Titration of Cu (II) IONS WITH SOLUTIONS of ORGANIC REAGENTS

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ABSTRACT

In the development of methods of amperometric titration of different metal ions mixed with one or two solid indicator electrodes allows to know the voltamperometric nature of the ions of the detected metals, the reagent used and its metal complexes and to select the optimal conditions for this titration.

As in any other medium, the rate of formation of Cu(II) ion complexes in acetic acid medium decreases with increasing concentration of lionium (hydrogenated ions) ions. This factor allows to control the selectivity of complexonometric titration of metal ions by controlling the acidity of the solution.

The concentration of lion ions in anhydrous acetic acid solutions (CH_3COOH) can be controlled by selecting background electrolytes. For example, in an acetic acid environment, potassium and sodium acetates exhibit strong basic properties and neutralize lion ions to the following reaction:

$$CN_3\,COON^+_2 + CN_3\,COO^- \rightarrow 2CN_3COON.$$

Therefore, cations with low strength of acetate complexes can be titrated. Conversely, in such media, lithium perchlorate exhibits acidic properties and increases the concentration of lion ions by the following reaction:

$$LiCIO_4 + 2CH_3COOH \rightarrow CH_3COOLi + CH_3COOH_2^+ + CIO^-_4 ,$$
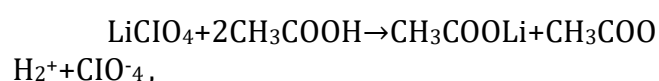
The balance shifts to the right. Therefore, only cations of metals that form stable complexes in lithium perchlorate can be titrated. As mentioned above, ions of precious metals are cations that form strong complex compounds with sulfur and nitrogen-containing reagents, so the possibility of titrating them in an anhydrous acetic acid medium was tested. The voltage effect was studied on the basis of the voltammeter properties of diphenylthiocarbazone and dithiocarbamate reagents obtained in a platinum disk microanode in the presence of various background electrolytes in acetic acid.

The amperometric value of the end point of titration of different metals with two indicator electrodes is 0,50-0,65 V for titration, respectively; The background voltage used at 0,90-1,25 V and 0,50-0,85 V should be in the potential range, depending on the nature of the electrolyte.

If the acidity of the ion solution is high, the potential applied to the indicator electrode during titration should be increased to 0,1-0,2 V.

When metal ions are titrated on two indicator electrodes, the voltage at the platinum electrodes must be 0,40 V, because the reagents must be oxidized at the anode (E min = 0,60 V) and oxygen is returned at the cathode (Emakc = 0,2 V). If the voltage exceeds the minimum value by 0,2-0,3 V, the oxidation of the metal complex at the anode and the return of the titrated cations at the cathode during Hg (II) titration occur simultaneously before reaching TON [1].

In this case, the amperometric titration curve is formed in the form with the maximum in the left branch.

Experimental results show that it is possible to titrate metal ions and that the assumptions about the shape of the titration curves are correct. Cu(II) titrates rapidly and accurately, and has good resilience, with a relative standard deviation of 0,061 [2-3].

According to experiments, during the titration of metal ions in acetic acid, complexes with a content of - Me: Reagent =1:1 for monovalents, -1:2 for divalent, -1:3 for trivalent metals are formed, in which the reagent is a single electron for RK, this ratio is expressed in the ratios Me: Reagent =2:1 for monovalents, 1:1- for divalent and 2:3- for trivalent metals. The proportionality between the amounts of metals obtained (especially from the titration of metal ions with diphenylthiocarbazone and dithiocarbamate) and the volumes of reagents consumed is well maintained.

Table-1
The voltage was 13,00 mcg of Cu (II) diphenylthiocarbazone
and amperometric with solutions of diethyldithiocarbamate in acetic acid

effect on vibration results (n = 5)

Volt age valu e, V	Found Me, mcg					
	diphenylthiocar bazon			diethyldithiocarb amate		
	Cu(II)	S	Sr	Cu(II)	S	Sr
0,30	13,58 ± 0,47	0,47	0,0262	13,49 ± 0,48	0,42	0,025

The results of the study of the effect of metal ions on the shape and results of amperometric titration curves with diphenylthiocarbazone and dithiocarbamate solutions were carried out under optimal conditions: background – 0,15 M lithium nitrate, titrant concentration - 0,001 M and 0,05 M, volume of the analyzed solution – 10 ml, temperature 20 ^{0}S, solvent - anhydrous acetic acid or other protolytic solvent. The voltage at the indicator electrodes was changed in steps of 0,2 V in the range of 0,1-1,1 V. The titration was repeated 3-5 times to study the magnitude of the voltage [4-5].

A change in the magnitude of the voltage across the electrodes over a given range affects the length of its straight line sections and the slope of both branches of the titration curve, but does not affect the results of the titration of Cu(II) [6].

The results show that the average consumption of the reagent at the end point of titration (TON) does not depend on the voltage value of the indicator electrodes. However, at its small values (less than 0,3V), the right branch of the curve becomes very flat and quickly loses its accuracy, which adversely affects the repeatability and accuracy of the results obtained. rsatadi. In the range of values obtained of the voltage, the indicator current returns the titrated cation at the cathode at the equivalent point, oxidizing the water at the anode. After TON, the diffusion current increases due to the return of oxygen at the cathode and the oxidation of the free titrant at the anode [7].

Depending on the shape of the amperometric titration curves, the best is observed in the voltage range of 0,50-0,75 V

(depending on the nature of the background and the vibrating medium). When the potential value is high, along with the simultaneous oxidation of the reagent at the electrodes, other additional processes take place, which have a strong effect on the useful AS. Therefore, the optimum voltage values for titration of metal ions in the selected media should be kept within the studied potential values.

The titration conditions used for this purpose were as follows: voltage 0,5-0,65V (depending on the nature of the background and the nature of the proteolytic solvent), the volume of the solution under study -10,0 ml; temperature   -18-20 ^{0}S;   the   titrant concentration is 0,01 M. The amount of electrolyte in the solution under study varied in the range of 0,025-0,5 M.

Experiments have shown that when the Cu (II) ion is amperometrically titrated with a diphenylthiocarbazone solution, a small amount (less than 5%) of secondary complexes is formed in addition to the primary carbons in the acetic acid environment against the background of potassium acetate, resulting in much lower titrant consumption.

Table-2
The effect of the nature and concentration of the background electrolyte on the results of amperometric titration of 15.63 mcg Cu (II) in diphenylthiocarbazone solutions in acetic acid on different backgrounds (n = 5)

Background amount mol/l	Topildi Me, mcg					
	Lithium perchlorate			Potassium acetate		
	diphenylthiocarbazon			diethyldithiocarbamate		
	Cu(II)	S	S_r	Cu(II)	S	Sr
0,025	15,52 ± 0,47	0,47	0,025	15,45 ± 0,46	0,42	0,026
0,050	15,46 ± 0,53	0,46	0,029	15,35 ± 0,39	0,33	0,024

As can be seen from the table, the optimal background concentration in solution

for amperometric titration of metal ions is 0,05 to 0,2 M.

To solve the problem of the possibility of using the amperometric method developed for the determination of Cu(II), it is necessary to study the effect of the nature and concentration of the inert solvent added. The effect of the addition of chloroform, carbon tetrochloride, benzene, hexane, methyl ethyl ketone and other inert solvents on the titrated solution shows that the slope of the titration curve after the equivalence point increases in all cases with increasing concentration of inert solvents. can be seen from the increase in the ECU of the complex.

Experiments show that when an inert solvent is added to acetic acid, first the shape of the titration curve does not change, and then its slope decreases. However, when 30 and even 40-50 % of the volume is solvent, the curve is clearly visible, and when the volume of solvent exceeds 50 %, the right branch of the titration curve is flattened and straight. will have a linear segment.

As a result of the rapid development of physicochemical methods in analysis, the development of sensitive, selective, efficient and inexpensive methods for the detection of copper and gold using organic reagents in complex objects is one of the current problems of analytical chemistry. For amperometric titration of metals in real objects, it is important to modify the carbon electrode with organic reagents that form a complex with a wide range of metals - hydrazine sulfate, thioacetamide, EDTA, dithizone, sodium diethyldithiocarbamate, diphenylcarbazide, 8-oxyquinoline. It is known that in amperometric titration, the diffusion current depends on the concentration of the active substance. Titration of metals with diphenylthiocarbazone and dithiocarbamate in the range of pH 1,60-12,50 HCl, $HClO_4$, H_2SO_4, KCl, NaCl background solutions   and   Britton-Robinson   buffer solutions current voltage 0,75 V, the volume of the analyzed solution is 10 ml, temperature 25 °S, the titrant concentration was carried out at 0,01 M. In this case, the concentration of metals was determined in the range of 10-40 mcg. The results of the experiments were processed

using the rules of mathematical statistics, and the amperometric method developed was metrologically evaluated.

The pH dependence of the complex of metals with organic reagents was studied, taking into account that each chemical reaction takes place in a specific environment. Based on the obtained optimal conditions, the subordination of the complex to the electrochemical laws was studied in order to apply it to the quantitative determination of the metal. According to him, the area of compliance of metals with the law of electrochemical laws is observed in the range of 0,7-40 mcg. In this case, the interfering ions were masked with masking reagents.

Acetic acid and n-propanol have hydrogen bonds (in protogenous solvents they are more pronounced due to the strong electronegativity of the carbonyl group oxygen).

In DMFA or DMCO, the molecules are in the form of chains, and the metal ions present in such solvents are coated with a polymer film formed by the molecules of the protophilic solvent. Therefore, when titrating metal ions with various chelating agents, in particular DDTKNa and (DDTK) 2Rb solutions, the attack of DDTK-ions on metal ions in protophilic solvents is difficult.

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