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# ASSAY OF AROMATIC SPIRIT OF AMMONIA, U. S. P. X.\*

BY J. D. BOOTH.1

## INTRODUCTION.

Since 1820, and up to the 10th Revision of the U. S. Pharmacopæia, Aromatic Spirit of Ammonia remained without an assay, although specifications of the alcoholic content and the specific gravity were made.

Kebler<sup>2</sup> reports a method for the assay which is as follows:

"Determine the total alkalinity by adding excess acid and titrating with an alkali. In a second sample precipitate the ammonium carbonate as barium carbonate, filter, add excess acid to the filtrate and titrate with an alkali. This titration represents the free ammonia. The difference in the two titrations represents the ammonium carbonate present."

Of the fifty-two samples which he assayed only eighteen per cent came within a ten per cent variation of the standard in the free ammonia content, and even a greater variation in the ammonium carbonate content. Due to the unsatisfactory results obtained, it was thought that by converting the ammonium carbonate to sodium carbonate and ammonium hydroxide that a more accurate assay could be developed.

- \* This paper is a development of a thesis presented to the faculty of the Philadelphia College of Pharmacy and Science as a part requirement for a Ph.C. degree, 1930.
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- <sup>2</sup> L. F. Kebler, "The Quality of Some Drug Available on the Market and Purchased on Prescription, with Method of Analysis," JOUR. A. PH. A., 6 (1917), 615.

#### EXPERIMENTAL.

Ammonium Carbonate and Free Ammonia.—Place a 10-cc, sample in an Erlenmeyer flask containing 30 cc. N/2 sulphuric acid, boil for three minutes and titrate the residual acid with N/2 sodium hydroxide. This represents the total alkalinity (reading A). To another 10-cc, sample, add 30 cc. N/2 sodium hydroxide, boil until free from the odor of ammonia, cool and titrate to neutrality with N/2 sulphuric acid, using phenolphthalein as an indicator. Note the reading on burette (reading B), add three drops of methyl orange and continue the titration (reading C). Sodium bicarbonate is neutral to phenolphthalein and alkaline to methyl orange; therefore, the difference in the titrations (B) and (C) represents one-half of the carbonate present. Subtract the calculated ammonium carbonate from the total alkalinity, the difference represents the ammonia in a free state.

Alcoholic Content.—Place a 20-cc. sample in a distillation flask, add 5 cc. (1-1) sulphuric acid and 200 cc. distilled water. Distil off 90 cc. and make up to exactly 100 cc. with distilled water. Determine the specific gravity by the use of a pycnometer, and calculate the per cent alcohol. The result represents one-fifth of the alcoholic content of the original solution.

Specific Gravity.—Determine the specific gravity of a new sample by the method given above, or by the use of a Westphal balance.

There being variations in the U. S. P. requirements for ammonia water and ammonium carbonate, several samples were prepared from assayed ingredients to contain the minimum and maximum amounts.

### VARIATION IN SAMPLES.

Sample A was prepared on a basis of 30 per cent ammonium carbonate and standard ammonia water. Sample B was prepared on a basis of 32 per cent ammonium carbonate and standard ammonia water. Sample C was prepared on a basis of 31 per cent ammonium carbonate, standard ammonia water, and to contain 68 per cent alcohol in the finished product. Sample D was prepared on a basis of 39 per cent ammonium carbonate, 10.5 per cent ammonia water, and to contain 62 per cent alcohol in the finished product.

RESULTS.				
Sample,	Specific Gravity.	Per Cent Free Ammonia.	Per Cent Ammonium Carbonate.	Per Cent Alcohol.
A	0.896	0.370	4.180	62.50
В	0.899	0.330	4.230	61.50
C	0.895	0.360	4.380	68.20
D	0.901	0.390	4.410	62.50

## CONCLUSION.

- 1. Ten samples of each of the various samples prepared were assayed with less than 0.2 per cent variation.
- 2. The results of this experimental work show that this is an accurate method for the determination of ammonium carbonate and free ammonia in the official Aromatic Spirit of Ammonia.

# GIFT OF BOOKS.

A collection of books pertaining to public health, assembled by the late Dr. John S. Fulton, first full time state health officer for Maryland, and executive officer of the state department of health for nearly thirty years, was recently presented to the department's library by Mrs. Fulton. There are more than a hundred volumes in the collection, in addition to pamphlets and charts.