AMPEROMETRIC TITRATION OF NOBLE METALS WITH SOLUTIONS OF NITROGEN-SULPHUR-CONTAINING REAGENTS IN NON-AQUEOUS AND MIXED MEDIA

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Abstract: The article shows the conditions and the possibility of amperometric titration of noble metals with 4methoxyphenylcarboxymethyldiethyldithiocarbamate (MFCMDETK) and 4-methoxyphenylcarboxymethyl-diethyldithiocarbazone solutions (MFCMDFDTK) in non-aqueous media (acetic acid, n-propanol, DMFA, DMSO) and their mixtures with background electrolytes having different acid-base properties. The methods of amperometric titration of micrograms of the quantities of noble metals ions in the presence of foreign ions containing ions

Key words: palladium, platinum, silver, gold, IFCMDETK, MFCMDFDTK, solution, acetic acid, n-propanol, DMFA, DMSO, background electrolytes.

Annotatsiya: Maqolada 4-metoksifenilkarboksimetildietilditiokarbamat (MFCMDETK) va 4-metoksifenilkarboksimetil-dietilditiokarbazon eritmalari (MFCMDFD) da, suvsiz muhitda (sirka kislota, n-propanol, DMFA, DMSO) va ularning turli kislota-asos xususiyatlariga ega fon elektrolitlari bilan aralashmalarida nodir metallarni amperometrik titrlash shartlari va imkoniyatlari ko'rsatilgan. Ionlarni o'z ichiga olgan begona ionlar ishtirokida nodir metallar ionlarining mikrogram miqdorini amperometrik titrlash usullari ishlab chiqilgan.

Kalit so'zlar: palladiy, platina, kumush, oltin, IFCMDETK, MFCMDFD, eritma, sirka kislotasi, n-propanol, DMFA, DMSO, fon elektrolitlari.

Аннотация: В статье показаны условия и возможность амперометрического титрования благородных металлов растворами 4-метоксифенилкарбоксиметилдиэтил-дитиокарбамата (MFCMDETK) и 4-метоксифенилкарбоксиметилдиэтилдитиокарбазона (*MFCMDFD*) в неводных средах (уксусная кислота, н-пропанол, ДМФА, ДМСО) и их смесях с фоновыми электролитами, имеющими различные кислотно-основные свойства. Разработаны методы амперометрического титрования

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микрограммовых количеств ионов благородных металлов в присутствии посторонних ионов, содержащих ионы

Ключевые слова: палладий, платина, серебро, золото, IFCMDETK, *MFCMDFD*, раствор, уксусная кислота, н-пропанол, ДМФА, ДМСО, фоновые электролиты.

Introduction

Amperometric titration of metal ions in non-aqueous and mixed media with various complexants makes it possible to expand their analytical capabilities and simplify the solution of many complex analytical problems. First of all, this is due to the fact that the nature of the solvent strongly affects the strength of the resulting complex, and it is not the same for different cations, which determines the selectivity and rapidity of the method. In addition, non-aqueous complexiметry methods successfully solve the problem of accurate and selective determination of metals in objects of organic origin, as well as directly in extracts obtained by concentration.

We tried to find the optimal conditions for the amperometric titration of a number of noble metals with solutions of 4-methoxyphenyl-carboxymethyldiethyldithiocarbamate (MPKMDETK) and (4-methoxyphenylcarboxymethyl)-diphenylthiocarbazone (MPKMDFTK) in non-aqueous protolytic media, on background electrolytes with different acid-base properties. There are no data in the literature on the amperometric titration of various metal ions with solutions of the above reagents, since they were synthesized relatively recently [1] and, apart from their biological activity, their other properties have not yet been studied [2].

Reagents and apparatus

The initial 0.002 M solutions of Na_2PdCl_4, K_2PtCl_6, $AuCl_3$ и $AgNO_3$ as well as 0.01 M solutions of MFCMDETC and MFCMDFTC, were prepared by dissolving the corresponding weighed portions of these reagents in acetic acid (n-propanol, DMF, and DMSO). The concentration of noble metals was determined amperimetrically by 0.01 M potassium iodide solution. Amperometric titration was carried out on a setup with two platinum wire electrodes rotating (1000 rpm) on a common axis. The design of the electrodes, piston automatic microburette and apparatus are described in detail in [3].

Amperometric titration was carried out on a setup with two rotating (1000 rpm) electrodes on a common axis on a platinum wire. The design of the electrodes, piston automatic microburette and equipment are described in detail in [3].

In accordance with the voltammetric behavior of MFCMDETK, MFCMDFTK and other products involved in electrochemical media,

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amperometric titration of noble metal ions must be carried out at a polarization voltage of 0.75-1.15 V, depending on the nature and concentration of the background electrolyte (acetates, nitrates, chlorides, perchlorates of alkali metals and ammonium) [4]. In this case, the indicator current should occur beyond the equivalence point (ie) due to the oxidation of the free reagent and the reduction of dissolved oxygen in the air.

The experimental data showed that in the studied media and backgrounds, 0.15-0.40 M solutions of noble metal ions with solutions of MFCMDETC and MFCMDFTC are titrated quite well and quickly, and the shape of the curve coincides with the expected only with some constancy of the current at the beginning of titration, followed by a sharp transition (kink) at the end point of the titration (CTT).

Determination of noble metal ions in individual solutions.

It has been established that when titrating ions with the following noble metals, the corresponding molar ratio of Me:reagent is: Pd:reagent 1:2 and Pt:reagent 1:4, the titrated solution acquires a reddish-brown color. When passing from acetate backgrounds to perchlorate backgrounds containing some amount of perchloric acid, the shape of the titration curve for noble metal ions deteriorates significantly, which ultimately leads to a decrease in the reproducibility and correctness of the results. This is explained by an increase in the acidity of the analyzed medium upon passing from acetates to perchlorates [4]. Some of the data obtained are shown in Table 1.

The results of determining various concentrations of noble metal ions with a solution of MFCMDETK in 10.0 ml of the test solution under optimal conditions indicate a good accuracy of the developed technique. The influence on the correctness and reproducibility of the titration of noble metal ions of additives to acetic acid, n-propanol, DMF, DMSO of such foreign solvents as chloroform, carbon tetrachloride, benzene, toluene, hexane, methyl ethyl ketone, dioxane, etc., which are often used as extractants, has been studied. , as in the case of titration of noble metal ions in their individual solutions, with the only difference that the content of the protolytic solvent in the analyzed sample was regulated in strict accordance with the volume of the added inert solvent. Due to the decrease in the solubility of the background electrolyte under these conditions to values less than 0.2 M under the influence of large additions of an inert solvent, the background concentration (from 40-50 vol.% of an inert solvent) must be continuously reduced down to values of the order of 0.05 M. The addition of any of the above solvents in an amount of 10-20 vol.% (depending on the nature of the solvent) practically does not interfere with the shape of the titration curve becomes less steeply inclined

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to the volume axis. For the same reason, at solvent contents above 50-60 vol.%, the reproducibility and accuracy of the determinations of noble metal ions deteriorate.

Table 1.

Results of amperometric titration of various amounts of palladium (II) and platinum (IV) ions with a solution of MFCMDETC in DMSO against the background of 0.20 M lithium perchlorate

Introduced Me, mkg	Found Me, mkg (P=0,95; x±ΔX	n	S	S_r
Pd 15,44	15,42±0,16	3	0,06	0,004
Pt 123,50	123,61±0,20	4	0,10	0,001
Pd 247,00	246.91±0,41	4	0,21	0,001
Pt 493,10	493,80±0,52	3	0,62	0,001
Pd 740,71	739,45±1,43	4	0,91	0,001
Pt 998,10	987,91±1,54	4	0,63	0,001

The revealed nature of the influence of inert solvents on the shape of the titration curve is explained by the decrease in the electrical conductivity of the titrated solution at a high content of the inert solvent in the protolytic medium, leading to a significant and continuously increasing with increasing indicator current ohmic voltage in the analyzed solution.

Determination of silver (I) and gold (III) ions in model mixtures.

The possibility of amperometric titration of silver (I) and gold (III) with a solution of MFCMDFTK was tested on various artificial mixtures of salts (simulating natural and industrial materials) containing large amounts of other metals, often and widely accompanying these metals in nature. Silver (I) and gold (III) ions can be determined in two ways: 1) directly in an aliquot of the analyzed sample, with strict observance of all optimization conditions when assessing the degree of influence of various foreign cations, which also form strong complexes with the studied reagent; 2) a combination of preliminary extraction separation of silver (I) and gold (III) ions from other elements with their subsequent titration with a standard solution of MFCMDFTK in an aliquot of the obtained extract after the destruction of the extraction reagent and complex in it, as well as adding the necessary amounts of protolytic solvent and background (Table .2).

Table 2

Results of amperometric titration of various amounts of silver (I) and gold (III) ions with MPKMDFTK solution

Состав смеси, %	Найдено Me, мкг (P=0,95; x±ΔX	n	S	S_r

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Au(0,684)+Os(0,329)+Cd(39,42)+ Ni(48,18)+Co(10,06) +Ru(1,327)	Au, 0,675±0,224	3	0,09	0,133
Ag (0,342)+Ru(0,664)+Ir(0,954)+ Cu(59,81)+Mn(28,37)+Ti(9,86)	Ag, 0,338±0,075	4	0,03	0,089
Au(1,027)+Ru(0,534)+Ir(0,419)+ Cu(32,05)+ Mn(12,58)+Zn(53,39)	Au, 1,019±0,111	3	0,07	0,066
Ag(0,664)+Os(03,31)+Cd(39,40)+ Ni(48,20)+Co(10,03) +Ru(1,33)	Ag, 0,671±0,223	4	0,09	0,129

Consequently, amperometric procedures for the determination of silver (I) and gold (III) ions with a solution of MFCMDFTK are distinguished by high selectivity and reproducibility with a relative standard deviation not exceeding 0.133.

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