

ESTIMATION OF NITRITE IN PRESENCE OF NITRATE

M.H. HASHMI, S.P. HAMDANI and M. SARWAR

PCSIR Laboratories, Lahore

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Abstract. In this investigation sulphamic acid has been used as a titrant for the determination of nitrite in presence of nitrate. The end point is located by the pH measurement. Sulphite, phosphate, chloride and sulphate do not interfere in the procedure. The method is quite precise and accurate and maximum error was 1.3% when a 31.0-mg sample was analyzed.

Many titrimetric methods for the estimation of nitrite in presence of nitrate are known in literature.¹⁻⁶ In the method of Flitman and Miriam¹ they used standard solution of 4,4'-sulphonyldianiline and diphenylamine was used as internal indicator. The method is tedious and titration is to be performed at exactly 45°C. Whiteman² estimated nitrite in presence of nitrate by employing excess of Ce(IV) which is then back-titrated with Fe(II) standard solution. In this method back-titration is performed and the method is lengthy and time consuming. Excess of chloramine-T has also been applied for the oxidation of nitrite in presence of nitrate and then monitoring the residual amount iodometrically.³ The method again is time consuming. Sarjit and Dula⁴ used KMnO₄ for the estimation of nitrite. Back-titration was required using quite stringent conditions and interference due to nitrate was not checked. Winograd⁵ determined nitrite in presence of nitrate iodometrically. But bubbling of CO₂ through the test solution was required. Hydrolytic titration using sulphamic acid was also tried⁶ but the Griess reagent was used as external indicator in such titration, the results may not be accurate.

In the present investigation we have used sulphamic acid for the estimation of nitrite in presence of nitrate. Though the method is volumetric yet end point is located by pH measurements. The method is convenient to perform and it gives quite sharp end point. No indicator is required in this method. Nitrate, sulphate, sulphite and phosphate do not interfere in the procedure.

Experimental

Sulphamic Acid. 0.1M standard solution was prepared by dissolving 2.4275 g in distilled water and diluting to 250 ml.

Potassium Nitrite 2.1275 g pure crystalline substance was dissolved in distilled water and diluted to 250 ml. It was standardized by the method of Singh and Dula.⁴ It was found to be 97.5%. All other chemicals used were of analytical grade.

Procedure. An accurately measured volume (20 ml) of the test solution was placed in a 50-ml beaker in which the electrodes of a pH meter were dipped. This solution was then titrated with standard solution of sulphamic acid added in small instalments from a microburette, graduated at 0.02 ml intervals. The contents were constantly stirred during titration.

The pH was noted after every addition and the end-point was reached when the pH became constant.

Results and Discussion

It is indicated from Table 1 that nitrite can be successfully estimated in presence of nitrate using sulphamic acid as titrant. The end-point was detected with pH measurement. A representative titration is shown in Fig. 1. As a result of the stoichiometric reaction between sulphamic acid and nitrite KHSO₄ is produced according to the following equation:

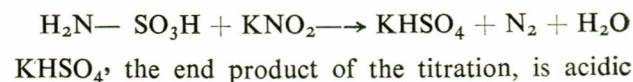
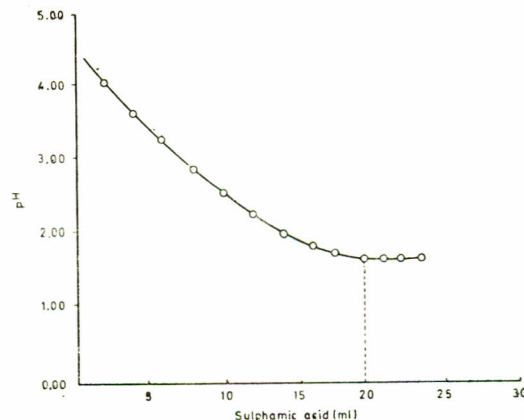


TABLE 1. ESTIMATION OF NITRITE.

Nitrite taken (mg)	Nitrite found (mg)	Deviation (%)
164.0	163.3	0.43
132.2	130.7	1.15
97.6	96.9	0.71
82.2	81.4	0.98
63.0	62.5	0.80
49.1	49.0	0.2
31.0	30.6	1.3

Fig. 1. Titration of 0.1M KNO₂ with 0.1M Sulphamic acid.

in nature. Large amount of this salt and slight excess of sulphamic acid will be present at the equivalence point which will make a buffer of constant pH at a low pH range. The endpoint is taken when the pH becomes constant. Results are quite accurate and the maximum error is 1.3% when 31.0 mg sample was analyzed.

Effect of Diverse Ions. Besides nitrate which is usually present in nitrite, the interference due to phosphate, sulphate chloride and sulphite was examined, and it was found that none of these ions interfere in the procedure.

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