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## DETERMINATION OF Z-HYDROGEN N. N-DIETHYLDITHIOCARBAMATE SODIUM AND NON-HYDROGEN SOLUTION

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### Abstract

The article presents the results obtained in determining the dielectric constant, electrical conductivity and viscosity of solutions containing Sb(III) and Bi(III) ions, individual solvents and their mixtures containing indifferent salts of different nature: potassium acetate, sodium, lithium nitrate and perchlorate and sodium, used as background electrolytes in determining microconcentrations of the studied ions, optimizing the conditions for determining Sb(III) and Bi(III) ions with various reagents.

**Keywords:** *dielectric constant, electrical conductivity, viscosity, antimony, bismuth, voltammetry*

### Introduction

The content of microelements in waters and industrial waste products usually does not exceed 0.00001%, therefore, their reliable determination requires the use of highly sensitive analytical methods, and therefore instrumental methods of analysis. The choice of an instrumental method of analysis for the determination of microelements in waters and industrial waste products is determined by a number of the following characteristics of the method: the required sensitivity and accuracy of the method, the number of elements to be determined and objects of analysis, the availability of certified and sponsored analysis methods, ease of analysis, and the cost of the analyzer

(Yakhshieva, M. Sh., 2015; Yakhshieva Z. Z., Kalonov R. M., 2020).

Voltammetric methods of analysis are based on deciphering polarization curves – voltammograms obtained in an electrochemical cell filled with a solution of the analyzed sample. A new impetus for the widespread use of voltammetry was given by the need to control the content of toxic microelements in food products.

The high sensitivity of the method is achieved by the preliminary electrochemical concentration of the analyte on the surface of the indicator electrode.

The analytical signal is the maximum dissolution current of the concentrate of the element being determined, recorded on the vol-

tammogram in the form of a peak or several peaks if several elements are concentrated and determined simultaneously. The position of the peak on the potential axis characterizes the nature of the component being determined, and the height or area is proportional to its concentration in solution under constant conditions of electron accumulation.

Since by selecting appropriate conditions (solvent, electrode material, reagents, etc.) a wide variety of substances can be converted into an electroactive state, methods have now been developed for the voltammetric determination of Sb(III) and Bi(III) ions in the concentration range from  $10^{-3}$  to  $10^{-11}$  M.

The advantages of a modern voltammetric analyzer (polarograph) are relative ease of operation, low cost; high sensitivity; sufficient selectivity and rapidity of determination; the ability to automate the process of measuring the analytical signal (Gevorgyan, A.M. Ziyaev, D.A. Kireeva, G.V., 2010; Ziyaev D.A., 2021; Shaidarova L.G., Chelnokova I.A., Gafiatova I.A., Gedmina A.V., Budnikov G.K., 2022; Aronbaev S.D., Aronbaev D.M., Narmaeva G.Z., 2018).

During the experiments, the dishes were pre-soaked for a day in deionized water. After this, rinse 2–3 times.

Solutions were prepared in double-distilled water using twice-recrystallized thiocarbamide  $(\text{NH}_2)_2\text{CS}$  ("pure for analysis"), disodium salt of ethylenediaminetetraacetic acid  $\text{Na}_2\text{H}_2\text{EDTA}$  in the form of a standard titer, and recrystallized  $\text{NaNO}_3$ . A solution of bismuth (III) perchlorate was prepared by dissolving a weighed portion of bismuth (III) oxide ("reagent grade") in perchloric acid ("reagent grade"). Measurements of the acidity of electrolytes were carried out at a temperature of  $20 \pm 0.1$  °C on a pH-150M device with an ES-10601/7 glass electrode.

Anodic dissolution and cathodic reduction of bismuth ionization products were studied in solutions: 1) containing only the disodium salt of ethylenediaminetetraacetic acid ( $\text{Na}_2\text{H}_2\text{EDTA}$ ); 2) in the combined presence of  $\text{Na}_2\text{H}_2\text{EDTA}$  and thiocarbamide (Thio) of various concentrations; 3) in the combined presence of  $\text{Na}_2\text{H}_2\text{EDTA}$  and thiocarbamide (Thio) of varying concentrations and a constant concentration of bismuth (III) chlorate. All studies were carried out at pH 6.0. The choice of

medium acidity is not random. As an analysis of literature data shows, at  $\text{pH} < 7$ , complexes with dissimilar ligands of the composition (Bi EDTAThio)– (Yakhshieva Z.Z., Kalonov R.M., 2020; Bakahonov A.A. Yakhshieva Z.Z., Kalonov R.M., 2020) are formed in the bulk solution.

Antimony solutions were prepared in bidistilled water using the following reagents:  $\text{Sb}_2\text{O}_3$  (reagent grade);  $\text{HClO}_4$  ("reagent grade"); sodium thiosulfate  $\text{Na}_2\text{S}_2\text{O}_3$  (analytical grade), disodium salt of ethylenediaminetetraacetic acid  $\text{Na}_2\text{H}_2\text{EDTA}$  in the form of standard titer, recrystallized  $\text{NaNO}_3$  (analytical grade). The study was carried out against a background of 0.5 M  $\text{NaNO}_3$  at pH 6.0. Measurements of the acidity of electrolytes were carried out with an accuracy of  $\pm 0.05$  pH units on a pH-150M device with an ES-10601/7 glass electrode, previously calibrated using standard buffer solutions. The acidity of the solutions was adjusted to the required value using solutions of  $\text{HNO}_3$  and  $\text{NaOH}$  (reagent grade).

When determining antimony and bismuth ions, we used a voltammetric analyzer TA-Lab, which is designed for highly sensitive measurements of the content of As, Cd, Pb, Cu, Zn, Mn, Hg, Se, I, Ni, Co, Fe, Ag, **Sb**, **Bi**, Fe ions, Sn in drinking, natural, wastewaters, aqueous solutions of soil samples, food products, industrial concentrates.

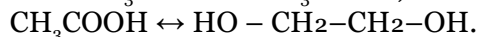
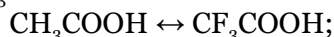
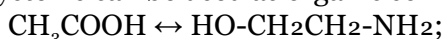
To successfully carry out the complexometric titration of Sb(III) and Bi(III) ions in non-aqueous media, we chose a solution of sodium N, N-diethyldithiocarbamate, which is a water-soluble salt of the organosulfur composition of pale yellow color with the formula  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na} \cdot 3\text{H}_2\text{O}$  (Yakhshieva Z.Z., Kalonov R.M., Abdurahmonov B., 2020).

Since there are no quantitative data in the literature on the solubility of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na} \cdot 3\text{H}_2\text{O}$  and its salts in various organic solvents, it was necessary to determine the solubility of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na} \cdot 3\text{H}_2\text{O}$  in  $\text{CH}_3\text{COOH}$ ,  $\text{C}_2\text{H}_5\text{OH}$ , dimethylformamide (DMFA),  $\text{CF}_3\text{COOH}$ ,  $\text{CH}_3\text{CN}$ ,  $\text{CH}_3\text{CH}(\text{OH})\text{CH}_3$ ,  $\text{HO}-\text{CH}_2\text{CH}_2-\text{NH}_2$  and with their mixtures  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CCl}_4$ ,  $\text{C}_6\text{H}_{14}$ ,  $\text{CH}_3-\text{CO}-\text{CH}_2-\text{CH}_3$ ,  $\text{HO}-\text{CH}_2-\text{CH}_2-\text{OH}$ ,  $\text{CH}_3-\text{C}(\text{O})-\text{CH}_3$  and  $\text{C}_4\text{H}_8\text{O}_2$ .

Experiments have shown that the highest solubility of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na} \cdot 3\text{H}_2\text{O}$  and its salts

without the addition of indifferent electrolytes is observed in DMFA,  $\text{CF}_3\text{COOH}$ , and in a mixture of  $\text{HO-CH}_2\text{CH}_2\text{-NH}_2$  and  $\text{HO-CH}_2\text{-CH}_2\text{-OH}$ . It has been established that under the influence of additives of inert solvents, the solubility of the organic reagent  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  decreases significantly.

Thus, when preparing standard solutions of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  and its salts, the following systems can be used as organic solvents:



However, the solubility of the titrants used in protolytic media and their mixtures with other solvents is not high enough ( $10^{-3}$  M) and therefore it is necessary to find a way to prepare at least  $5 \cdot 10^{-3}$  M  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  and its salts in anhydrous acetic acid.

To solve this problem, we took advantage of the well-known fact that the solubility of many electrolytes both in water and in organic solvents increases under the influence of the high ionic strength of the solution, caused by the presence of foreign indifferent salts in it (Lebedeva M. I., Isaeva B. I., Borisova R. V., 2012). In the protolytic solvents used, this effect is much stronger than in the case of water (Denesh I., 2003). The solubility of the titrant in organic solvents also strongly depends on the dielectric constant of the solvent itself (Ahadov Ya. Yu., 2007) and is expressed by the equation:

$$\lg S_m = \text{const} + (\beta_\mu^1 - \beta) \cdot \frac{1}{D_M} \quad (1)$$

where  $S_m$  – solubility in non-aqueous solvent ( $\text{mol}/\text{m}^3$ );  $\beta_\mu^1, \beta$  – permanent ( $\text{F} \cdot \text{mol}/\text{m}^2$ );  $D_M$  – solvent dielectric constant ( $\text{F} \cdot \text{m}$ );  $\mu$  – dipole moment (Kl.m.).

From this we can conclude that the solubility of ions of the metals and complexants under study is a linear function of the solvent. Magnitude  $\beta_\mu^1 - \beta$ , as rule, has a negative value and solubility decreases with decreasing dielectric constant.

The solubility of a substance also depends on the ratio of the polarities of the solute and solvent. It will be maximum when the ratio of both components – solute and solvent is close to unity:

$$\frac{\mu_{1\text{of}}}{\mu_{2\text{of}}} \approx 1, \text{ where } \mu_{\text{of}} = \frac{M}{V} \quad (2)$$

where  $V$  – is the volume of the molecule

The solubility of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  increases with increasing concentration of background electrolytes. It was experimentally found that when the content of potassium acetate (1.5–1.6 M) the solubility of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  in anhydrous acetic acid increases so much that even 0.01–0.02 M solutions of this reagent can be easily prepared. Under the influence of lithium perchlorate, its solubility increases, but in this case, at a 1.5 M content of this salt, a 0.01 M solution of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  can be prepared.

Thus, using the above-mentioned indifferent salts to effectively increase the solubility of  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  and its salts, it is possible to obtain standard 0.004–0.02 M solutions of reagents, with which, provided that a 2–5 ml piston microburette is used, it is possible to titrate 0.10–10, 0  $\mu\text{g}/\text{ml}$  of the studied ions Sb(III) and Bi(III) quantitatively reacting with  $\text{C}_5\text{H}_{10}\text{NS}_2\text{Na}\cdot 3\text{H}_2\text{O}$  in non-aqueous media. To optimize the conditions for determining Sb(III) and Bi(III) ions with various reagents, it is necessary to determine their dielectric constant, viscosity, and electrical conductivity of titrated non-aqueous solutions.

Literary (Kreshkov A. P., 2009; Kreshkov A. P., Yarovenko A. N., Bartikova O. D., 2003) and our data confirmed that among alkali metal salts, lithium salts (due to the small radius of the cation) are the most strongly solvated compounds compared to sodium and potassium salts.

It could be expected that the addition of an inert solvent (chloroform, benzene, carbon tetrachloride, hexane, methyl ethyl ketone, etc.) with an even lower dielectric constant than that of the protolytic solvent: acetic acid, n-propanol, DMFA, would increase the degree of titration. On the other hand, a significant increase in the ohmic resistance of the solution under study causes a significant drop in its potential in it, which will inevitably entail a rapid violation of the proportionality between the magnitude of the current and the concentration of the complexant in the analyzed solution. Thus, there must be some optimal and maximum permissible concentrations of the inert solvent in acetic acid, as well as in n-propanol, DMFA, at which correct and reproducible titration results should be ensured.

Experimental data was obtained in determining the specific electrical conductivity

and viscosity of titrated solutions, individual solvents, and their mixtures containing in-different salts of different nature: potassium acetate, sodium acetate, lithium, and sodium nitrate, and perchlorate, which were used as background electrolytes when determining micro concentrations of the studied ions, confirmed our assumptions.

A study of the effect of chloroform on the viscosity of acetic acid solutions containing

potassium acetate, lithium nitrate, and perchlorate showed that the greatest decrease in viscosity is observed in solutions containing potassium acetate, and the smallest in lithium nitrate.

Experiments have shown that chloroform greatly reduces the viscosity of propanol, acetic acid, and dimethyl sulfoxide solutions (background – potassium acetate) and increases the viscosity of dimethylformamide solutions.

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