



5-12-1939

## The Determination, Analysis, or Assay of Sodium Nitrite

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THE DETERMINATION, ANALYSIS, OR ASSAY OF SODIUM NITRITE.

Submitted in partial fulfillment of the requirements  
for Honors in Chemistry.

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May 12, 1939

THE DETERMINATION, ANALYSIS, OR ASSAY OF SODIUM NITRITE.

In this experimental work on the determination of sodium nitrite, three procedures were put under observation. The first used was a volumetric analysis involving the use of p-nitroaniline, and was devised by W. H. Bell<sup>1</sup>. The second was a gravimetric determination set forth in Scott<sup>2</sup>, as the work of Buwold<sup>3-4</sup>. The third and most successful assay was another volumetric analysis given in Griffin<sup>5</sup>, and involved the use of potassium permanganate, potassium iodide and sodium thiosulfate, the indicator being a freshly prepared starch solution.

In the first analysis, using the method of Bell<sup>1</sup>, the procedure is as follows: "Dissolve 20 g. of sodium nitrite in 1 liter of distilled water. Weigh 3.4512 g. of p-nitroaniline, wash into 600 cc. beaker and add 10 cc. of concentrated HCl and 100 cc. of water. Heat to dissolve. Add ice and water until the temperature falls to 15°C and the volume reaches 350 cc.. Start the stirrer and add nitrite dropwise from a burette. If the solution is molar, 25 cc. of nitrite are required. Test the solution with KI and starch paper at 22 cc.. When an excess of free nitrous acid is shown by the darkening of the test paper, shut off the burette and test frequently to secure a permanent end point".

However, when the nitrite was titrated into the standard solution of p-nitroaniline, it was found that, instead of using 20-25 cc. of the nitrite, the end point was distinctly reached after approximately 3-8 cc. of the nitrite was used. Definitely, this was not correct, and, due to the inability to secure the correct journal for the original article, this procedure was not carried any further.

The second method, that of Buwold, was a little more successful. The following procedure was carried out: "1g. of silver bromate was dissolved

in 100 cc. of water and 110 cc. of 2 N acetic acid in a flask. 200 cc. of nitrite (1 g. sodium nitrite) is added from a burette, with constant stirring. A pale green precipitate is formed. 30 cc. of sulfuric acid (1:4) is added, and warmed to 85°C.. The whole was filtered on a Gooch crucible and washed with hot water, dried and weighed as AgBr and AgCl. The chlorine was then weighed on separate portions and AgCl subtracted." However, in our work, it was not necessary to run the chloride test, as the sample of nitrite was found to be free of this interference.

As the work progressed, it was found that the silver bromate that was to be used would not completely go into solution with the given amount of water and acetic acid, nor even with reasonable amounts in excess of these two solvents. Therefore, when the silver bromide was precipitated for weighing, it was found that some bromate still remained unchanged, and entered the Gooch along with the precipitated bromide and was weighed along with it. With the exception of this obvious error, this procedure was fairly accurate. We are reasonably sure that the bromate is the cause of the error found because, in different trials, varying amounts of nitrite and varying amounts of bromate were used in conjunction with each other. It was found, as shown by the following results, that the amount of error in each calculation is directly proportional to the excess of bromate that remained in solution, showing that the error might well be caused by the aforementioned excess.

Sample	ppt.	bromate used	error
0.9971 g.	1.05404 g.	1.352 g.	0.05694 g. 5.7%
1.0360 g.	1.0928 g.	1.350 g.	0.0568 g. 5.4%
0.9991 g.	1.03103 g.	1.000 g.	0.03193 g. 3.19%
1.0002 g.	1.01328 g.	0.800 g.	0.01308 g. 1.3%
1.0015 g.	1.1386 g.	1.25 g.	0.1371 g. 13.6%

sample	ppt.	bromate used	error
1.0016 g.	1.2330 g.	2.000g.	0.2320 g. 23.1%
1.0007 g.	1.2329 g.	2.000g.	0.2322 g. 23.2%

The third method, that set forth in Griffin, was by far the most successful of those tried. The procedure is as follows: "Weigh on <sup>a</sup>blanced glass 4 g. of sample and dissolve in water. Filter and dilute to 4 L.

Pipette 50 cc. of 0.1 N potassium permanganate, and dilute with 150 cc. of water. Add 25 cc. of sample solution. Heat to boiling. Add 20 cc. of dilute sulfuric acid, and let stand for ten minutes. Cool under tap. Add 30 cc. of 10% KI solution. The solution should now be clear brownish red. Titrate with 0.1 N thiosulfate, adding 5 cc. of starch solution when the color begins to get pale. Titrate to the end point, as denoted by faint blue color of the starch solution.

Run blank with 50 cc. of permanganate, omitting addition of sample, and subtract from titration of sample. Calculate difference to sodium nitrite. (1 cc. 0.1 N sodium thiosulfate --- 0.003451 g. sodium nitrite.) "

The permanganate solution was first standardize against a known solution of sodium oxalate, and the sodium thiosulfate solution was next standardized against a solution of potassium iodide, which in turn was determined by titration with a standard arsenious oxide solution. The reaction proceeded without any difficulty whatsoever. The end point with the starch solution was very distinct in both titrations, with and without the sample. As the results will show, the whole determination was quite accurate, and it is fairly safe to assume that part of the error might be absorbed in the fact that, while the nitrite sample was taken as 100% pure, a degree of impurity might be present. The sample was made up by recrystallizing the stock supply of sodium nitrite several times with hot water, until a high degree of purity was obtained. However, the 100% purity factor was

not verified.

The results areas follows:

3.9992 g. nitrite/liter water----- 0.0999800 g./25 cc. used in analysis.

Sample	gms. determined	error	
0.09998g.	0.09867 g.	0.00131 g.	
"	0.09859 g.	0.00139g.	
"	0.09858 g.	0.00140 g.	
"	0.09847 g.	0.00152 g.	
"	0.09871 g.	0.00121 g.	
"	0.09868 g.	0.00130 g.	
"	0.09881 g.	0.00117 g.	
"	0.09880 g.	0.00118 g.	
"	<u>0.09862 g.</u>	<u>0.00136 g.</u>	
ave. 0.09998 g.	0.09866 g.	0.00131 g.	ave.

ave. deviation of samples from mean-----0.00008 g.

ave. percentage of error-----1.31%

<sup>1</sup> Chem. Met. Eng. 22, 1173(1920).

<sup>2</sup> Wilfred W. Scott, Standard Methods of Chemical Analysis, (N. Y., 1925) 1, 28.

<sup>3</sup> Chem. Ztg. 38,28.

<sup>4</sup> C. A. 8-1250(1914)

<sup>5</sup> Roger C. Griffin, Technical Methods of Analysis, (N. Y., 1921) 358.