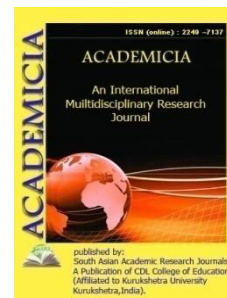




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**ELECTROCHEMICAL DETERMINATION OF MERCURY (II) WITH A
 MFCMDEDTK SOLUTION IN THE MEDIUM OF
 DIMETHYLPHARMAMIDE**

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ABSTRACT

The article studies the voltammetric behavior of MPCMDEDTK in dimethylpharmamide (DMF) in the presence of background electrolytes of different acid-base properties in the anodic region of polarization of a platinum microdisk electrode. MPKMDEDTK is oxidized against the background of 0.125 M lithium perchlorate, forming one clearly pronounced anodic wave with $E_1 / 2 = 0.85$ V, while the wave height changes in proportion to the depolarizer concentration. Amperometric titration of mercury (II) with two indicator electrodes with a standard solution of MPKMDEDTK in DMF medium is proposed.

KEYWORDS: *Mercury (II), 4-Methoxyphenyl Carboxymethyl Diethyl Dithio Carbamate, Solution, Lithium Perchlorate, N-Propanol, Dimethylpharmamide, Microdisk Electrode, Indicator Electrode, Half-Wave Potential, Depolarizer, Background Electrolytes*

INTRODUCTION

Earlier, some features of the amperometric titration of a number of cations in glacial acetic acid [1-2] and in n-propanol [3-4] were found. It was of interest to investigate the possibility of amperometric titration with a solution of 4-methoxyphenylcarboxymethyl-

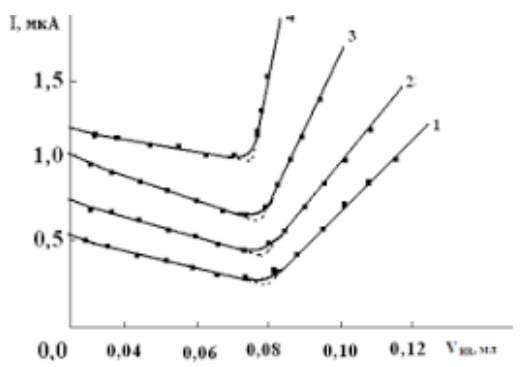
diethyldithiocarbamate (MPCMDEDTC) in a medium of non-aqueous solvents with acid-base properties.

This work is devoted to the study of the possibility of amperometric titration of mercury (II) with two indicator electrodes with a solution of MPCMDEDTC in the medium of an aprotic dipolar solvent widely used in electrochemical studies - dimethylpharmamide (DMF), which exhibits weak basic properties.

The initial 0.002 M solution of mercury (II) nitrate was obtained by dissolving a weighed portion of $\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$ of chemically pure grade. in DMF and was amperometrically standardized against an aqueous solution of KI [5]. Solutions of lower concentrations were obtained by diluting the original immediately before their use. A standard 0.005 M solution of MPKMDEDTC was prepared according to an accurate weighed portion of the reagent. Stock solutions of supporting electrolytes (10% LiClO_4 ; 7.5% CH_3COONa and CH_3COOH) were prepared by dissolving the corresponding salts in DMF.

Amperometric titration with two indicator rotating platinum electrodes was performed using the setup described in [6]. The titrant is a piston microburette with an accuracy of 0.0005 ml. the final volume of the solution prepared for titration was 10.0 ml.

To obtain the voltammetric curves of MFKMDEDTK, a complex of mercury (II) with MFKMDEDTK, and free ions of mercury (II) at anodic and cathodic polarization of a platinum microdisk electrode ($l = 1.0$ mm), a three-electrode cell and a PPT-1 polarograph were used; we served as a reference electrode. k.e.



Pic. Hg (II) AT curves with a solution of MPKMDEDTA in the presence of various concentrations of LiClO_4 in a mixture of CH_3COOH and CHCl_3 (1: 1).

LiClO_4 content, mol / l:

1 - 0.031; 2 - 0.062;

3 - 0.125; 4 - 0.250.

Taking into account the voltammetric behavior of all components of the formation reaction of mercury (II) complexonate, it can be concluded that the amperometric titration of mercury (II) should be carried out at a half-wave potential in the range of $Y_{1/2} = 0.80 - 0.90$ V. Under these conditions, up to the point equivalence, the indicator current will be due to the simultaneous reduction of titratable mercury (II) ions at the cathode and oxidation of the resulting mercury (II) complexonate at the anode. Since in this case the concentration of mercury (II) will continuously fall from its initial to almost zero value, and the concentration of mercury (II) complexonate, on the contrary, will increase, the indicator current will first rise to a certain maximum value, which is reached at the moment of titration of about half of all mercury. (II), and then fall until a continuously increasing current is observed, due to the reduction of mercury (II) complexonate at the cathode and oxidation of free MPCMDEDTC at the anode.

Experiments have fully confirmed this proposal. When titrating mercury (II) against the background of 0.125 M LiClO₄, a fairly clear titration curve (Fig.) Of the above-described form is obtained. The smooth curve rounding in the vicinity of the titration end point indicates a slightly lower strength of mercury (II) complexonate in DMF as compared to its strength in water. The end point of the titration, determined by the conventional graphical method, corresponds to the formation of a normal complex of mercury (II) with MPKMDEDTC with a ratio of mercury (II): MPKMDEDTC = 1: 1. In contrast to solutions containing lithium perchlorate as a background, solutions of mercury (II) nitrate against the background of sodium acetate and acetic acid are titrated much worse: the titration curve turns out to be less clear and the time to reach equilibrium after each addition of the titrant sharply increases. This is due to the ability of mercury (II) to form very strong complexes with acetate ions.

TABLE RESULTS OF AMPEROMETRIC TITRATION OF MERCURY (II) WITH 0.005 M SOLUTION OF MPKMDEDTC IN DMF ($E_{1/2}=0,85; V =10,0; 0,125 \text{ M LiClO}_4$)

<i>Introduced Hg (II), μg</i>	<i>Found Me, μg ($P = 0,95; \bar{x} \pm \Delta X$)</i>	<i>n</i>	<i>S</i>	<i>S_r</i>
25,48	25,43±0,52	3	0,21	0,008
50,96	51,23±0,27	3	0,11	0,002
101,9	101,4±0,54	4	0,34	0,003
231,4	234,0±0,41	4	0,26	0,001
462,7	462,8±0,90	3	0,36	0,001

In order to assess the reproducibility and correctness of the developed technique, various amounts of mercury (II) were titrated against the background of 0.1 M lithium perchlorate with 3-fold repetition of each determination. The results of titration of mercury (II) with a standard solution of MPKMDEDTC are shown in the table. The difference between the taken and found amounts of mercury (II) in all cases does not go beyond the confidence interval, which indicates the absence of systematic errors.

Amperometric titration of mercury (II) with a solution of MPKMDEDTK can find application in the analysis of some paints, pharmaceutical and perfumery mercury-containing preparations that are poorly soluble in water.

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