NOTES ON THE ASSAY OF SODIUM NITRITE.*

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The reports on the assay of nitrites by volumetric, colorimetric and gasometric methods indicate greatest interest in colorimetric methods applicable particularly to small quantities of nitrite. Yoe's "Photometric Chemical Analysis" gives references to one hundred and sixteen colorimetric methods, and new ones constantly make their appearance.

The United States Pharmacopæia seventh revision, in the assay of sodium nitrite, gave a gasometric assay; the succeeding revisions give volumetric assays, depending on the reduction of potassium permanganate in acid solution according to the equation:

$$5 \text{ HNO}_2 + 2 \text{ KMnO}_4 + 3 \text{ H}_2\text{SO}_4 \longrightarrow 5 \text{ HNO}_3 + \text{K}_2\text{SO}_4 + 2 \text{ MnSO}_4 + 3 \text{ H}_2\text{O}_4$$

The method U. S. P. X follows:

Dry about one Gm. of sodium nitrite to constant weight over sulphuric acid, dissolve salt in sufficient water to make 100 cc. and add 10 cc. of this solution to a mixture of 50 cc. of tenth normal potassium permanganate, 100 cc. of water and 5 cc. of sulphuric acid; warm the liquid to 40° C., allow to stand for 5 minutes and titrate with tenth normal oxalic acid.

The reduction of the permanganate, however, is incomplete, and a precipitate of manganese dioxide results, which may be represented by the equation:

$$4 \text{ HNO}_2 + 2 \text{ KMnO}_4 + 2 \text{ H}_2\text{SO}_4 \longrightarrow 4 \text{ HNO}_3 + \text{K}_2\text{SO}_4 + \text{MnSO}_4 + \text{MnO}_2 + 2 \text{ H}_2\text{O}_4$$

The method was tried at the specified temperature and at temperatures of 60° , 75° and 100° with the same result. The oxalic acid reacts slowly with the manganese dioxide in back titration, and the absence of precipitate is the only indication of an end-point.

It is suggested that either of two modifications of the U. S. P. X method be adopted.

First.—Proceed as in the tenth revision method to the titration with oxalic acid, and instead, cool the mixture, add potassium iodide and titrate the liberated iodine with sodium thiosulphate.

Second.—As for foregoing, but instead, add a measured quantity in excess of tenth normal oxalic acid; heat to 80° and titrate with tenth normal potassium permanganate.

These modifications were checked against a solution representing approximately one per cent sodium nitrite with the following results:

First method, 1. 98.15%; 2. 98.24%. Second method, 1. 98.11%; 2. 98.04%.

The methods here reported are not new, but are offered as more practical and accurate for the assay of sodium nitrite.

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