The determination of the bromoform of crystallization by loss in weight on drying was unsuccessful. A sample kept under vacuum for 18 hours at 20° lost about 24%. When it was dried at 80° for 3 hours the total loss amounted to about 40%, which was not appreciably increased by longer drying at this temperature. Further drying at successive higher temperatures progressively raised the loss. At 120° C. the loss was nearly 70%, but the residue appeared much decomposed. We, therefore, had recourse to the determination of the total bromine. This determination was made by the method of Bacon.[1]

The analysis of two samples gave the following results:

0.4013 Gm. substance required 31.58 cc. 0.1 *N* $AgNO_3$ equivalent to 62.9% bromine
0.4710 Gm. substance yielded 0.7240 Gm. AgBr equivalent to 65.5% bromine

The bromine found in the preparation approximates two mols of bromoform. The theoretical bromine for quinine hydrobromide with two mols of bromoform is 61.4%.

Attempts to titrate the hydrobromic acid, as was done in the case of the crystals containing chloroform of crystallization, failed. The acid found was higher than the theoretical amount corresponding to two mols of bromoform. The reason for this we ascribe to the decomposition of bromoform when in contact with alkali during the titration.

We expect to continue our investigation for an improved method for the preparation of the bromoform compound, and to study the physical and chemical properties of the new compounds.

SUMMARY.

Two crystalline compounds of quinine hydrobromide with trihalogenmethane of crystallization have been prepared and their composition established. One contains two and one-half mols of chloroform and the other two mols of bromoform of crystallization. Their chemical and physical properties will be reported at a later date.

MICROSCOPIC IDENTIFICATION OF EPHEDRINE WITH NITROUS ACID.

BY FREDERICK GRILL.*

It has been suggested that a more efficient means be developed of identifying ephedrine microscopically. Ephedrine appears to crystallize slowly with the usual alkaloidal reagents and sometimes the crystals are not characteristic. Tsiang and Brown (1) have obtained well-defined crystals from solutions of ephedrine using gold chloride, platinic chloride and Kraut's reagent. However, it would seem that the crystals appear rather slowly at times with the reagents mentioned.

The purpose of this paper is to present another means of identifying ephedrine by the formation of characteristic crystals, using nitrous acid.

The structural formula of ephedrine shows it to be a secondary amine of the aromatic series (2).

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[1] *J. Am. Chem. Soc.*, 31 (1909), 50.

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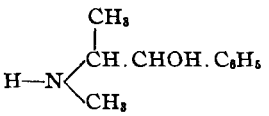
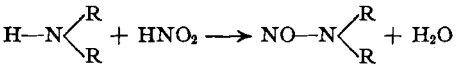
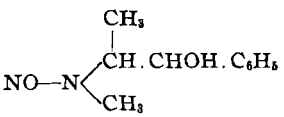


Secondary amines react with nitrous acid to form a nitroso compound according to the general equation:



It follows that ephedrine should react in a similar manner with nitrous acid to form a nitroso compound as follows:



The nitroso compound crystallizes and may be identified microscopically.

A solution of ephedrine hydrochloride (1:100) was treated as follows:

To one drop of the ephedrine solution on a slide, add one drop of a freshly prepared saturated solution of potassium nitrite, and one drop of a 6 *N* solution of sulphuric acid. An amorphous white precipitate was formed. After the liquid had evaporated, long crystals, with or without terminal branching, were observed.

The white precipitate does not always appear.

Comparative crystallizations were made using single reagents and combinations of reagents.

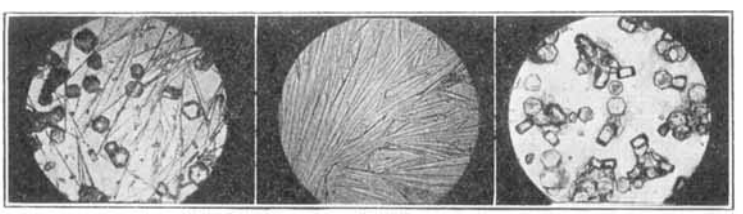


Fig. 1.—Crystals formed by treating ephedrine hydrochloride solutions with nitrous acid.

Fig. 2.—Ephedrine hydrochloride.

Fig. 3.—Saturated solution potassium nitrite and 6 *N* sulphuric acid.

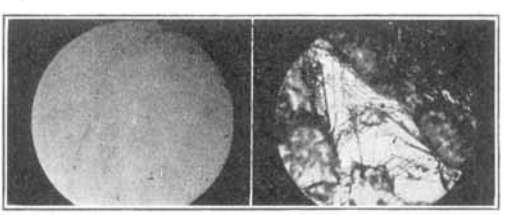


Fig. 4.—Ephedrine hydrochloride and 6 *N* sulphuric acid.

Fig. 5.—Ephedrine hydrochloride and saturated solution potassium nitrite.

Comparative crystallizations were made using the same procedure as outlined for ephedrine but substituting other alkaloids:

Adrenalin	Hexagonal plates.	Aconitine	Crystals not characteristic.
Morphine	Hexagonal plates.	Berberine	Crystals of berberine sulphate.
Quinine	No crystals.	Strychnine	Crystals not characteristic.

1:100 solutions of ephedrine appear to give the best crystals with nitrous acid. Crystallizations have been made from 1:500 solutions of ephedrine hydrochloride; however, solutions to be tested should be more concentrated. An excess of potassium nitrite was used in order to obtain a minimum residual sulphuric acid. An excess of sulphuric acid might cause the formation of ephedrine sulphate. There are other crystals formed in the procedure which are possibly potassium acid sulphate. The nitroso compound, presumably formed with ephedrine, may be extracted with ether and ephedrine hydrochloride reformed by the addition of dilute hydrochloric acid.

SUMMARY.

1. Ephedrine hydrochloride reacts with nitrous acid yielding a product which is, perhaps, a nitroso compound analogous to the nitroso compounds formed by secondary amines of the aliphatic series and nitrous acid.

2. The reaction appears to give characteristic crystals for ephedrine.

3. The method is rapid and quite easy to carry out.

REFERENCES.

(1) Tsiang and Brown, JOUR. A. PH. A., 4 (1927), 294.

(2) C. W. Porter, "The Carbon Compounds," 2nd Revised Edition (1931).

ENDOWMENTS FOR PHARMACY.

The following resolution (No. 17) was adopted at the Toronto meeting of the AMERICAN PHARMACEUTICAL ASSOCIATION. It is hoped that pharmacists will bear this resolution in mind whenever a favorable opportunity is presented.

WHEREAS, many large fortunes have been built largely through sales in retail drug stores and through other pharmaceutical activities, and

WHEREAS, the possessors of many of these fortunes have not been made acquainted with the endowment needs of pharmacy at a time when they were planning the disposition of their wealth, with the result that the proportion devoted to pharmacy, as compared with other professions is not in keeping with its importance and with its services to humanity, and

WHEREAS, the needs of the American Institute of Pharmacy in Washington, of the many Schools and Colleges of Pharmacy throughout the land, of the necessary researches and surveys in the professional and economic phases of pharmacy, particularly in the improvement of standards and of proper publicity for pharmacy are very great and pressing, if the profession is to fully discharge its obligations, and

WHEREAS, such endowments would not only place pharmacy in a position conforming to its importance but would also enable it to increase its contributions to the comfort and safety of life, and

WHEREAS, it is necessary and timely that it be explained in a dignified but forceful manner, that a proper proportion of the means made in pharmacy should be devoted to its advancement

Resolved that the AMERICAN PHARMACEUTICAL ASSOCIATION coöperate with other organizations in this important effort.