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ELECTROCHEMICAL DETERMINATION OF PLATINUM (IV) WITH SOLUTIONS OF DIETHYLAMINO-4-METHYL-HEXINE-2-OLA-4 IN AQUEOUS AND MIXED MEDIA

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ABSTRACT

The article shows the conditions and the possibility of amperometric titration of palladium (II)ions with solutions of diethylamino-4-methyl-hexine -2-ol-4 (DEMGO) in non-aqueous media(acetic acid, n-propanol, DMF, DMSO) and their mixtures with background electrolytes havingdifferent acid-base properties. Methods of amperometric titration of micrograms of amounts ofpalladium (II) ions in the presence of foreign ions containing foreign ions are proposed

KEYWORDS: *Palladium, Diethylamino-4-Methyl-Hexin-2-Ol-4, Solution, Acetic Acid, Npropanol, 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 the selectivity and rapidity of the method. In addition, the methods of non-aqueous complexim etry successfully solve the problem of accurate and selective determination of metals in objects of organic origin, as well as directly in extracts obtained during concentration.

We tried to find the optimal conditions for amperometric titration of a number of noble metals with solutions of diethylamino-4-methyl-hexin-2-ol-4 (DEMGO) in non-aqueous protolytic media, on background electrolytes of different acid-base properties.

Reagents and equipment

The initial 0.002 M solutions of Na2PdCl4, K2PtCl6,AuCl3and AgNO3, as well as a 0.01 MDEMGO solution, were prepared by dissolving the corresponding weighed portions of thesereagents in acetic acid (n-propanol, DMF, and DMSO). The concentration of noble metals wasdetermined amperimetrically using a 0.01 M potassium iodide solution [1]. Amperometrictitration was carried out on a setup with two platinum wire electrodes rotating (1000 rpm) on acommon axis. The design of electrodes, piston automatic microburettes and apparatus aredescribed in detail in [2].

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

In accordance with the voltammetric behavior of DEMGO and other products participating inelectrochemical media, amperometric titration of noble metal ions must be carried out at apolarization voltage of 0.75-1.15 V, depending on the nature and concentration of thebackground electrolyte (acetates, nitrates, chlorides, alkali metal perchlorates and ammonium)[4]. In this case, the indicator current should arise beyond the equivalence point (i.e.) due to theoxidation of the free reagent and the reduction of the dissolved oxygen in the air.

The experimental data showed that in the studied media and backgrounds 0.15-0.40 M solutionsof noble metal ions with DEMGO solutions are titrated quite well and quickly, and the shape ofthe curve coincides with the expected one with some constant current at the beginning of titrationwith a subsequent sharp transition (break) at the end point of titration (CTT).

Determination of ions and platinum (IV) 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 Pt: reagent 1: 4, the titrated solution acquires a reddish-brown color. When passing from acetate backgrounds to perchlorate ones, containing a certain amount of perchloric acid, the shape of the titration curve of noble metal ions deteriorates significantly, 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 [5]. Some of the data obtained are shown in the table.

TABLE RESULTS OF AMPEROMETRIC TITRATION OF VARIOUS AMOUNTS OF PLATINUM (IV) IONS WITH A DEMGO SOLUTION IN DMSO AGAINST A BACKGROUND OF 0.20 M LITHIUM PERCHLORATE

Mixture composition,%	Found Me, μg (P=0,95; x ± ΔX	N	S	S_r
Pd 15,44	15,423±0,16	3	0,061	0,004
Pd 30,88	30,854±0,12	4	0,052	0,002
Pd 61,75	61,782±0,18	3	0,033	0,001
Pd 123,50	123,494±0,20	4	0,102	0,001
Pd 247,00	246.951±0,41	4	0,213	0,001
Pd 493,10	493,793±0,52	3	0,624	0,001

The results of the determination of various concentrations of noble metal ions with a DEMGO solution in 10.0 ml of the test solution under optimal conditions testify to the good accuracy of the developed technique. The effect of additives to acetic acid, n-propanol, DMF, DMSO, such as chloroform, tetrachloromethane, benzene, toluene, hexane, methyl ethyl ketone, dioxane, etc. as in the titration of noble metal ions in their individual solutions, with the only 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 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 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 and accuracy of determinations of noble metal ions deteriorate.

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, which leads to a significant and continuously increasing ohmic voltage drop in the analyzed solution with an increase in the indicator current.

Consequently, amperometric methods for the determination of platinum (IV) ions with a DEMGO solution are distinguished by high selectivity and reproducibility with a relative standard deviation not exceeding 0.133.

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