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ELECTROCHEMICAL TITRATION OF PALLADIUM IN NON-AQUEOUS MEDIA

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ABSTRACT

The article shows the conditions and the possibility of amperometric titration of noble metal ions with solutions of 4-methoxyphenylcarboxymethyl-diethyldithiocarbamate (MFKMDETC) and 4-methoxy phenylcarboxy methyldithio-carbamate (MFKMDFTC) in non-aqueous and mixed media (n-acetic acid), DFA with background electrolytes with 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, Mpkmdetk, Mpkmdftk, 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 the selectivity and rapidity of the method. In addition, the methods of non-aqueous

complexiметry 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 the amperometric titration of a number of noble metals with solutions of 4-methoxyphenyl-carboxymethyldiethyldithiocarbamate (MFKMDETC) and (4-methoxyphenylcar-boxmethyl) -diphenylthiocarbazone (MFKMDFTK) in various non-aqueous protolytic acids, on basic electrolyte media. 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 [1] and, in addition to their biological activity, their other properties have not yet been studied [2].

Reagents and equipment. The initial 0.002 M Na_2PdCl_4 solutions, as well as 0.01 M solutions of MPKMDETK and MPKMDFTK 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 apparatus are described in detail in [3].

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 [3].

In accordance with the voltammetric behavior of MPKMDETK, MPKMDFTK and other products participating in electrochemical media, 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 arise beyond the equivalence point (i.e.) due to the oxidation 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 solutions of noble metal ions with solutions of MPKMDETC and MPKMDFTK titrated quite well and quickly, and the shape of the curve coincides with the expected one with some constancy of current at the beginning of titration, followed by a sharp transition (kink) at the titration endpoint (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 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 [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 MPKMDETK in 10.0 ml of the test solution under optimal conditions indicate the good accuracy

of the developed method. 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 the 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.

TABLE 1 RESULTS OF AMPEROMETRIC TITRATION OF VARIOUS AMOUNTS OF PALLADIUM (II) IONS WITH A SOLUTION OF MPKMDETC IN DMSO AGAINST THE BACKGROUND OF 0.20 M LITHIUM PERCHLORATE

Mixture composition,%	**Found Me, μg (P=0,95; x±ΔX**	**n**	**S**	**S_r**
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 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.

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