Spectrophotometric Determination of Nitrite in Nitrate and Sulfate

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The purpose of this paper is to develop a rapid and sensitive method for the spectrophotometric determination of trace amounts of nitrite in the presence of large amounts of nitrate and sulfate. The method is based on the colored compound formed by the reaction between disodium 1-(4′-aminobenzeneazo)-2-amino-8-hydroxynaphthalene-2′, 6-disulfonate and nitrous acid. Details of this compound have been described in our previous paper[1].

For the determination of trace amounts of nitrite useing this compound, seventeen kinds of ions were found to cause the interference (As^{3+}, Fe^{3+}, $Ce^{3+,4+}$ *, $Hg^{+,2+}$, Mn^{2+}, $Sn^{2+,4+}$, Ti^{4+}, Zr^{4+}, Br^-, F^-, I^-, MnO_4^-, MoO_4^{2-}, SeO_3^{2-}**). The study of the effect of other ions in the previous paper suggested the possibility that they could be used for the determination of nitrite in nitrate and sulfate without any special procedure.

Experimental

The apparatus and reagents were the same as those used in our previous work.

Procedure.—Transfer to a 25 ml. volumetric flask 1 ml. of the sample solution adjusted so as to contain 5 to 60 p.p.m. of nitrous acid. Add 20 ml. of distilled water, and 2.5 ml. of 0.05% reagent solution. Add 0.3 ml. of 6 N hydrochloric acid and shake well. Bring up to the mark with distilled water, thoroughly mix and, after ten minutes, measure the absorbance at 625 mμ, using a reagent blank solution*** as a reference.

Results

The determination of nitrite in the presence of nitrate and sulfate was made. The results are given in Table I.

Summary

A simple procedure has been developed for the determination of trace amounts of nitrite in large amounts of nitrate and sulfate, with disodium 1-(4′-aminobenzeneazo)-2-amino-8-hydroxy-naphthalene-2′, 6-disulfonate. The method is accurate and precise.

TABLE I. DETERMINATION OF NITROUS ACID

Taken HNO_2 p. p. m.	Added NO_3^- mg./25 ml.	Added SO_4^{2-} mg./25 ml.	Absorbance* at 625 mμ	Standard deviation in absorbance unit
0.20			0.080	0.002
	0.5		0.082	0.003
	50.0		0.076	0.003
	5000.0		0.082	0.003
		0.5	0.079	0.002
		50.0	0.079	0.003
		5000.0	0.082	0.004
	500.0	500.0	0.079	0.004
0.75			0.307	0.002
	500.0	500.0	0.304	0.004

KNO_3 and K_2SO_4 were used to make up the solution.

* These values were the averages of six replicate determinations.

1) Y. Katsube and J. H. Yoe, This Bulletin, 33, 190 (1960).

* In the previous paper, the reagent was tested with 67 kinds of ions not including Ce^{4+}.

** In the previous paper, SeO_3^{2-} was mistyped as CeO_3^{2-}.

*** Use the sample solution as the reference when the sample contains Cu^{2+} or Ni^{2+}.

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