

TABLE IV.—VOLUMETRIC TITRATION OF STRYCHNINE SULPHATE.

Strychnine Weighed Out. Gm.	Strychnine Recovered. Gm.	Per Cent Recovery.
0.1381	0.1594	115.4
0.2379	0.2807	118.0
0.0752	0.0879	117.0
0.0832	0.0978	117.6
0.1331	0.1556	116.9
0.1083	0.1268	117.1

Molecular weight, strychnine sulphate, $C_{21}H_{22}O_2N_2 = 334.19$.

Molecular weight, strychnine silicotungstate, $2H_2O \cdot SiO_2 \cdot 12WO_3 \cdot 4C_{21}H_{22}O_2N_2 = 4217.5$.

Therefore, 1 cc. acid = $\frac{2 \times 334.19}{294.19} \times 0.0057 = 0.01295$ Gm. strychnine silicotungstate.

SUMMARY.

1. Night blue has been found to serve as an excellent indicator for the silicotungstic titrations of alkaloids.
2. The method developed here is rapid and useful in routine analysis. If the method for the analysis of cinchona bark suggested by North and Beal (4) and modified by Coleman and Beal (2) is accepted as official, the analysis would be simplified by the use of this indicator.
3. The method developed is applicable to the analysis of other amines which form insoluble precipitates with silicotungstic acid.
4. All phases of the discussion presented in this paper are being further investigated.

LITERATURE CITED.

- (1) E. R. Caley, *Ind. Eng. Chem., Analyt. Ed.*, Vol. 2, No. 1 (1930), 77.
- (2) Coleman and Beal, Unpublished Thesis, University of Illinois (1928).
- (3) North and Beal, *JOUR. A. PH. A.*, 13 (1924), 889.
- (4) North and Beal, *Ibid.*, 13 (1924), 1001.
- (5) A. G. Scroggie, *J. Am. Chem. Soc.*, 51 (1929), 1057.

UNIVERSITY OF NORTH DAKOTA,
GRAND FORKS.

GASOMETRIC ANALYSIS OF SODIUM NITRITE IN COATED TABLETS, IN THE PRESENCE OF A BICARBONATE AND NITRATE.*

BY LESTER C. DICK.

Powder a sufficient number of tablets in a mortar, equivalent to about ten grains of sodium nitrite. Extract the powdered material with water until 250 cc. has been collected, testing to make certain that all sodium nitrite has been extracted. This solution will contain the bicarbonate, nitrate and the sodium nitrite.

Twenty-cc. aliquots are used for the determination, transferred to a small beaker and the bicarbonate neutralized with $N/2$ hydrochloric acid using methyl orange as the indicator.

The above solution is carefully transferred to the measuring cup of a Lunge nitrometer and introduced into the measuring tube. Follow this with 5 cc. of dilute

* Scientific Section, A. PH. A., Toronto meeting, 1932.

sulphuric acid and take a reading on the graduated equilibrium tube. Now add exactly 5 cc. of potassium iodide solution and add this to the reading previously taken. As the evolution of gas lessens rotate the measuring tube, being certain to maintain it at a higher level than the equilibrium tube. After the evolution of gas has completely stopped the liquid columns are adjusted to the same level and a reading taken on the equilibrium tube. The above reading minus the reading taken after the addition of the sulphuric acid plus the 5 cc. of potassium iodide solution, gives the cc. of gas evolved.

A blank determination is run by making up a standard solution of sodium nitrite containing 10 grains in 250 cc. of a solution containing approximately the same amounts of the bicarbonate and nitrate as the unknown. This solution is assayed by the foregoing method, the cc. of gas noted and adjusted to normal conditions. The amount of sodium nitrite taken, as represented by the aliquot divided by the cc. of gas evolved, gives the factor for 1 cc. of gas under these conditions.

The following results were obtained by this method:

Solution A—containing in 250 cc., 0.648 Gm. of sodium nitrite, 5 Gm. sodium bicarbonate and 2.0 Gm. potassium nitrate; 20-cc. aliquots representing 0.05184 Gm. of sodium nitrite gave readings of 17.7 cc. of gas and 17.5 cc.

Solution B—containing in 250 cc., 1.296 Gm. of sodium nitrite with the same amounts of the nitrate and bicarbonate as solution A; 10-cc. aliquots of the above solution gave readings of 17.5 and 17.6 cc. of gas.

The average of the readings was taken and the equivalent of 1 cc. was found to be 0.002948 Gm. sodium nitrite at 25° C. and 760 mm.

Tablets containing sodium nitrite, sodium bicarbonate and potassium nitrate were assayed by this method and the following results recorded:

Labeled.	Theoretical Na NO ₂ Content.	Quantity Determined.	Percentage Found.
1-	0.0324 Gm.	0.0330 Gm.	101.85
2-	0.0324 Gm.	0.03376 Gm.	104.19
3-	0.0324 Gm.	0.03211 Gm.	99.10
4-	0.0324 Gm.	0.03156 Gm.	97.40

CONTROL LABORATORY,
G. S. STODDART & Co., INC.,
NEW YORK.

SEPARATION OF ALKALOIDS BY BUFFERS.

Acetic acid plus sodium acetate and ammonia plus ammonium chloride are used as buffers. Harmine and harmaline are separated by first buffer by dissolving one Kg. of the hydrochlorides in 10 kilos water at 80–85° C. Solution is filtered through animal charcoal and treated with three kilos sodium acetate, which precipitates 80% of pure harmine. To separate rest of harmine, operation is repeated on mother liquor. Strychnine is separated from brucine by dissolving one part of mixed salts in 10 parts of 10% ammonium chloride and treating with 10% ammonia water at 80–85° C. until precipitation is complete. Strychnine is filtered and cooled mother liquor is treated for brucine with sodium hydroxide. S. Elgazin, *Khim. Farm. Prom.* (1932), 128–131. Through *Drug and Cosmetic Industry*.