

DISCOLORATION OF AROMATIC SPIRIT OF AMMONIA.*

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Aromatic spirit of ammonia has been used extensively in the practice of medicine for the last few centuries. When it is diluted sufficiently with water, aromatic spirit of ammonia, due to its reflex action and palatability, has been used chiefly to correct the hyperacidity of the stomach, more especially when accompanied by nausea or sick headache. Early records indicate that this preparation was recognized first officially in the London Pharmacopœia of 1721 under the name of *Spiritus Salis Volatilis Oleosus* (1). Shortly afterward this name was changed to *Spiritus Ammoniaë Aromaticus*. Since then various modifications have been suggested in the method of manufacture and in the formula from time to time, but the present American formula has been in use for a number of years without any change.

The fact that this product discolors readily is indicated by the following description given in the U. S. P. XI for aromatic spirit of ammonia: "a nearly colorless liquid when freshly prepared, but gradually acquiring a yellow color on standing." Since discoloration may be indicative of some change that has taken place in the galenical preparation subsequent to manufacture, it was thought desirable to investigate the possibilities of overcoming such a tendency. Upon making a review of the literature, very little could be found about discoloration, or methods to overcome it. Various methods of storage have been suggested to overcome this tendency, and the present U. S. P. has incorporated some of these ideas, giving instructions to preserve the spirit in glass-stoppered bottles in a cool place protected from light.

Maisch (2), in view of the fact that essential oils were not the only volatile constituents of plants, suggested the continuation of making the spirit by distillation from the drugs (same as U. S. P. 1840 procedure) since by following such a procedure the appearance was improved. A. Gertrude Flanders (3), after experimenting on a number of different pharmacopœial processes including suggested modifications for the preparation of this spirit, concluded that distillation was necessary in order to obtain a colorless spirit.

In the seventh revision of the U. S. P., oil of myristica was substituted for the oil of pimenta to prevent the tendency to darken (4), and since that time the formula has not been changed.

Thresh (5), some fifty-five years ago, proposed to avoid the distillation of the ammonium carbonate in making this preparation. The present British Pharmacopœia directs that this spirit be made by the same procedure. The amount of oils distilled by such a method is not definite and this offers a primary objection to the formula. The method has an advantage in the fact that a spirit made in such a manner will not discolor readily, although it will change color in time (6). Seward (7) suggested a modification of the method of the British Pharmacopœia of 1885. He recommended introducing less water into the still, distilling slowly and thereby securing all of the modified spirit in the distillate.

* Presented before the Section on Practical Pharmacy and Dispensing, A. Ph. A., Minneapolis meeting, 1938.

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Pollard (8) suggested still another modification. He proposed to avoid the distillation of the alcohol, and instead, use the distillation of the oils with water to obtain the first portion of the product, rejecting certain fractions toward the last, the presence of which he maintained would impair the delicate aroma.

It was the opinion of the authors that the terpene content of the oil of lemon might be a possible factor in the discoloration of the spirit upon aging. Accordingly, several experiments were set up for comparison, based upon the terpene content of the oil of lemon, using the various oils which could be purchased on the open market, *i. e.*, oil of lemon, Sicilian, U. S. P., oil of lemon cold pressed Californian, U. S. P., and oil of lemon (terpeneless), the latter containing not less than 60 per cent citral. The citral content of the various oils of lemon was determined by the assay method outlined in the U. S. P. IX. The following table gives the resulting data:

	% Citral By Weight.
1. Oil of lemon, U. S. P. (Sicilian)	4.84
2. Oil of lemon, U. S. P. (Sicilian)	4.32
3. Oil of lemon cold pressed Californian, U. S. P.	2.62
4. Oil of lemon (terpeneless)	71.10

By preparing aromatic spirit of ammonia from the above oils, and calculating the equivalent amounts, based on the citral content of the oil of lemon, the following results were obtained:

Experiment Number.	Procedure.	Oil of Lemon Used.	Results after 30 Days' Aging.	Results after 60 Days' Aging.	Results after 120 Days' Aging.
1.	U. S. P. XI	No. 2	Slight color	Slight color	Slight color
2.	U. S. P. XI	No. 2	Slight color	Slight color	Slight color
3.	U. S. P. XI	No. 4	Trace	Trace	Trace
4.	U. S. P. XI	No. 3	Slight color	Slight color	Slight color
5.	B. P.	No. 2	Colorless	Colorless	Trace

Several things were noticed in making the above experiments. Spirit No. 3 was free from haziness when first made; consequently the rate of filtration was rapid and easy. Spirits No. 1 and No. 2 prepared following U. S. P. XI procedures were cloudy when finished, and required the aid of a filtering medium to clarify the finished product. This necessitated slower filtration, with the possible loss of ammonia. In following the instructions of the British Pharmacopœia in making experiment No. 5 some difficulty was encountered, as only 820 cc. of distillate could be obtained absolutely clear, whereas the British Pharmacopœia directs that 875 cc. of clear distillate be obtained. After 820 cc. of clear distillate had been obtained and held separately, the next distillate started to become milky as it came through the condenser, and a gradual rise in temperature occurred. However, the milky distillate was collected until the necessary volume was obtained, *i. e.*, 110 cc. The ammonium carbonate and the strong solution of ammonia were added and dissolved in this 110-cc. portion with subsequent filtration. This filtrate was added to the reserve of 820 cc. and enough distilled water added to make 1000 cc.

A second series of experiments was made in order that the results of the first series might be checked. The results are given in the following table:

Experiment No.	Procedure.	Oil of Lemon Used.	Results after 30 Days' Aging.	Results after 90 Days' Aging.
6.	U. S. P. XI	No. 2	Slight color	Slight color
7.	U. S. P. XI	No. 2	Slight color	Slight color
8.	U. S. P. XI	No. 4	Trace	Trace
9.	B. P.	No. 2	Colorless	Very slight
10.	U. S. P. XI	No. 3	Slight color	Slight color
11.	U. S. P. XI	No. 1	Slight color	Slight color

All of the experiments made by the U. S. P. XI procedure fell within the assay standards given, while those made according to the British Pharmacopœia were just under the lower limits. The following formula, the authors believe, will give a product which is equivalent to one produced by the U. S. P. XI method with the added advantages of easy filtration and of developing less color upon aging:

Ammonium carbonate, in translucent pieces	34.000 Gm.
Ammonia water	90.000 cc.
Oil of lemon (terpeneless)	0.667 cc.
Oil of lavender	1.000 cc.
Oil of myristica	1.000 cc.
Alcohol	700.000 cc.
Water, Dist. q. s. to make	1000.000 cc.

Follow directions given in the U. S. P. for making the spirit.

CONCLUSIONS.

1. Aromatic spirit of ammonia made by distillation does not discolor as readily as one made by simple solution, thereby confirming previous investigations.
2. A spirit made in accordance with the U. S. P. XI directions, but modifying the formula to use an equivalent amount of oil of lemon (terpeneless), will develop less color upon aging.
3. A spirit made using terpeneless oil of lemon is water-clear and can be filtered rapidly without danger of loss of volatile constituents.

REFERENCES.

- (1) Remington Practice of Pharmacy, 8th Edition, page 329.
- (2) Maisch, PROCEEDINGS, A. PH. A., page 303 (1864).
- (3) Flanders, A. G., *Proceedings, Mich. State Pharm. Assoc.*, 163-167 (1886).
- (4) U. S. P. Dispensatory, 22nd Edition, page 1018.
- (5) Thresh, *Pharm. J. and Trans.*, 13 (1883).
- (6) U. S. P. Dispensatory, 19th Edition, page 1171.
- (7) Seward, *Pharm. J. and Trans.*, 701 (1886).
- (8) Pollard, *Trans. British Pharm. Conf. (Year Book of Pharmacy)*, 408 (1911).

THE VALUE OF THE HOSPITAL TO THE PHARMACIST.*

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Last year at our New York meeting an interesting paper was presented on the "Value of the Pharmacist to the Hospital."

* Presented before the Sub-Section on Hospital Pharmacy, A. PH. A., Minneapolis meeting, 1938.

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