

<https://doi.org/10.29013/AJT-23-3.4-27-29>

*Umarov Fakhridin Abdulazizovich,
Independent researcher of the Jizzakh State Pedagogical
University. Uzbekistan,
Yakhshieva Zuhra Ziyatovna,
Doctor of Chemical Sciences, Professor of Jizzakh State
Pedagogical University. Uzbekistan*

TITRATION OF THE ZINC ION WITH A SOLUTION OF 2-PHENYL-HYDRAZIDE PHENYLAZOTHIOMIC ACID

Abstract. Sensitive and selective reactions of sulfur-containing reagents to metal ions in non-aqueous (acetic acid and n-propanol) media were the basis for establishing the possibility and developing voltammetric procedures for titration of zinc ion with a solution of phenylazothioformic acid 2-phenylhydrazide in dimethylformamide and dimethylsulfoxide. The article presents the results of experiments on the voltammetric determination of the zinc ion by phenylazothioformic acid 2-phenylhydrazide in medicinal zinc-containing preparations.

Key words: diphenylthiocarbazone, zinc, tying, electrodes, sulfur-containing reagents.

The literature presents the possibility of using amperometric indication of the end point of titration of ions of some heavy metals with solutions of phenylazothioformic acid 2-phenylhydrazide (according to their emerging anodic current using two platinum indicator electrodes in an amphiprotic solvent – n-propanol. It was of interest to check the possibility of similar titrations with solutions of the same titrants in protophilic media, the acid-base properties of which are very different from amphoteric ones – dimethylformamide and dimethylsulfoxide [HCON(CH₃)] and (CH₃)₂SO.

We found that the reagent phenylazothioformic acid 2-phenylhydrazide, oxidized on a platinum disc microanode in protophilic media on the backgrounds of potassium acetate, lithium nitrate and perchlorate, gave quite clearly pronounced anodic waves with half-wave potentials equal to 0.49 V, 0.58 V and 0.70 V respectively.

It was shown that in dimethylformamide and dimethylsulfoxide on backgrounds of different acid-base properties, of all the studied cations, zinc (II) is best titrated with solutions of the above reagents,

while the position of i.e., as a rule, is always proportional to its taken amounts, which predetermines the possibility of obtaining correct and reproducible results in the voltammetric determination of zinc(II) based on the stoichiometric reaction of complex formation in the ratio Me: Reagent= 1:2 (Zn - differential current protection).

Influence of external voltage. From the current-voltage curves of differential current protection oxidation, it can be concluded that amperometric titration of Zn(II) is possible based on the cathodic reduction current of both the titrated ion and the emerging anodic current of the reagent. Zn (II), as has already been shown, form very strong dithizonates that do not decompose even in fairly acidic and basic media. Therefore, it could be expected that the formation of zinc dithizonate complexes in dimethylformamide and dimethylsulfoxide would also proceed quantitatively, especially in the presence of potassium acetate and lithium perchlorate. Zinc dithizonate is a zinc metal chelate compound with the formula $Zn(C_{13}H_{11}N_4S)_2$. Under normal conditions, it is purple-red crystals.

Comparing the voltammograms of zinc cations, titrating reagents, their complexes (dithizonates) and oxygen, obtained in dimethylformamide and dimethylsulfoxide, we can conclude that already at a voltage of about 0.4–0.5 V, the current at the first stage of acid-base titration should arise due to the reduction of titratable zinc cations at the cathode and the oxidation of their respective metal complexes (zinc dithizonate) at the anode. After acid-base titration, the current should again arise, but this time due to the reduction of oxygen and the oxidation of the reagent at the anode. The experiments carried out confirmed our assumptions; when titrating zinc ions with differential current protection solutions, curves of sufficiently clear shape, correct and reproducible acid-base titration results were obtained in the range of voltages applied to the indicator electrodes that did not exceed the optimal values (0.4–0.8 V). Experiments have shown that the formation of complexes of Zn (II) with differential current protection occurs quite quickly and the measured indicator current becomes constant at any stage of the titration after 5–10 s. after each addition of titrant. Very clear voltammetric determination curves, which make it quite easy and accurate to find acid-base titration, are observed on the backgrounds of potassium acetate and lithium perchlorate at an electrode voltage of 0.4–0.8 V.

Influence of the nature and concentration of the supporting electrolyte. During the voltammetric determination of Zn (II) with a differential current protection solution in dimethylformamide and dimethylsulfoxide, it was found that the concentration of supporting electrolytes (potassium and sodium acetates, lithium chloride, nitrate and perchlorate) has a significant effect on the clarity of titration curves, and at their low content (less than 0.025 M) even on the correctness of the results.

The experiments showed that potassium acetate and lithium perchlorate turned out to be the best of all the studied backgrounds. On other backgrounds, it is difficult to accurately find the end point of the titration of the zinc ion.

It was found that the optimal concentrations of background electrolytes in the voltammetric determination of a zinc ion with a differential current protection solution are: for potassium acetate – 0.10–0.30 M, lithium nitrate – 0.05–0.20 M, and lithium perchlorate – 0.05–0.25M.

Evaluation of the correctness and reproducibility of the developed methods. To assess the degree of accuracy of the proposed method for the voltammetric determination of zinc(II) with a solution of differential current protection in dimethylformamide and dimethylsulfoxide, as well as their mixtures with various inert solvents, voltammetric determinations of various amounts of zinc ion were carried out with 3–5-fold repetition of titration of each amount. The results of the voltammetric determination of 5–1000 mg of zinc ion in 10 ml of the test solution indicate the correctness and reproducibility of the data obtained (relative standard deviation is not more than 0.045), and the average value found, compared with the introduction, does not go beyond the confidence interval.

The obtained experimental data show that the developed procedures for the voltammetric determination of zinc(II) by a solution of diphenylthiocarbazone in dimethylformamide and dimethylsulfoxide, as well as in their mixtures with inert solvents, are quite selective.

Summing up the results of the studies on the positive zinc ion with differential current protection solution in protolytic media (acetic acid, n-propanol, dimethylformamide and dimethylsulfoxide) and their mixtures with some inert solvents, we can conclude that the most favorable medium for such titrations is acetic acid and its mixtures with chloroform and carbon tetrachloride, then n-propanol and its mixtures with benzene and chloroform, and finally dimethylformamide and dimethylsulfoxide and their mixtures with chloroform and hexane.

Of all the above solvents, anhydrous acetic acid and its mixtures with inert solvents are the most versatile in terms of titration of 2–4 cations in one aliquot

in the presence of a large number of interfering metal ions, because in such media high selectivity can be achieved quite well and quickly by increasing the acidity of the titratable solutions by adding nitric, perchloric or other strong acids. Under such conditions, zinc (II) can be determined very accurately in the presence of large amounts of heavy metal ions without the use of special masking agents or other compounds.

With regard to the rapidity of performing the voltammetric determination of metal ions in one analyzed sample, other things being equal, of all the studied media, dimethylformamide and its mixtures with inert solvents are better, since the time for titration of one aliquot of the analyzed sample does not exceed 2–3 min.

The determination of zinc (II) in artificial mixtures was carried out both by the current of zinc reduction on a platinum indicator electrode by the current of differential current protection oxidation.

The results of the analyzes performed show that the developed methods for determining the zinc (II) ion with a differential current protection solution are quite correct and selective with a relative standard deviation not exceeding 0.05. the time of the analysis of one mixture is no more than 10–15 minutes. The developed procedures for the voltammetric determination of the zinc ion are characterized by fairly good selectivity in protophilic media with relative accuracy, rapidity, and a low detection limit.

References:

1. Yakhshieva Z. Amperometric determination of some metals sulfur-containing organic reagents in non-aqueous, and mixed aqueous media // Austrian Journal of Technical and Natural Science. Austria.– No. 5–6. 2015.– P. 151–154.
2. Yakhshieva Z., Bakaxanov A., Kalonov R. The Influence of Toxic and Ecologically Harmful Components on the Environment // EPRA International Journal of Multidisciplinary Research (IJMR) – Peer Reviewed Journal V: 6 | Issue: 10 | October 2020. ISSN (Online): 2455–3662. Indiya / 2020.– P. 92–95.
3. Akhmadjonova Y. T., Yakhshieva Z. Z. Effects of heavy toxic metals on water quality // Science and Education – No. 72020.– P. 8–11.
4. Yakhshieva Z. Z., Akhmadjonova Y. T. Pollution of water bodies with inorganic toxicants // Science and Education.– Vol. 2.– No. 9. 2021.– P. 106–121.