

3. Results and Discussion

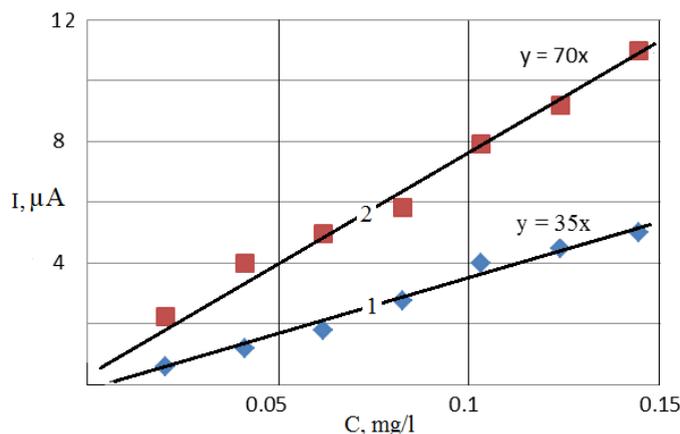


Fig.1. Electrooxidation currents dependence of gold from ion concentration of gold (III) in a solution: 1 - currents of gold electrooxidation from the surface of the graphite electrode; 2 - currents of gold electrooxidation from the surface of the graphite electrode modified by bismuth.

Fig.1 (1,2) shows the calibration dependence on the determination of gold ions (III) by stripping voltammetry using graphite electrode without modifier (1) and graphite electrode modified by bismuth (2). As it is seen from Figure 1, GE bismuth modification increases the sensitivity of the gold (III) determination in two times.

Determination of gold was carried out in Bakchar (blade of hydraulic and silt) ore deposits and the Urals copper-molybdenum porphyry ores. Minerals contained in their composition either ignoble sample components, or platinum metals and silver. Ignoble sample components being deposited with gold, do not interfere the stripping voltammetry of gold determination. It was determined with the use of a bismuth-graphite electrode that even the phase structures (solid solutions and intermetallic compounds) of gold produced on the surface of graphite electrode in the presence of ignoble sample components are oxidized at potentials significantly more negative than potential of gold electrooxidation, therefore the phase structures do not interfere with gold definition by stripping voltammetry method. The presence of the silver and platinum metals is undesirable, as they form the solid solutions, the oxidation

of which distorts the current-voltage curves of gold electrooxidation. Fig.2 shows the current-voltage curves of electrooxidation of Pd-Au binary alloy, described in⁵. Therefore, gold determination by the stripping voltammetry method in the objects is possible only after sample preparation. For separation of gold (III) from the sample matrix, we proposed to extract it from the solution of 6M HCl with ether.

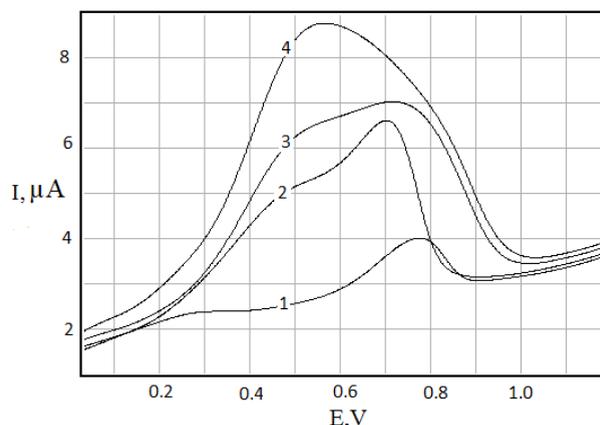


Fig.2. Current-voltage curves of electrooxidation Pd-Au precipitate from graphite electrode surface. C, mg/d³: 1 - C(Au³⁺)=1·10⁻²; 2 - C(Au³⁺)=1·10⁻² and C(Pd²⁺)=1·10⁻²; 3 - C(Au³⁺)=1·10⁻² and C(Pd²⁺)=2·10⁻²; 4 - C(Au³⁺)=1·10⁻² and C(Pd²⁺)=3·10⁻².

4. Analysis technique

Analyzed sample is dried in an oven at 105-110° C to constant weight and poured onto a flat surface, aligned, split into four parts. Then, the ¼ part was taken from each part for analysis, these parts were mixed in the combined sample. A sample of 1 g. was taken from the combined sample using an analytical balance. Weighed sample is placed in a corundum crucible and, then, placed in a cold muffle. The muffle temperature was raised in steps to 850° C and held for 45 minutes. The muffle is disabled and the sample is cooled to room temperature. 5 ml of concentrated HF were added to the sample and evaporated on a hot plate to a dry residue (in this case silicon is removed in the form SiF₄). If necessary, this procedure is repeated. 25 ml of aqua regia are added to the sample and heated for 15 minutes. 6 M HCl is added to the resulting solution, the crucible volume is made up to 25 ml and the solution is boiled for 10-15 minutes. Then, the solution is cooled to 50 ° C and filtered through a "blue" or "red" tape. The precipitate is washed with twice distilled water. The filtrate was transferred to a separatory funnel, 10 ml diethyl ether was poured and gold (III) extraction was carried out twice. After lamination (the ethereal layer is above) the acid solution is poured back into the flask and diethyl ether (the extract) was washed with bidistilled water, shaking the mixture several times in a separatory funnel. The washed ether is poured into a quartz beaker and evaporated with gentle heating until a wet cake condition. 1 ml of 6M HCl is added to the solution and evaporated to a minimum sample (approximately 0.5 ml). The solution volume was made up to 10 ml with bidistilled water and the content of gold (III) was determined by the stripping voltammetry method.

Determining correctness of gold by the stripping voltammetry method evaluated by comparing the analysis results with the data obtained by the atomic absorption spectroscopy method (Table 1).

Table 1. The results gold determination by the atomic absorption spectroscopy method and the stripping voltammetry method in the ores (weigh is 1 g, n = 5, P = 0.95)

№ sample	The sample material	atomic absorption spectroscopy Au, g/t	stripping voltammetry Au, g/t
7207/039a	Cataclastic yellowish-brown-gray mottled with frequent streaks of black quartz, morion and fluorite gneisses.	(0.786±0.097)	(0.754±0.060)
7207/085	Metasomatites (pyrite-carbonate-K-feldspar) by pyroxene pink-gray with a yellow tinge gneisses. Blotches fluorite.	(0.277±0.033)	(0.301±0.033)

7401/019	Pyroxene gneisses. Species intensively limonitized by weight, argillised with frequent weather pits done by limonite, cataclastic partly	(2.227±0.249)	(2.187±0,209)
7403/007	Cataclasites by granites with frequent streaks of quartz and carbonate, carbonate inclusions	(0.199±0.021)	(0.173±0.021)
7403/017	Alaskite granites	(0.103±0.012)	(0.112±0.014)
7403/036	Pyroxene gneisses with small areas of granitisation, irregular cataclasted with frequent streaks of dark gray and black quartz	(0.455±0.049)	(0.459±0.053)
7403/057	Pyrite-carbonate-feldspar metasomatites by pyroxene gneisses	(1.681±0.162)	(1.657±0.162)
7404/002	Pyrite-carbonate-feldspar breccia	(1.387±0.155)	(1.413±0.175)
7405/062	Irregularly mineralized cataclasites by granite-gneisses, with fine mylonite sutures, metasomatites, breccias inclusions	(0.080±0.009)	(0.073±0.008)

Comparison of results of gold determination by stripping voltammetry and atomic absorption spectroscopy method (table 1) shows the equivalence of the two methods as the absolute value of concentration and determination error.

5. Conclusion

1. Experimentally proved that the studding method with using the analyzer TA-4 allows to define the gold in the mineral ores from gold concentrations 0.1 g/t with an error of about 10%;
2. Stripping voltammetry method using bismuth-graphite electrode selective for gold and allows the analysis of the presence of a wide range of interfering impurities.
3. Modification graphite electrode bismuth improves lower concentration limits for gold 2 times.
4. Stripping voltammetry method is comparable in quality, error determination and rapidity with atomic absorption and atomic emission method, but the cost of analysis in times less due to the simplicity and low cost equipment

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