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Paper Authors

Safarova Guljakhon Eshtemirovna¹ Rakhmatov Khudoyor Boboniyozovich²





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AMPEROMETRIC TITRATION OF PALLADIUM (II) AND SILVER (I) IONS WITH A SOLUTION OF DIETHYLAMINO-4-METHYL-HEXINE-OLA-4 IN NONAQUE AND MIXED MEDIA

Safarova Guljakhon Eshtemirovna¹ Rakhmatov Khudovor Boboniyozovich²

Karshi Engineering and Economic Institute^{1,2}

Email: gsafarova990@gmail.ru,

Abstract: The article shows the conditions and the possibility of amperometric titration of noble metal ions with a solution of diethylamino-4-methyl-hexin-ol-4 in non-aqueous and mixed media (acetic acid, n-propanol, DMF, DMSO) and their mixtures with background electrolytes, having different acid-base properties. Methods of amperometric titration of micrograms of amounts of noble metal ions in the presence of foreign ions containing foreign ions are proposed.

Keywords.: palladium, silver, diethylamino-4-methyl-hexin-ol-4, solution, acetic acid, n-propanol, DMF, DMSO, background electrolytes.

Introduction

Amperometric titration of metal ions in non-aqueous and mixed media with various complexants will 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, moreover, it is not the same for different cations, which determines selectivity and rapidity of the method. In the methods of non-aqueous compleximetry 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 amperometric titration of a number of noble metals with a solution of diethylamino-4-methyl-hexin-ol-4 in non-aqueous protolytic media, on background electrolytes of different acid-base properties. There are no data in the literature on the amperometric titration of ions of various metals with solutions of the above reagents, since they were synthesized relatively recently and, in addition to their biological activity, their other properties have not yet been studied.

The initial 0.002 M solutions of Na₂PdCl₄, and AgNO₃, as well as a 0.01 M solution of diethylamino-4-methyl-hexin-ol-4,

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 using a 0.01 M potassium iodide solution. Amperometric titration was performed on a setup with two platinum wire electrodes rotating (1000 rpm) on a common axis. The design of electrodes, piston automatic microburettes and equipment and are described in detail in [1]

Amperometric titration was carried out on a setup with two rotating (1000 rpm) electrodes on a platinum wire on a common axis. The design of electrodes, automatic piston microburettes and equipment are described in detail in [2].

In accordance with the voltammetric behavior of diethylamino-4-methyl-hexin-ol-4 other products participating electrochemical media, amperometric titration of noble metal ions should be carried out at a polarization voltage of 0.751.15 V, depending on the nature and concentration of the background electrolyte (acetates, nitrates, chlorides, perchlorates of alkali metals and ammonium) [1]. In this case, the indicator current should arise beyond the equivalence point (i.e.) due to the oxidation of the free reagent and the reduction of dissolved oxygen in the air.



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The experimental data showed that in the studied media and backgrounds 0.15-0.40 M solutions of noble metal ions with a solution of diethylamino-4-methyl-hexin-ol-4 titrate fairly well and quickly, and the shape of the curve coincides with the expected one with some constancy current at the beginning of titration, followed by a sharp transition (kink) at the end point of titration (CTT).

Determination of noble metal ions in individual solutions. It was found that when titrating ions of the following noble metals, the corresponding molar ratio Me: reagent is: Pd: reagent 1: 2 and Ag: reagent 1: 1, the titrated solution acquires a reddish-brown color. When from acetate backgrounds perchlorate ones, containing a certain amount of perchloric acid, the shape of the titration curve of noble metal ions significantly deteriorates, which ultimately leads to a decrease in the reproducibility and accuracy of the results. This is explained by an increase in the acidity of the analyzed medium during the transition from acetates to perchlorates [3]. Some of the data obtained are shown in

Table 1.

The results of determining various concentrations of noble metal ions with a solution of diethylamino-4-methyl-hexin-ol-4 in 10.0 ml of the test solution under optimal conditions indicate a good accuracy of the developed method. The influence of additives to acetic acid, npropanol, DMF, DMSO, such as chloroform, tetrachloromethane, benzene, toluene, hexane, methyl ethyl ketone, dioxane, etc. and during titration of ions of noble metals in their individual solutions, with the difference that the content of protolytic solvent in the analyzed sample was controlled in strict accordance with the volume of the added inert solvent. Due to a decrease in the solubility of background electrolyte under 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 close to values of the order of 0.05 M.

Addition of any of the above solvents in the 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 to the axis of the volumes. For the same reason, at solvent contents above 50-60 vol.%. The reproducibility correctness and the determination of noble metal ions deteriorate.

Table 1

Results of amperometric titration of various amounts of palladium (II) and silver (I) ions with a solution of diethylamino-4-methylhexin-ol-4 in DMSO against the background of 0.20 M lithium perchlorate

Introduced	Found Me, μg	n	s	Sr
Ме, цд	(P=0,95; x±ΔX			
Pd 15,44	15,42±0,16	3	0,06	0,004
Ag 30,88	30,91±0,12	4	0,05	0,002
Pd 61,75	61,79±0,18	3	0,03	0,001
Ag 123,50	123,61±0,20	4	0,10	0,001
Pd 247,00	246.91±0,41	4	0,21	0,001
Ag 493,10	493,80±0,52	3	0,62	0,001
Pd 740,71	739,45±1,43	4	0,91	0,001
Ag 998,10	987,91±1,54	4	0,63	0,001

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

Determination of silver (I) and palladium (II) ions in model mixtures. The possibility of amperometric titration of silver (I) and palladium (II) with a solution of diethylamino-4-methyl-hexin-ol-4 was tested on various artificial mixtures of salts (imitating natural and industrial materials) containing large amounts of other metals, which are often widely associated with these metals. in nature. Silver (I) and palladium (II) ions can be



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determined in two ways: 1) directly in an aliquot of the analyzed sample under 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 palladium (II) ions from other elements, followed by their titration with a standard solution of diethylamino-4methyl-hexin-ol-4 in an aliquot of the obtained extract after the destruction of the extraction reagent and the complex in it, and also adding the necessary amounts of protolytic solvent and background.

Table 2

Results of amperometric titration of various amounts of palladium (II) and silver (I) ions with a solution of diethylamino-4-methylhexin-ol-4

The composition of the	Found Me,		s	Sr
mixture, %	μg (P=0,95; x±ΔX			
Pd(0,684)+Os(0,329)+Cd(39,	Pd,	3	0,09	0,133
42)+ Ni(48,18)+Co(10,06)	0,675±0,224			
+Ru(1,327)				
Ag (0,342)+Ru(0,664)+	Ag.	4	0,03	0,089
<u>Ir</u> (0,954) +Cu(59,81)+Mn(28,37)	0,338±0,075			
+ <u>Ti</u> (9,86)				
Pd(1,027)+Ru(0,534)+Ir(0,41	Pd,	3	0,07	0,066
9)+ Cu(32,05)+ Mn(12,58)+	1,019±0,111			
Zn(53,39)				
Ag(0,664)+Os(03,31)+Cd(39,	Ag.	4	0,09	0,129
40)+ Ni(48,20)+Co(10,03)	0,671±0,223			
+Ru(1,33)				

Consequently, amperometric methods for the determination of silver (I) and palladium (II) ions with a solution of diethylamino-4-methylhexin-ol-4 are distinguished by high selectivity and reproducibility with a relative standard deviation not exceeding 0.133.

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