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Д.В. Сокольский атындағы «Жанармай,
катализ және электрохимия институты» АҚ

ХАБАРЛАРЫ

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН
АО «Институт топлива, катализа и
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Қазақстан Республикасы Ұлттық ғылым академиясы "ҚР ҰҒА Хабарлары. Химия және технология сериясы" ғылыми журналының Web of Science-тің жаңаланған нұсқасы Emerging Sources Citation Index-те индекстелуге қабылданғанын хабарлайды. Бұл индекстелу барысында Clarivate Analytics компаниясы журналды одан әрі the Science Citation Index Expanded, the Social Sciences Citation Index және the Arts & Humanities Citation Index-ке қабылдау мәселесін қарастыруда. Webof Science зерттеушілер, авторлар, баспашылар мен мекемелерге контент тереңдігі мен сапасын ұсынады. ҚР ҰҒА Хабарлары. Химия және технология сериясы Emerging Sources Citation Index-ке енуі біздің қоғамдастық үшін ең өзекті және беделді химиялық ғылымдар бойынша контентке адалдығымызды білдіреді.

НАН РК сообщает, что научный журнал «Известия НАН РК. Серия химии и технологий» был принят для индексирования в Emerging Sources Citation Index, обновленной версии Web of Science. Содержание в этом индексировании находится в стадии рассмотрения компанией Clarivate Analytics для дальнейшего принятия журнала в the Science Citation Index Expanded, the Social Sciences Citation Index и the Arts & Humanities Citation Index. Web of Science предлагает качество и глубину контента для исследователей, авторов, издателей и учреждений. Включение Известия НАН РК в Emerging Sources Citation Index демонстрирует нашу приверженность к наиболее актуальному и влиятельному контенту по химическим наукам для нашего сообщества.

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SELECTIVE EXTRACTION OF GOLD (III) BY INERT ORGANIC SOLVENTS AND DETERMINATION OF ITS METHYLENE BLUE (MB) DIRECTLY IN THE ORGANIC PHASE

Abstract. A new highly selective, accelerated and simple extraction photometric method for determining gold with MB directly in the organic phase has been elaborated. The developed method is tested on the analysis of production solutions of hydrometallurgical plants, concentrates and rocks without separation of accompanying elements. The developed method of extraction-photometric determination of gold with MB is recommended for the analysis of industrial solutions, waste water, ores, concentrates, rocks and other materials complex in terms of chemical composition without separation of the accompanying elements directly in the organic phase.

Key words*:* photometric method, gold, complex compounds, chloroform, gold extraction.

Introduction. Existing photometric and extraction-photometric methods for the determination of gold (III) for the use of organic dyes [1,2,3,4] are not very selective. Only a few works we can see investigates about minerals and receiving concentrates for extraction chloride of metal ions [5]. The results of the leaching of the tailings from the heap leaching section with the aim of extracting gold associated with sulfides are presented [6,7].Since the complexation of gold (III) with organic reagents happens during the water phase, the resulting ions often form complex colored compounds and conflict with the definition of gold. In the work [8] given general information on the chemical composition of ash and slag wastes and the content of valuable components of the most developed and used deposits in Kazakhstan and attractive from the point of view of extracting valuable materials from them.

Recently for increase of selectivity of methods for determination of elements a new method is applied - selective extraction of colorless complexes of elements by inert organic solvents, with the subsequent addition to the extract of organic dyes and their complexation directly in the organic phase [9,10].

This paper discusses a new selective, simple method based on selective extraction of gold chloride complex (III) with inert organic solvents and its complexation with MB directly in the organic phase.

Source solution of gold (III) with a concentration of 1MB/ml was prepared from the salt of chloride gold. Titer of solution was set by potentiometric titration with Mora salt [11]. Aqueous solution of MB was used.

The spectra of extracts of gold (III) and reagent complexes were shot on SP-26 spectrophotometer, ℓ=1 see. Optical density of the solution was measured on the photoelectric colorimeter KPC-2.

Experiments have shown that gold (III) from highly acidic solutions in the presence of chloride ions and dimethylformamide (DMFA) is well extracted by chloroform. In the absence of DMFA, gold (III) is not extracted. The study of gold extraction (III) by chloroform depending on the concentration of hydrogen ions, chloride ions and DMFA showed that the optimal conditions for gold extraction (III) are: 0.50 - 6.0 g ion/l for hydrogen ions; 0.06 - 2.5 g ion/l for chloride ions and 13 - 26 % (by volume) for DMFA, shaking duration of phases 5 - 10 s. At equal volumes of aqueous and organic phases extraction of gold (III) at a single extraction is 99.9 % and does not change to a ratio of 20:1 phase volume [12].

In these optimum conditions, the composition of the extractable gold chloride complex (III) by the equilibrium shift method has been determined [13]. For this purpose, the dependence of gold extraction (III) on concentration was studied: 1) hydrogen ions (0.030 - 0.96 g ion/l) at constant concentrations of chloride ions (1.0 g ion/l), DMFA (20%) and ionic force (μ = 1) created by adding NaCl solution; 2) chloride ions (0.01 - 0.34 g ion/l), at constant concentrations of hydrogen ions (1.0 g ion/l), DMFA (20%) and ionic strength (μ=1); 3) DMFA (5 - 13%), at constant concentrations of hydrogen ions (1.0 g ion/l), chloride ions (1.0 g ion/l) and ionic strength (μ=1).

The concentration of gold (III) in the extract was determined by the photometric method with the MB reagent. For this purpose, an aqueous solution of MB was added to the extract and the phases in the organic phase were shaken. The resulting colored gold complex (III) with MB was photometrically photometrical relative to the blank experience extract prepared in a similar way. The data obtained are given in tables 1 - 3 and Figures 1 - 3, shows that in bilogorithmic coordinates $\ell g D_{Au} - \ell g C_{H+}$, $\ell g D_{Au} - \ell g C_{Cl-}$, $\ell g D_{Au} - \ell g C_{DMFA}$ DMFA (where D is the distribution coefficient, C is the equilibrium concentration, moths) there is a straight-line dependence with tangent angles of straight lines equal to 1,4,3 respectively. Consequently, gold (III) is extracted by chloroform in the form of $HAuCl_4$; the solvate number $HAuCl_4$ in the extract is 3 [14].

Table 1 - Determination of the number of H^+-ions involved in the extraction of gold by chloroform. $A_{пр}$=0,57; C_{Au}=0,91·10^{-5} m

C_{H+}, mol	A	D	ℓgD	-ℓgC_{H+}
0.015	0,340	1,100	0,040	1,820
0,030	0,400	2,350	0,372	1,580
0,60	0,470	4,700	0,671	1,320
0,119	0,510	8,500	0,930	0,919
0,340	0,540	18,000	1,360	0,630
0,260	0,550	27,500	1,440	0,440

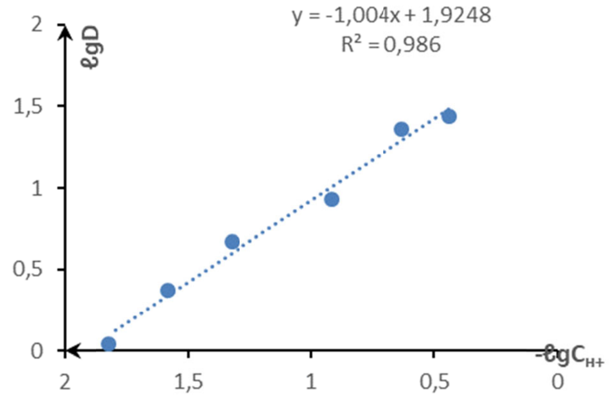

Figure 1 - Determination of molar ratios of Au: H^+ - ions by the equilibrium shift method during chloroform extraction, V_0=10 ml ℓ = 1 cm

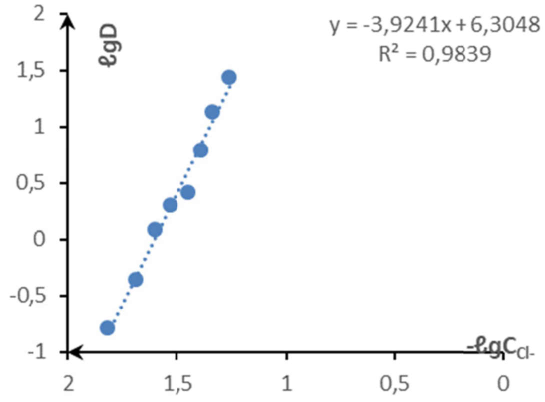

Figure 2 - Determination of molar Au: Cl^- ratios by the equilibrium shift method during chloroform extraction, V_0 =10 ml ℓ = 1 cm

Table 2 - Determination of the number of chloride ions involved
in the extraction of gold by chloroform. $A_{пр}=0,57$; $C_{Au}=0,91 \cdot 10^{-5}$ m

C_{Cl}^{-}, mol	A	D	ℓgD	$-\ell gC_{Cl-}$
0,015	0,080	0,160	0,790	1,819
0,019	0,180	0,450	0,350	1,690
0,035	0,260	0,810	0,090	1,600
0,030	0,360	1,630	0,310	1,530
0,035	0,419	2,630	0,419	1,450
0,040	0,500	6,250	0,790	1,390
0,045	0,540	13,500	1,130	1,340
0,050	0,560	38,000	1,440	1,260

Таблица 3 - Determination of the number of DMFA molecules involved
in the extraction of gold by chloroform. $A_{пр}=0,57$; $C_{Au}=0,91 \cdot 10^{-5}$ m

C_{DMFA}^{-}, mol	A	D	ℓgD	$-\ell gC_{DMFA}$
0,519	0,135	0,310	- 0,508	0,285
0,789	0,280	0,960	-0,0177	0,103
0,909	0,340	1,480	0,170	0,040
1,039	0,390	3,160	0,499	-0,016
1,169	0,440	3,380	0,530	-0,067
1,300	0,480	5,300	0,724	-0,113

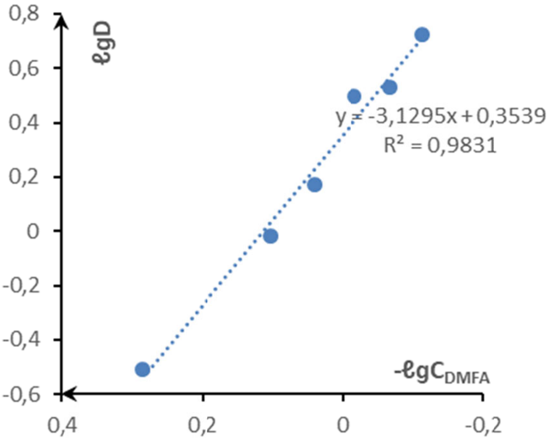

Figure 3 - Determination of Au: DMFA molar ratios by the equilibrium shift method during chloroform extraction

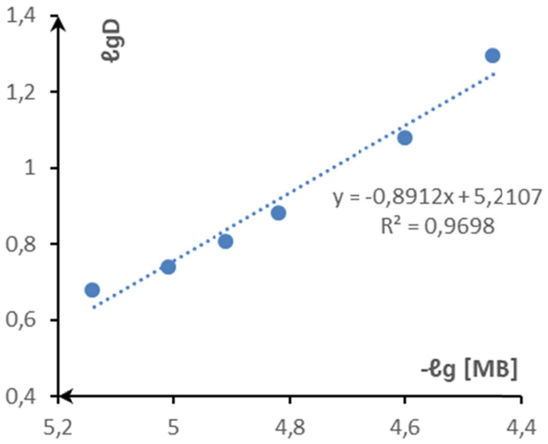

Figure 4 - Determination of the composition of the gold complex with MB method of balance shift

To fully identify the mechanism of extraction of gold (III) studied the number of hydrated water molecules in the extract. For this purpose, 10 ml of a mixture containing 1 M HCl and 20% DMFA, 10 ml of chloroform and shaking for 5 - 10 seconds was added to 5 MB of gold (III). A blank solution without gold was prepared in a similar way (III). After complete separation of the aqueous phase extract, the extracts were poured into dry cylinders with grated plugs. Determination of water was carried out by the Fisher method [15] by amperometry titration. For this purpose, Fischer reagent from micro burette was poured to 1 ml of the test extract taken by the dispenser, the titer of which was determined in advance by water suspension. Under the action of the Fisher reagent the arrow of the galvanometer was shifted towards the current increase and set constant at the end of titration. Knowing the Fisher reagent titer (T=0.0018 g/ml), the water content was determined by formula: $g_{H2o} = T \cdot V$, (where V - is the volume of Fisher's reagent that went on hydrated water titration (V=0.101 ml), T - is the Fisher's reagent titer). The amount of hydrated water in chloroform extraction: $g_{H2o} = 0,0018 \cdot 0,101 = 0,0001818$ gr/ml = 0,1818 gr/l.

Given that the titration of the taken 5 MB of gold (III) and the volume of chloroform 10 ml we calculate the number of hydrated water molecules: $n_{H2o} = \frac{g_{H2o}}{M_{H2o}} : \frac{g_{Au}}{A_{Au}} = \frac{0,1818}{18} : \frac{0,5}{196,9} = \frac{35,7964}{9} = 3,9777 \approx 4.$

Consequently, during the extraction of gold (III) chloroform hydrate 4 water molecules. Considering this, the mechanism of extraction of gold (III) can be represented by an equation: $AuCl_4{}^{-}{}_{(в)} + H(H_2O)_4{}^{+} + 3$ DMFA(в) $\leftrightarrows [H(H_2O)_4\,(DMFA)_3]^{+}\,[AuCl_4]^{-}{}_{(0)}$.

Therefore, the gold chloride complex (III) in the presence of DMFA is extracted by the chloroform for the hydro-solvate mechanism [16].

After extraction of gold (III) chloroform in optimal conditions, separation of the aqueous phase of the addition to the extract of aqueous solution of MB and shaking phases for 10-15 seconds is complexing gold (III) with MB in the organic phase. The study showed that almost complete complex formation of gold (III) with MB in the chloroform occurs in the pH = 6-10 range. The stability of the gold (III) extract has been studied depending on the volume of MB aqueous solution. It is found that the chloroform extract of gold (III) is stable to the ratio of water and organic phases of 1.5:1.0. Gold complex (III) with MB in the organic phase is stable for more than 3 days.

The influence of MB concentration on the completeness of gold complexation has been studied (III). For this purpose, to a constant amount of gold (III), after extraction in optimal conditions, added an increasing amount of MB and photometric. Experimental data showed that the full complex formation of gold (III) is achieved at 18-fold molar excess of MB (table 4 and figure 4).

Table 4 - Determination of the equilibrium composition
of the gold complex with MB method of balance shift. C_{Au} =7,65 · 10^{-6} m, $A_{пр}$=0,52

C_{MB}, M·10^{-5}	A	D	ℓgD	[kompl]M·10^{6}	[MB] M·10^{6}	-ℓg [MB]
1,357	0,430	4,777	0,679	6,319	7,255	5,140
1,630	0,440	5,500	0,740	6,470	9,819	5,010
1,900	0,450	6,419	0,808	6,619	13,385	4,910
2,172	0,460	7,619	0,880	6,760	14,950	4,819
3,358	0,480	12,000	1,079	7,060	25,530	4,600
4,344	0,500	25,000	1,297	7,360	36,085	4,450

The absorption spectra of gold (III) complexes with MB and reagent MB are shown in figure 5.

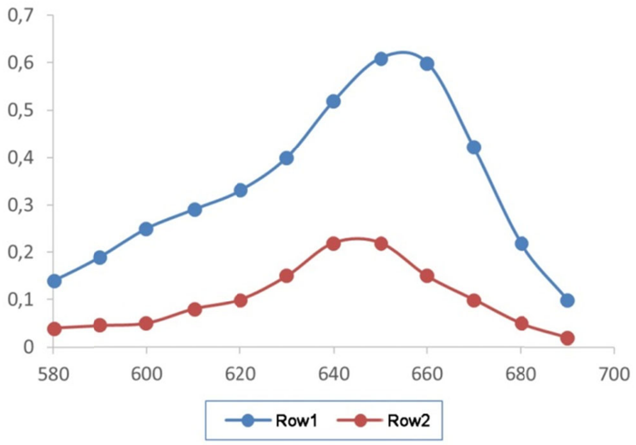

Figure 5 - Absorption spectra of gold complexes (III) with MB (Row1)
and MB reagent (Row2) in chloroform (C_{Au} =0,51 · 10^{-5} M; V_0=10 ml; ℓ =1см)

As can be seen from figure 5, the maximum light absorption of the complex with MB (Row1) is at 650 nm. The molar repayment ratio of the complex is 1.22·105. The Ber law is observed in the interval of 1-120 µg of gold (III) in 10 ml of the extract. Reproducibility of definitions is in the range of 2-5%. There are three graded charts in the intervals of 10 - 10, 10 - 60, 60 - 120 µg of gold. Optical density of complexes was measured in cuvettes with thickness of 20, 10, 3 mm.

The determination of molar ratios of the components in the resulting gold (III) and MB complexes was carried out by the equilibrium shift method [8]. To determine the composition of the gold (III) complex with MB to chloroform extract containing a constant amount of gold (III) (1,326 · 10-5 M, $A_{пр}$ = 0,7) were added 10 ml from 1,359 · 10-5 to 24,403 · 10-5 M aqueous solution of MB in the chloroform and shaken the phases 10-15 sec. The resulting painted gold complex (III) with MB was photometrically photometrical relative to the blank solution prepared in a similar way with the same amounts of reagent. From the tabular data (Table 4) we made a graph of dependence of ℓgD on -ℓg[MB] (figure 4).

The tangent angle of the straight line on the graph shows that gold (III) in the chloroform interacts in a molar ratio of 1:1. Considering the mechanism of extraction of gold chloride complex (III) and the composition of complex compounds with MB, the reaction of complexation of gold (III) with the MB reagent in the organic phase can be presented in the following scheme: $[H(H_2O)_4(DMFA)_3]^+[AuCl_4]^-_{(o)} + PCl_{(B)} \rightleftarrows PAuCl_4 + 3DMFA + HCl + H_2O$. Where the P - is the MB cation.

Methods for determining gold in pure solutions. The dividing funnel is filled with the analyzed solution containing 1.0-120 µg of gold (III), 1 ml of concentrated hydrochloric acid is added to the dividing funnel and the volume of the solution is brought to 8 ml with water, 2 ml of DMFA, 10 ml of chloroform is added and 5-10 s are shaken. The extract is washed with 10 ml of solution containing 1 M HCl and 20% DMFA, and 5-10 s are shaken. Then add 10 ml of 0.010% aqueous solution of MB to the extract and shake 10-15 sec. The resulting colored complex is filtered through filter paper into a cuvette and measured the optical density relative to the blank extract.

In industrial samples and minerals, gold is found in mixtures with many elements. In order to identify the possibility of determining gold from complex chemical samples was studied the influence of foreign ions in determining gold with MB. The method for determining gold in the presence of foreign ions is the same as for determining it in pure solutions, with the only difference being that certain amounts of foreign ions or compounds are preliminarily added to the determined solution and in some cases, if necessary, masking agents are added and the extract is washed once with a washing solution containing HCl and DMFA.

Under conditions of extraction of gold (III) partially extracted thallium ions (III) and antimony (V) and interfered with the definition. However, thallium (III) in chloroform extract with MB in the medium of 0.05 - 0.5 M of borax does not form a colored complex compound and its 5000-fold amount does not interfere with the definition of gold. In the presence of tungsten to prevent the deposition of tungsten acid and to eliminate the interfering effects of antimony (V) previously added 0.01 - 0.5 g tartaric acid. Data on the definition of gold in the presence of foreign ions are given in table 5 with a relative error within 2-5%.

Table 5 - Determination of gold (III) with MB in the presence
of foreign ions (10 µg gold was taken)

M	M/Au	M	M/Au	M	M/Au	M	M/Au
Ag (I)	2000	Mn (II)	100000	Cr (III)	20000	W (VI)	2000^2
Ag (I)	15000	Cd (II)	80000	Ti (IV)	15000	Mo (VI)	15000
Pb (II)	2000	Hg (II)	10000	Sn (IV)	50000	Te (VI)	30000
Pb (II)	20000^3	Bi (III)	20000	Th (IV)	22000	Mn (VII)	1000
Zn (II)	50000	Fe (III)	30000	Pt (IV)	2000	Os (VIII)	200
Ca (II)	20000	Iu (III)	15000	Se (IV)	3000	NaF	5000
Ba (II)	20000	Al (III)	15000	Zr (IV)	15000	NO_3^-	6000
Cu (II)	20000	Ga (III)	10000	Sb (V)	150	PO_4^{-3}	2000
Ni (II)	30000	La (III)	20000	Sb (V)	3000^2	tartaric acid	100000
Co (II)	35000	Tl (III)	1000^1	As (V)	20000	citric acid	100000
Pd (II)	2000	Tl (III)	5000^4	U (VI)	10000		
Be (II)	2000	Tl (III)	10000^1	W (VI)	150	thionalide	10000

Where M - ion or compound; M/Au - allowable ratio to gold by weight; 1 - in the presence of 100 MB thionalide; 2 - in the presence of 0.5 g of tartaric acid; 3 - after heating the solution with sludge in a water bath for 2 - 3 minutes; 4 - when shaking the extract with aqueous solution of MB, containing 0.05 - 0.5 borax.

In order to find out the possibility of determining gold in various objects, we carried out the analysis of production solutions, (Table 6) concentrates and rocks (table 7).

In determining the gold (III) in the production solutions are taken 1 - 2 ml of aliquot part of the solution and further analysis is carried out as in the analysis of gold from pure solutions. The gold content is determined from the calibration chart.

Table 6 - Determination of gold in production solutions (n = 4; p = 0.95)

Content of gold in the production solution, MB/l	Found ($\bar{x}$) gold, MB/l	Sr	$\Delta_{\bar{x}}$	$\pm\frac{\Delta x}{x}\cdot 100$
1,60	1,55	0,0160	± 0,039	± 2,50
15,20	15,30	0,0094	± 0,220	± 1,50
45,00	45,50	0,0056	± 0,410	± 0,990
101,00	101,60	0,0075	± 1,220	± 1,20
142,50	142,30	0,0094	± 2,134	± 1,50

To determine the gold (III) in the concentrates and rocks calcined at 600–650^0 C for 1.5 hours a canopy (10 - 20 g) is placed in a conical flask for 250 ml, 30-60 ml of a mixture of hydrochloric (p 1.19) and nitric (p 1.4) acids (3:1) and evaporated to wet salts. Then add 10 - 20 ml of the same mixture of acids and again evaporate to wet salts, then pour 30 - 40 ml of 1 M HCl and boil for 5 - 10 minutes. Cool the solution and filter it into a measuring flask for 50 or 100 ml, then dilute the filtrate 1 M HCl to the mark and determine the gold content from the aliquot of the solution.

Table 7 - Results of gold (III) determination in concentrates and rocks at chloroform extraction (n = 4; p = 0.95)

Sample	Contents, Au gr/t	Found Au ($\bar{x}$), gr/t	Sr	$\Delta_{\bar{x}}$	$\pm\frac{\Delta x}{x}\cdot 100$
Concentrate	50,00	51,20	0,034	± 2,780	± 5,42
Concentrate	37,50	96,50	0,022	± 1,277	± 3,50
Concentrate	22,00	21,60	0,028	± 0,972	± 4,50
Breed	8,30	7,60	0,036	± 0,441	± 4,80
Breed	5,60	5,15	0,041	± 0,334	± 6,50

Discussion of results. Therefore, our research allows us to conclude that the low selectivity of metalloids definitions using basic dyes, as well as the use of other organic reagents in the complex formation in the aqueous phase is the main disadvantage of existing methods.

Our research has focused on ways to increase the selectivity and sensitivity of photometric definitions, as these analytical characteristics play an important role in the practice of analytical chemistry. We have investigated the conditions of selective extraction of the gold chloride complex in the presence of DMFA inert organic solvents and its determination directly in the organic phase without separation of the accompanying elements.

Researches have shown that gold (III) from a highly acidic environment is selectively extracted by chloroform in the presence of chloride ions and DMFA. At 5 - 10 second phase shaking gold (III) is extracted by 99.9% without changes to the ratio of phase volumes 20:1.

Under these optimal conditions, thallium (III), antimony (V) and tungsten (VI) are partially extracted together with gold and interfere with the definition. However, thallium, antimony, and tungsten interfere with the extraction by adding masking agents to the extraction mixture.

The research of substance of extracting gold chloride complex (III) in the extract has shown that the gold chloride complex (III) is extracted by the hydro-solvate mechanism. Gold chloride complexes (III) in the extract are stable for a long time and subject to the Beer`s law in a wide concentration range. The method is valuable also by the fact that after the extraction of gold in the organic phase there is a possibility of forming complexes with more sensitive organic reagents, which due to low selectivity have not received wide application.

Conclusions. The research resulted in the development of a new highly selective, accelerated and quick spectrophotometric extraction method for the determination of gold directly in the organic phase. It has been established that gold (III) is extracted by chloroform from a highly acidic medium in the presence of chloride ions and DMFA by the hydro-solvate mechanism. The influence of foreign ions that do not interfere with the definition of gold in large multiples has been studied. The developed method was tested on the analysis of production solutions, concentrates and rocks without separation of accompanying elements. The method is easy to perform, highly selective and rapid.

The developed method of extraction-spectrophotometric determination of gold is therefore suggested for the analysis of industrial solutions, wastewater, concentrates, ores, rocks and other materials with complex chemical composition without the separation of the corresponding elements directly in the organic phase.

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АЛТЫНДЫ (III) ИНЕРТТІ ОРГАНИКАЛЫҚ ЕРІТКІШТЕРМЕН ІРІКТЕУ ЭКСТРАКЦИЯСЫ ЖӘНЕ ТІКЕЛЕЙ ОРГАНИКАЛЫҚ ФАЗАДА МЕТИЛЕНДІ КӨКПЕН (МГ) АНЫҚТАУ

Аннотация. Органикалық бояғыш қолдану арқылы алтын (III) анықтаудың фотометриялық және экстракциялық-фотометриялық әдістері аз іріктелген. Органикалық реагенттермен алтынның (III) кешенді түзілуі су фазасында жүреді, жанама иондар боялған кешенді қосылыс түзеді және алтынды анықтауға бөгет жасайды.

Соңғы уақытта элемент анықтау әдістерінің селективтілігін арттыру үшін жаңа тәсіл – инертті органикалық еріткіштермен элементтердің түссіз кешенін таңдап алу экстракциясы қолданылады, онда кезектегі органикалық бояғыштарды тұндырғышқа қосу және оларды тікелей органикалық фазада кешенді түзілісі жүреді.

Жұмыста инертті органикалық еріткіштері бар және тікелей органикалық фазада МГ кешенді түзу арқылы алтынның (III) хлоридті кешенінің таңдаулы тұндырмасына негізделген жаңа селективті, қарапайым әдіс талқыланады.

Алтынның (III) бастапқы ерітіндісінің 1мг/мл концентрациясы алтын хлориді тұзынан дайындалады. Ерітіндінің титрі Мор тұзымен потенциометриялық титрлеу арқылы орнатылады. МГ су ерітіндісі қолданылады. Алтын (III) кешені және реагенттер тұндырмасының спектрін СФ-26 спектрофотометрде түсіреді, ℓ=1 см тең. Ерітіндінің оптикалық тығыздығы КФК-2 фотоэлектроколориметрінде өлшенеді. Тәжірибе нәтижесі көрсеткендей, хлорид-иондар және диметилформамидтің (ДМФА) қатысуы негізінде күшті қышқылды ерітінділерден алтын (III) хлороформмен жақсы тұндырылады. ДМФА болмаған жағдайда алтын (III) тұнбаға түспейді. Сутегі иондары, хлорид иондар және ДМФА концентрациясына байланысты алтынды (III) хлороформмен тұнбаға түсіруді зерттеу жұмыстары көрсеткендей, алтын (III) тұндырудың оңтайлы шарттары: сутегі иондары бойынша 0,50-6,0 г-ион/л; хлорид иондары бойынша 0,06-2,5 г-ион/л және ДМФА бойынша 13 -26 % (көлемі негізінде), фазаларды сілку ұзақтығы – 5-10 с. Су және органикалық фазалардың бірдей көлемінде алтын (III) бөлу бірреттік тұндыруда 99,9% құрайды және 20:1 фаза көлемінің арақатынасына дейін өзгермейді.

Көрсетілген оңтайлы жағдайда тепе-теңдікті жылжыту әдісімен тұндырылатын алтын (III) хлоридті кешен құрамы анықталады. Бұл үшін алтын (III) тұндырмасының концентрацияға тәуелділігі зерттеледі: 1) хлорид-иондары (1,0 г - ион/л), ДМФА (20%) тұрақты концентрациясы және NaCI ерітіндісін қосу арқылы жасалатын ион күші барысында сутегі иондары (1,0 г-ион/л); 2) сутегі иондары (1,0 г-ион/л), ДМФА (20%) тұрақты концентрациясы және ион күші барысында хлорид-иондар (0,01-0,34 г-ион/л); 3) сутегі иондары (1,0 г-ион/л), хлорид-иондарының (1,0 г-ион/л) тұрақты концентрациясы және ион күші кезіндегі ДМФА (5-13%).

Зерттеу нәтижесінде тікелей органикалық фазада алтын анықтаудың жаңа жоғары селективті, жылдамдатылған және қарапайым тұндырмалы-спектрофотометриялық әдісі әзірленеді. Тәжірибе барысында анықталғандай, алтын (III) хлорид-ионы және гидрат-сольватты механизм бойынша ДМФА қатысуымен күшті қышқылды ортадан хлороформмен тұнбаға түседі. Көпеселік мөлшерде алтын анықтауға бөгет болмайтын басқа иондар әсері зерттеледі. Жасалған әдіс өндірістік ерітінді, концентрат және жынысты қосалқы элементтерді бөлмей талдауда тексеріледі. Әдіс қарапайым орындау арқылы, жоғары талдау бойынша экспрессиямен ерекшеленеді.

Осылайша алтынды тұндыру-спектрофотометриялық анықтау негізінде жасалған әдіс өндірістік ерітінді, ағынды су, концентрат, кен, тікелей органикалық фазада жанама элементтерді бөлмей материалдардың химиялық құрамы бойынша жыныс және басқа күрделі талдау жасауға ұсынылады.

Түйін сөздер: фотометриялық әдіс, алтын, кешенді қосылыстар, хлороформ, тұндыру.

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ИЗБИРАТЕЛЬНАЯ ЭКСТРАКЦИЯ ЗОЛОТА (III) ИНЕРТНЫМИ ОРГАНИЧЕСКИМИ РАСТВОРИТЕЛЯМИ И ОПРЕДЕЛЕНИЕ ЕГО МЕТИЛЕНОВЫМ ГОЛУБЫМ (МГ) НЕПОСРЕДСТВЕННО В ОРГАНИЧЕСКОЙ ФАЗЕ

Аннотация. Существующие фотометрические и экстракционно-фотометрические методы определения золота (III) с применением органических красителей малоизбирательны. Так как комплексообразование золота (III) с органическими реагентами происходит в водной фазе, в которой сопутствующие ионы также образуют окрашенные комплексные соединения и мешают определению золота.

В последнее время для повышения селективности методов определения элементов применяют новый приём – избирательная экстракция бесцветных комплексов элементов инертными органическими растворителями, с последующим добавлением к экстракту органических красителей и комплексообразования их непосредственно в органической фазе**.**

В настоящей работе обсуждается новый селективный, простой метод, основанный на избирательной экстракции хлоридного комплекса золота (III) с инертными органическими растворителями и комплексообразование его с МГ непосредственно в органической фазе.

Исходный раствор золота (III) концентрацией 1мг/мл готовили из соли хлоридного золота. Титр раствора устанавливали потенциометрически титрованием с солью Мора. Использовали водный раствор МГ.

Спектры экстрактов комплексов золота (III) и реагента снимали на спектрофотометре СФ-26, ℓ=1 см. Оптическую плотность раствора измеряли на фотоэлектроколориметре КФК-2. Опыты показали, что золото (III) из сильнокислых растворов в присутствии хлорид-ионов и диметилформамида (ДМФА) хорошо экстрагируется хлороформом. В отсутствии ДМФА золото (III) не экстрагируется. Исследование экстракции золота (III) хлороформом в зависимости от концентрации водород-ионов, хлорид-ионов и ДМФА показало, что оптимальными условиями экстракции золота (III) являются: 0,50 – 6,0 г-ион/л по водород-ионов; 0,06 – 2,5 г-ион/л по хлорид-ионов и 13 – 26 % (по объёму) по ДМФА, продолжительность встряхивания фаз 5–10 с. При равных объёмах водной и органической фаз извлечение золота (III) при однократной экстракции составляет 99,9 % и не изменяется до соотношения объёмов фаз 20:1.

В указанных оптимальных условиях определён состав экстрагирующегося хлоридного комплекса золота (III) методом сдвига равновесия. Для этого изучена зависимость экстракции золота (III) от концентрации: 1) водород-ионов (0,030 - 0,96 г-ион/л) при постоянных концентрациях хлорид-ионов (1,0 г-ион/л), ДМФА (20%) и ионной силе ($\mu = 1$), создаваемой добавлением раствора NaCl; 2) хлорид-ионов (0,01 - 0,34 г-ион/л), при постоянных концентрациях водород-ионов (1,0 г-ион/л), ДМФА (20%) и ионной силе ($\mu = 1$); 3) ДМФА (5 – 13%), при постоянных концентрациях водород-ионов (1,0 г-ион/л), хлорид-ионов (1,0 г-ион/л) и ионной силе ($\mu = 1$).

В результате исследования разработан новый высокоизбирательный, ускоренный и простой экстракционно-спектрофотометрический метод определения золота непосредственно в органической фазе. Установлено, что золото (III) экстрагируется хлороформом из сильнокислой среды в присутствии хлорид-ионов и ДМФА по гидратно-сольватному механизму. Изучено влияние посторонних ионов, которые не мешают определению золота в больших кратных количествах. Разработанный метод проверен на анализе производственных растворов, концентратов и пород без отделения сопутствующих элементов. Метод отличается простотой выполнения, высокой избирательностью и экспрессностью.

Таким образом, разработанный метод экстракционно-спектрофотометрического определения золота рекомендуется для анализа производственных растворов, сточных вод, концентратов, руд, пород и других, сложных по химическому составу материалов без отделения сопутствующих элементов непосредственно в органической фазе.

Ключевые слова: фотометрический метод, золото, комплексное соединение, хлороформ, экстракция.

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