

COMMENTS.

By reducing the size of the assay sample to correspond with 25 grains of powdered extract the filtration during extraction of the alkaloids is considerably facilitated.

The first ether extraction, which is made in an acid medium, removes all of the available ether-soluble matter from the tablet sample, including much of the chlorophyl. Unless this material is removed the color of the indicator will be so masked in the titration as to make it difficult to determine the final end-point. This discarded ether extract has been found to yield negative results in the test for alkaloids. The alkaloids are then removed by ammoniacal ether extraction until complete exhaustion of the assay sample is obtained. The tablet filler remains behind with the exhausted drug and presents no difficulty in making subsequent extractions.

A distinctive advantage in the method is found in the fact that it requires a minimum amount of working time on the part of the analyst.

CONCLUSIONS.

1. A method is proposed for the assay of Tablets of Belladonna Extract which appears to be distinctly superior to a modified procedure based upon a combination of the U. S. P. X and the U. S. P. XI assay methods for Extract of Belladonna.

2. In principle this method may be advantageously applied to the determination of mydriatic alkaloids in other related pharmaceutical products.

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NOTE ON ASSAY OF CHLOROFORM LINIMENT, UNITED STATES
PHARMACOPŒIA XI.*

BY SAMUEL W. GOLDSTEIN AND WILLIAM F. REINDOLLAR.

The method adopted by the Revision Committee of the United States Pharmacopœia XI for the determination of chloroform in chloroform liniment appears to be unnecessarily complicated, and in the hands of an inexperienced operator it is doubtful whether even approximate results would be obtained. Simpler procedures have been reported by Kunke (1) and Beal and Szalkowski (2).

Moraw (3) and Willgerodt (4) found that distillation methods applied to the determination of chloroform yielded low results. Beal and Szalkowski (2) used both the distillation and pressure bottle procedures and reported slightly lower results with the distillation method.

We found that, according to results obtained using the United States Pharmacopœia XI assay procedure, many of the commercial samples tested ran below the lower limit¹ prescribed for chloroform in chloroform liniment by the United States Pharmacopœia XI; therefore the following experiments were carried out.

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¹ Since this work was completed the lower limit for chloroform in chloroform liniment was changed in the First Supplement of the United States Pharmacopœia XI from 40 Gm. to 35 Gm. of chloroform in 100 cc. of the liniment.

Reagents.—The reagents prescribed in the United States Pharmacopœia XI were used with the exception that nitric acid was employed in place of diluted nitric acid.

Chloroform Liniment.—A. 44.2347 Gm. (30 cc.) of chloroform U. S. P. (99.50%) were accurately weighed and made up to 100 cc. with soap liniment at 25° C.

B. 44.2926 Gm. (30 cc.) of chloroform U. S. P. (99.37%) were accurately weighed and made up to 100 cc. with soap liniment at 25° C.

In each case, except where noted, 10 cc. of the chloroform liniment was diluted to 100 cc. with alcohol. Determinations of chloroform were then made on these diluted solutions by the official procedure, and also by transferring 10 cc. directly into a pressure (or citrate) bottle containing 50 cc. of the alcoholic potassium hydroxide solution (3 in 10). When the pressure bottle method was used, the bottles and contents were placed in a wire basket which was set in a bath of cool water and covered with a towel. The water was then heated to boiling and maintained at that temperature for a half hour. The bottle was then cooled, the contents were transferred to a 500-cc. volumetric flask, and the official procedure was carried out from that point with the following exceptions. Kunke (1) found that it was unnecessary to evaporate the alcohol before acidifying with nitric acid preliminary to determining chloride by the Volhard method. After checking his results in several experiments this step was deleted. The use of nitric acid instead of diluted nitric acid was found to be necessary; inasmuch as the ferric ammonium sulfate T.S. colored the solution to such an extent that a sharp end-point could not be obtained in the titration. In certain cases the chloride was determined gravimetrically.

TABLE I.—RESULTS OBTAINED WITH STANDARD LINIMENT.

Sample.	U. S. P. XI Method.		Pressure Bottle Method.		Gravimetric.	
	Gm. Found/ 100 Cc.	% CHCl ₃ Recovered	Gm. Found/ 100 Cc.	% CHCl ₃ Recovered.	Gm. Found/ 100 Cc.	% CHCl ₃ Recovered.
A	40.04	90.98	43.53	98.91
A	38.50	87.48	43.53	98.91
A	38.00	86.35	42.83	97.32
A	40.55	92.14	43.62	99.11
A	41.34	93.93	42.74	97.10
A	40.80	92.71	43.23	98.23	43.83	99.59
B	43.18	98.11	43.83	99.59
B	43.13	98.00	43.78	99.47
B	43.23	98.23	43.74	99.41
B*	43.58	99.02	43.83	99.59
B*	43.58	99.02	44.00	99.97

* In experiments Nos. 10 and 11, 1 cc. of chloroform liniment was measured with a 1-cc. pipette directly into pressure bottles containing 50 cc. of alcoholic potassium hydroxide.

The results indicate that with the pressure bottle method more accurate results are obtainable than when the official procedure is employed.

Proposed Method.—Place 50 cc. of alcohol in a 100-cc. volumetric flask, and measure exactly 10 cc. of chloroform liniment at 25° C. into the flask by means of a pipette, placing the tip of the pipette just beneath the surface of the alcohol. Make up to a volume of 100 cc. at 25° C. with alcohol, and mix thoroughly. Transfer 10 cc. of the alcoholic solution to a pressure (or citrate) bottle containing 50 cc. of an alcoholic solution of potassium hydroxide (3 in 10), holding the end of the pipette just above the alkali solution. Seal the bottle and place in a water-bath, heat the water to boiling and maintain at the boiling temperature for a half hour. Cool the mixture, and transfer it to a 500-cc. volumetric flask; rinse the reaction flask with distilled water, add the rinsings to the reaction mixture and finally add enough distilled water to make a volume of 500 cc. at 25° C. Mix the contents of the flask thoroughly and transfer a 100-cc. portion of the solution to a 250-cc. volumetric flask; then add a few drops of phenolphthalein T.S. and acidify with nitric acid, adding an excess of about 5 cc. of acid. Add 40 cc. of tenth-normal silver nitrate, shake until the precipitate coagulates, make up the volume with distilled water at 25° C. and mix thoroughly. Filter the supernatant liquid through a dry filter, rejecting the first 15 cc. of filtrate. Titrate 000

cc. of the filtrate with tenth-normal thiocyanate solution using 3 cc. of ferric ammonium sulfate T.S. as indicator. Carry out a blank determination, omitting only the chloroform liniment, and correct the volume of silver nitrate consumed in the assay by the volume consumed in the blank determination. Each cc. of tenth-normal silver nitrate is equivalent to 0.00398 Gm. of CHCl_3 .

The following results were obtained with commercial samples run by the official procedure and the pressure bottle method.

TABLE II.—RESULTS EXPRESSED AS GM. OF CHCl_3 PER 100 Cc.

Sample.	U. S. P. XI Method, Tinfoil on Cork Stopper.	U. S. P. XI Method, Tinfoil on Rubber Stopper.	Pressure Bottle Method.
1	35.47	37.11	41.34
2	28.06	37.76	40.30
3	...	32.14	34.18
4	...	33.98	40.99
5	...	40.99	43.48
6	...	36.47	38.95

DISCUSSION.

The proposed method eliminates distillation, the four-hour refluxing period, and a transfer of the reaction mixture; thereby decreasing the opportunity for error and at the same time greatly shortening the time required for the determination. Slightly higher results are obtained by the gravimetric modification of the recommended procedure.

SUMMARY.

1. A simpler and less time-consuming procedure for the assay of chloroform liniment is recommended.
2. The average recovery of chloroform by this procedure is 98.4 per cent.

REFERENCES.

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- (4) Willgerodt, T. M., *Am. J. Pharm.*, 97, 584 (1925).

HYDROGENATED CASTOR OIL IN OINTMENTS.—PART II COSMETICS.*

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Solidifying agents employed in cosmetics are chiefly waxes such as white beeswax, spermaceti, paraffin, ceresin, etc. Waxes differ from true fats in that they are not saponifiable (*i. e.*, not glycerides of fatty acids) and supposedly are not as readily absorbed by the skin. The use of animal fats, such as lard, is unsatisfactory because of the tendency to rancidity.

Hydrogenated castor oil was found satisfactory as a base for ointments (1). Since ointments are closely related to many cosmetics, the use of the oil as a substi-

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