A GASOMETRIC ASSAY OF SODIUM NITRITE.

BY EDMOND E. MOORE.

Sodium nitrite assays are nearly all based upon the ability of this compound to reduce $KMnO_4$. The U. S. P. X assay (3) calls for an excess of $KMnO_4$ and the subsequent titration of the latter with oxalic acid. Davy (1) calls attention to the objections to this method and suggests certain modifications.

Reduction methods cannot be safely used for the assay of sodium nitrite tablets as these tablets often contain varying amounts of other reducing substances such as milk sugar, starch, etc.

It is well known that a saturated solution of an ammonium salt will react quantitatively with sodium nitrite liberating nitrogen.

$$(I)$$
$$(NH_4)_2SO_4 + 2NaNO_2 \rightarrow Na_2SO_4 + 4H_2O + 2N_2$$
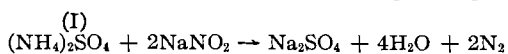
This reaction goes to completion in a relatively short time at 100° C. Gailhat (2) proposed a method in 1900 which was based upon this reaction, but due probably to its complexity it never came into favor.

The method outlined here is simple, requires no special apparatus, and but a small amount of the analyst's time. The results secured by different analysts check very well—see Fig. 1.

It is advisable to run two determinations simultaneously. This requires two sets of apparatus but, because of the simplicity of the set-up, it cannot be considered a real dis-

Fig. 1.—Nitrometer. A, Water-bath; B, 12–15-cc. test-tube; C, rubber stopper; D, capillary tube; E, pneumatic trough, partially filled with water; F, 50-cc. burette, filled with water. The end of the capillary tube (D) should be flush with the end of the stopper (C) and there should be a file mark on the test-tube (B) to indicate how far the stopper should be inserted each time.

advantage. A common pneumatic trough can be used for both sets.

Ammonium sulphate is used rather than ammonium chloride which was suggested by Gailhat (2) because of the greater solubility of the former. At 25° C., 100 cc. of water will dissolve 76.7 Gm. of $(NH_4)_2SO_4$ but only 28.2 Gm. of NH_4Cl (4).

It is possible to simplify the calculations without introducing an appreciable error by determining the value of a blank for each apparatus and using this value in all subsequent determinations.

Blank.—10.0 cc. of the saturated ammonium sulphate solution are placed in (B), the apparatus is connected as shown in the diagram and (A) is heated until no more gas collects in (F). The capillary tube is removed from the pneumatic trough and then the volume of gas in (F) is measured. This volume serves as a blank in the assays. The volume of gas here is so small that it can be assumed to be constant because variations in temperature and pressure which occur during different assays will not change it sufficiently to cause a significant error.

Determination.—The sample should contain from 0.08 to 0.12 Gm. of sodium nitrite or a sufficient number of tablets to give that amount of the salt. It is often advisable to weigh 40 tablets, reduce them to a powder and then take an accurately
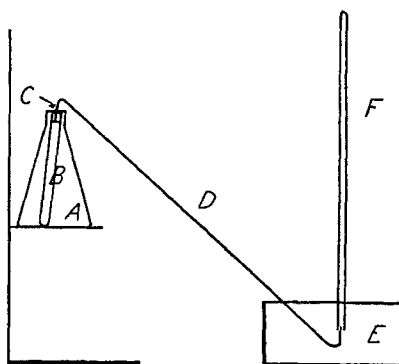
weighed 0.08 to 0.12-Gm. sample of the product for assay. The sample is placed in the test-tube (B), 10.0 cc. of saturated ammonium sulphate solution are added and the procedure is carried out as in the case of the blank. In order to be certain that the equilibrium is reached at least an hour should be allowed to elapse after the evolution of gas is complete before the burette is read. The temperature, barometric pressure and differences in water level inside and outside of the burette are recorded, and the volume of gas reduced to standard conditions.

$$V = (V_1 - B) \left(\frac{P - P_{H_2O} - \frac{(L.D)}{(13.6)}}{76} \right) \left(\frac{273}{273 + Tc} \right)$$

V = Volume of nitrogen corrected to $0°$ C. 76.0 cm. V_1 = Volume of gas in burette, uncorrected. P = Barometric pressure (cm. Hg). P_{H_2O} = Tension of water vapor at Tc (cm. Hg). $L. D.$ = Level of water inside burette − level of water outside (cm.). Tc = Temperature Centigrade.

From Equation I it will be evident that 69.01 Gm. of sodium nitrite with an excess of ammonium sulphate will liberate 22,413 cc. of nitrogen, the gas corrected to standard conditions. Therefore, 1.0 cc. of nitrogen gas at $0°$ C., 76.0-cm. pressure, represents 0.003079 Gm. of sodium nitrite.

$$\text{Per cent sodium nitrite} = \frac{(V) \ (0.003079)}{\text{Weight of sample}} \ (100)$$

The method has been used by different analysts using sodium nitrite samples of known composition, and tablets from different sources. The results have been very satisfactory.

LITERATURE CITED.

(1) Edward D. Davy, "Notes on the Assay of Sodium Nitrite," Jour. A. Ph. A., 18 (1929), 809.

(2) J. Gailhat, "Dosage gazometrique des nitrites en presence de nitrates ou autres sels solubles," J. pharm. chim., Par. 12 (1910), 9.

(3) Pharmacopœia of the United States X, page 344.

(4) A. Seidell, "Solubilities of Inorganic and Organic Substances (1916)," Van Nostrand Co., New York.

CORROSION OF METALS BY LIQUID IODINE AND BY MOIST IODINE VAPOR.

BY B. L. MEREDITH AND W. G. CHRISTIANSEN.

As part of a study of the corrosive properties of iodine, several samples of chemically resistant metals have been exposed to the action of molten iodine at $130°$ C., and of moist iodine vapor at $65-70°$ C., and quantitative measurements have been made.

In the first experiment six glass-stoppered bottles were filled with resublimed iodine and placed in an oven at $130°$ C. Each strip of metal was thoroughly cleaned, weighed, measured and immersed in the molten iodine, one strip to a bottle. The stoppered bottles were than allowed to remain in the oven 48 hours. At the end of this time the strips were removed and plunged into alcohol which dissolved off the adhering iodine. The metallic iodides were then washed off with