Received: 15-02-2014
Accepted: 12-03-2014

ISSN: 2321-4902
Volume 1 Issue 6

Online Available at www.chemijournal.com

International Journal of Chemical Studies

O-Anisidine as Indicator in Titrimetric Determination of Iron (II) in Pharmaceutical Formulations and Water Samples

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Inspite of the beautiful red coloured oxidized product of O-anisidine, the studies on its application in analytical techniques are scanty. So, authors have taken up the investigation on the utility of O-anisidine as a new Analytical reagent in the bromatometric-Indicator reaction. The detailed reaction on the potassium bromate and O-Anisidine has enabled the authors to utilize O-Anisidine in titration of Iron (II). Suitable conditions have been established with different acids viz., hydrochloric acid, sulfuric acid, phosphoric acid, acetic acid to give sharp colour change at the equivalence point. This method could be used successfully for determination of the Iron (II) content of Spiked water and Pharmaceutical samples. The results obtained are in good agreement with the values obtained by standard methods.

Keyword: O-Anisidine, Bromatometric, Iron (II), Pharmacy and Spiked Water samples.

1. Introduction

O-Anisidine is used as a reagent for the spectrophotometric determination of gold [Jenic Joseph. *Et al.* 1876] in biological samples and also in some salts such as silver nitrate in Acid media. Details of different substances, O-dianisidine, Xanthane, Indigold and dyes belonging to the classes of azo triphenyl, methane thiazine, oxazine and other substance proposed as Indicators in bromotometric titrations are reported in Table - 1 [1-28]. Smith and Bliss [29] were the first to report the bromatometric titration of Iron (II) using basic Mercuric bromate as the titrant and fachsin, chrysoidine R, bordeaux and naphthol blue black as indicators. Szebelledy and Madis [30] proposed potassium bromate as reagent for the titrimetric determination of Iron(II) at 60-70 °C using gold chloride as an indicator. Kolthoff [31] found that naphthol blue black can be employed as indicator in the Iron (II) - potassium bromate titration and that the titration can be conducted satisfactorily without any interference by tartaric, citric and succinic acids. Ottaway and Bishop [32] studied the Iron(Il) - bromate titration using potentiometric and visual indicator methods. They critically studied the effects of oxygen and the presumptive catalysts and found that the role of copper (II) reported by Smith and Bliss [29] was not to catalyse the Iron(ll) -Bromate reaction but only to bring about the rapid attainment of the equilibrium potentials of the indicator electrode. Gopal Rao & Krishna Murthy [33] recommended potentiometric titration of iron (II) with potassium bromate in 9.0-10.0M phosphoric acid medium and later Krishna' Murthy *et al.* [20, 21] proposed cacotheline as an indicator in the same titration in the phosphoric acid medium. Satyanarayana [6] has observed that methylene blue, thionine, azures A, B and C, toluidine blue, new methylene blue, p-ethoxychrysoidine and brilliant ponceau 5R can be used as indicators in the Iron(II) - bromate titration in a medium of 9-10M phosphoric acid and prescribed the addition of the indicators, except p-ethoxychrysoidine, towards the close of the titration. Rao and Ramana [13] have proposed three azine dyes neutral violet, neutral red and wool fast blue GL as indicators in the titration of iron (II) with bromate in the phosphoric acid medium. They have observed that wool fast blue GL can be satisfactorily employed as an indicator in the reverse titration also i.e., titration of bromate with Iron (Il), in phosphoric acid medium. Several kinetic methods were reported for the determination of Iron (II) [34-40]. In this paper, the authors would like to develop a selective, accurate, and simple titrimetric method for the determination of iron (II) based on the catalytic oxidation of crystal violet with bromate in real matrixes such as pharmaceutical products and water Samples.

Even though O-dianisidine is used as indicator in bromatometric titrations, the survey of the literature revealed, no attempts have been made in the application of O-anisidine as indicator in the titrations with bromated. So, the present investigation has taken up the study of o-anisidine as an indicator in titrations of Iron (II) with potassium bromate and the results obtained are incorporated.

2. Experimental

2.1 Titration of Iron (II)

The present author has investigated the use of O-anisidine as indicator in the titration of Iron (II) with potassium bromate in hydrochloric, sulphuric, phosphoric and acetic acid media and established suitable conditions for satisfactory titrations.

Reagents

2.2 Potassium bromate solution

Potassium bromate solution of 0.1N concentration is prepared by dissolving 2.784 g of the recrystallised and dried (at 180 °C for 2 hours) potassium bromate (E. Merck) in water and diluting the solution to one liter with triply distilled water.

All other reagents, hydrochloric acid, sulphuric acid, acetic acid and phosphoric acid used are of analytical reagent grade quality.

2.3 Method: In order to ascertain the effect of different acids studied viz., hydrochloric, sulphuric, phosphoric and acetic acid in the reaction between the indicator, O-anisidine and potassium bromate, the author has carried out the following experiments. 0.1 ml of 1% solution of o-anisidine is treated with sufficient quantity of hydrochloric, sulphuric, phosphoric and acetic acid and water to make upto 50 ml. To this solution, one drop of 0.1M potassium bromate solution is added and the observations are recorded in Table-2.

In order to establish the optimum conditions for the visual determination of iron (II), the author has earned out the following experiments in different acid media.

5.0 ml of 0.1036N iron (II) is taken in the titration vessel, required amount of acetic, hydrochloric, sulphuric or phosphoric acid are added to give desired concentration and 0.1 ml of 1% o-anisidine indicator and distilled water added to a total volume of 50 ml. The titration is carried out in an inert atmosphere of carbon dioxide with 0.1072N bromate to a colour transition from light yellow to red colour. The results are tabulated in Table-3.

From the experimental observations, it is found that the indicator is not functioning well at any hydrochloric and acetic acid concentrations. Turbid solutions are obtained with hydrochloric acid. In case of sulphuric acid medium between 2.0-8.0N the colour change is observed from colourless to red and waiting for about 15 seconds was necessary near the endpoint. The colour change is sharp in case of 6.0-12.0N phosphoric acid medium i.e., from light yellow to red colour in the titration of Iron (II) with bromate. So, the author has chosen 3N sulphuric acid and 10N phosphoric acid to carry out the titrations to get satisfactory end points.

2.4 Effect of indicator concentration

The effect of concentrations of o-anisidine is also studied using different volumes at 3N sulphuric acid and 10.ON phosphoric acid concentration in the titrations of Iron (II) with bromate (Table 4). The colour change of the indicator is sharp in 0.1-0.5 ml of 1% o-anisidine. So the author prescribes

0.2 ml of the indicator to be added in titrations of iron (II) with bromate.

2.5 Recommended Procedure:

An aliquot of 0.1036N Iron(II) is taken in the titration vessel, an overall acidity of 3N sulphuric or ION phosphoric acid is maintained in a total volume of 50 ml, 0.2 ml of indicator is added and the titration is carried out with 0.1072N bromate to a colour change from light yellow to red colour. Some typical results of the estimation of Iron (II) are given in Table-5.

2.6 Reverse Titrations

Reverse titrations i.e., the estimation of bromate with iron (II) is earned out by the author adopting the same procedure as above in different acid concentrations.

It is found that the indicator is not functioning well at any of the hydrochloric, sulphuric and acetic acid concentrations. But in 6.0-12.0N phosphoric acid concentrations the indicator functioned well and the colour change of the indicator is from red to light yellow colour. Hence an optimum concentrations of 10.0N phosphoric acid for the estimation of bromate with Iron (Il) is recommended. Some typical results obtained are given in Table-6 and 7.

3. Application of the Developed Method

To confirm the usefulness of the proposed method, Fe (II) was determined in pharmaceutical samples and spiked water samples.

3.1 In Pharmaceutical Formulations

The Indicator method developed can be applied successfully for determination of Iron content in pharmaceutical tablets as per recommended procedure, One tablet is ground to a free powder and dissolved in double distilled water and the solution filtered through G-4 sintered glass funnel and diluted to a known volume. An aliquot of this solution is titrated with 0.10N bromate solution in 2.0 N sulfuric acid medium using O-anisidine as an indicator. A similar aliquot is titrated with potassium bromate using Thiocyanate as an indicator. The samples of pharmaceutical tablets and typical results obtained while employing both the methods are present in the Table -8. It is observed that these results are in good agreement with standard methods.

4. In Water Samples

4.1 Treatment of samples

River water and wastewater samples were collected at different points from the polluted regions of Coastal Andhra. Prior to analysis, river water samples were treated as follows. 50 ml of the sample was evaporated to about 5 ml in a 1000 ml beaker on a hot plate at about 90 °C, the 1 ml of concentrated nitric acid and 0.5 ml of H_2O_2 (30%) was added and the contents were heated to dryness at 90 °C. After cooling, the digest was diluted to 25 ml with water and adjusted to $P^H = 5$ with saturated ammonium carbonate. It was boiled for 5 min to expel the dissolved CO_2. After cooling, the solution was transferred to a 50 ml volumetric flask and diluted to the mark with water, which was ready for analysis. Bottled mineral water samples were obtained from a local convenience store and analyzed directly without pretreatment. Results are tabulated in Table. 9.

5. Conclusion

A new reaction system was suggested for the Titrimetric Determination of iron(II) in Pharmaceutical and Spiked water samples. This method offers several advantages, as follows: high selectivity and sensitivity ease of operation and rapidity, and cheaper reagents. The reliability and simplicity of this method permit the analysis of pharmaceutical samples with satisfactory results. Results obtained are in good agreement with the values obtained by standard methods.

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