

## VOLUMETRIC DETERMINATION OF SULPHITE WITH N-BROMOSUCCINIMIDE USING BORDEAUX RED AS INDICATOR

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A new method for the determination of sodium sulphite has been described where N-Bromosuccinimide has been used as a direct titrant. Bordeaux red has been used as an indicator whose rose red colour changes to yellow at the end point. The interference due to thiosulphate, sulphide, sulphate and  $\text{Fe}^{+2}$  have also been studied and removed. The method is convenient, rapid and accurate.

### INTRODUCTION

Sulphites are potential reducing agents. They get immediately oxidized by air when in solution, however they can be stabilized by the addition of tetrachloromercurate solution [1]. The methods for their determination, therefore, utilize this reducing property. The most important method for their determination is by iodometry where the sample is added to an excess of standard iodine solution and then this excess iodine is back titrated with sodium thiosulphate [2,3], ascorbic acid or hydrazine [4,5]. The last two reagents are not very stable, therefore, they have not much advantage over sodium thiosulphate solution. Sulphites have also been determined by direct titration with iodine using thyodine [6] as indicator. In another method luminol has been used as indicator in the direct titration of sulphite with iodine in alkaline medium [7]. Sulphite, thiosulphate and sulphate can be determined in the presence of each other by using bromine and then measuring the total sulphur as bromium sulphate [8]. Iodine monochloride has also been used in the determination of sulphate and thiosulphate where ICI reacts with these compounds to give sulphate and iodine. The excess of ICI can be back titrated with standard arsenite solution or the iodine formed in the reaction can be titrated with standard periodate in presence of hydrochloric acid and potassium bromide with Andrew's titration end point [9,10]. Chloramine-T has also been used in the volumetric determination of sulphite where the excess of the reagent is back titrated iodometrically [11]. The same reagent has also been employed for the determination of sulphite, thiosulphate in the presence of each other [12]. Another volumetric method is based on the oxidation of sulphite to sulphate with potassium iodate [13,14]. Manchot and Oberhauser used bromate-bromide mixture

to convert sulphite into sulphate and the excess was back titrated with standard arsenite solution using indigo-carmin as indicator [15]. Hypobromite has also been used for the oxidation of sulphite and the excess of oxidizing agent is back titrated iodometrically [16,17]. Hypochlorite has also been recommended as titrant and the excess of titrant is back titrated iodometrically [18].

In most of the methods cited in literature either back titrations are involved or the reagents used are unstable. Further there are very few methods available for the determination of sulphite in presence of thiosulphate and sulphide.

In the present investigation we have used N-bromosuccinimide as direct titrant for determination of sulphite using Bordeaux red as indicator. This reagent is fairly stable. Its equivalent weight is about 2.5 times greater than that of potassium iodate which has been frequently used for the determination of thiosulphate. This greater equivalent weight warrants its use in this titration because the chances of error are much reduced. The method described is convenient, rapid and easy to perform.

### EXPERIMENTAL

**Equipment.** Microburette of 5-ml capacity graduated at 0.02 ml intervals. Graduated pipets of 0.5, 1 and 2 ml capacity. Measuring flasks of 100 ml capacity. A nitrogen cylinder.

**Reagents** Sodium sulphite: (BDH) This was standardized by the method of Kolthoff [3].

**N-Bromosuccinimide:** A 0.1N solution of N-Bromosuccinimide was prepared by dissolving 890 mg of recrystal-

lized compound in distilled water and making the volume to 100 ml, in which nitrogen was passed for about 5 min. In all these titrations nitrogen passed distilled water was used.

**Bordeaux red:** A 0.05% solution was prepared in nitrogen passed distilled water.

All other reagents used were of analytical grade and where ever the solution was used it was prepared in nitrogen passed distilled water.

#### Procedure

For the direct volumetric determination of sodium sulphite definite amount (from 10.0 – 20.1 mg) of solid compound was taken in an Erlenmyer flask which was dried and flushed with nitrogen gas. Just before titration 10 ml of nitrogen passed water was added to dissolve the solid sodium sulphite. To the resulting solution 2 drops of bordeaux red indicator was added and then titrated with 0.1N N-Bromosuccinimide. The end point was reached when the rose red colour of bordeaux red was changed to yellow. The blank for bordeaux red was taken and this was subtracted from the titer before calculation.

#### Calculations

mg of sodium sulphite = N.V.E.

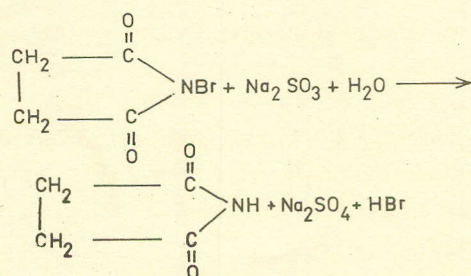
N = Normality of N-Bromosuccinimide

V = Volume of N-Bromosuccinimide used in the titer

E = Equivalent weight of sodium sulphite.

#### RESULTS AND DISCUSSION

N-Bromosuccinimide is a very potential oxidizing agent [19-23]. In the present investigation it has been used for the determination of sulphite. It oxidizes sulphite according to the following reaction:



The stoichiometry of the reaction is proved by the results shown in Table 1. The maximum error was 1% when 10.0 mg sodium sulphite sample was determined.

#### Effect of Thiosulphate on the Determination of Sulphite.

Sulphite was also determined in the presence of sodium thiosulphate. It has been found that when up to 0.2 mg sodium thiosulphate was present in 10 mg sample of sulphite it did not have any effect on the determination. The results are shown in Table 2.

Amounts of sodium thiosulphate greater than 0.2 mg had significant effect on the determination of sulphite. But this interference was removed by the addition of lead nitrate solution to the test sample.

#### Effect of Sulphide on the Determination of Sulphite:

The tolerance limit for sulphide in the determination of sulphite is 0.1 mg in 10 mg sample of sulphite. However, when greater amounts were present, it did interfere in the determination of sulphite. But this interference was removed again by the addition of lead nitrate. The results are shown in Table 3.

#### Effect of Sulphate on the Determination of Sulphite.

Sulphate does not interfere in this determination even if present equal in amounts to sulphite.

#### Effect of Fe<sup>+2</sup> on the Determination of Sulphite.

Iron (II) when present also interferes in the determination

Table 1. Determination of sodium sulphite.

Amount taken (mg)	Amount found (mg)	Error %
10.0	9.9	-1.0
12.2	12.1	-0.8
13.4	13.3	-0.7
13.8	13.7	-0.7
15.7	15.6	-0.6
16.1	16.0	-0.6
17.5	17.4	-0.6
18.0	17.9	-0.5
19.4	19.3	-0.5
20.1	20.0	-0.5

Table 2. Determination of sulphite in presence of thiosulphate.

Amount taken (mg)	Thiosulphate added (mg)	Amount found (mg)	Error %
10.0	0.1	9.9	-1
10.0	0.2	9.9	-1
10.0	0.3	10.2	+2
10.0	0.4	10.2	+2
10.0	0.5	10.4	+4

Table 3. Determination of sulphite in presence of sulphide.

Amount taken (mg)	Sulphide added (mg)	Amount found (mg)	Error %
10.0	0.1	9.9	-10
10.0	0.2	10.2	+20
10.0	0.3	10.3	+30
10.0	0.4	10.3	+30
10.0	0.5	10.4	+40

of sulphite. This interference was removed by the addition of sodium flouride to the test sample.

The method described is simple and less time consuming than the existing methods, since only one reagent (N-Bromosuccinimide) is required. The maximum error was 1% when 10.0 mg sample was analysed.

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