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AMPEROMETRIC TITRATION OF PALLADIUM WITH DIETHYLAMINO-4-METHYL-HEXINE-2-OLA-4 SOLUTIONS IN NONAQUEOUS ENVIRONMENTS

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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 having different acid-base properties. Methods of amperometric titration of micrograms of amounts of palladium (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, 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 compleximetry 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 Na_2PdCl_4 , K_2PtCl_6 , AuCl_3 and AgNO_3 , as well as a 0.01 M DEMGO solution, 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 amperometrically using a 0.01 M potassium iodide solution [1]. Amperometric titration was carried out 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 [2].

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, piston automatic microburettes and equipment are described in detail in [3].

In accordance with the voltammetric behavior of DEMGO 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, alkali metal perchlorates 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 DEMGO solutions are titrated quite well and quickly, and the shape of the curve coincides with the expected one with some constant current at the beginning of titration with a subsequent sharp transition (break) at the end point of titration (CTT).

Determination of palladium ions in model mixtures. The possibility of amperometric titration of palladium with DEMGO solutions has been tested on various artificial mixtures of salts (imitating natural and industrial materials) containing large amounts of other metals, often and widely associated with palladium in nature. Palladium 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 reagents; 2) a combination of preliminary extraction separation of palladium ions from other elements followed by their titration with a standard DEMGO solution in an aliquot of the obtained extract after the destruction of the extraction reagent and the complex in it, as well as the addition of the required amounts of protolytic solvent and background (table).

TABLE RESULTS OF THE DETERMINATION OF PALLADIUM IONS WITH A SOLUTION OF DIETHYLAMINO-4-METHYL-HEXIN-2-OL-4

Mixture composition, %	Found Me, μg ($P=0,95$; $x\pm\Delta X$)	n	S	S_r
Pd(0,684)+Os(0,329)+Cd(39,42)+Ni(48,18)+Co(10,06) +Ru(1,327)	Pd, 0,6765 \pm 0,223	3	0,087	0,133
Pd(0,341)+Ru(0,664)+Ir(0,954)+Cu(59,81)+Mn(28,37)+Ti(9,86)	Pd 0,337 \pm 0,076	4	0,028	0,089
Pd(1,021)+Ru(0,534)+Ir(0,419)+Cu(32,05)+ Mn(12,58)+Zn(53,39)	Pd, 1,019 \pm 0,113	3	0,069	0,066
Pd(0,667)+Os(03,31)+Cd(39,40)+Ni(48,20)+Co(10,03) +Ru(1,33)	Pd 0,67 \pm 0,221	4	0,086	0,129

Consequently, amperometric methods for the determination of palladium 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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