A METHOD FOR ASSAYING UNGUENTUM STRAMONII.*

BY A. R. BLISS, JR., AND M. F. BROWN.

INTRODUCTION.

The Pharmacopœia of the United States, Ninth Decennial Revision[1] gives no method for standardizing Unguentum Stramonii. The official formula for this ointment calls for

Pilular extract of stramonium	10 Gm.
Diluted alcohol	5 mils
Hydrous wool fat	20 Gm.
Benzoinated lard	65 Gm.
To make *about*	100 Gm.

Since the official Extractum Stramonii[2] is required to yield not less than 0.9 per cent. nor more than 1.1 per cent. of the alkaloids of Stramonium, it follows that the official Unguentum Stramonii should yield, when made by the official formula above, *about* 0.1 per cent. of the alkaloids of Stramonium.

THE METHOD.

The following method, adapted by the authors from the assays of Extractum Stramonii,[3] Extractum Belladonnæ Foliorum,[4] and Fluidextractum Belladonnæ Radicis,[5] gives very accurate results as reported below in the experimental data.

Introduce 30 Gm. of Ointment of Stramonium into a 250-mil centrifuge flask. Then add 150 mils of a mixture of ether, *2 volumes*, and chloroform, *1 volume*, followed by 10 mils of ammonia water. Shake the mixture vigorously until all fatty matter is dissolved, and then continue to shake for three hours on a mechanical shaker. Allow the mixture to stand† until complete separation has taken place, and then decant 100 mils of the clear liquid, representing 20 (twenty) grams of the ointment, into a separator. Then extract the alkaloids from the solution in the separator by shaking out repeatedly with weak sulphuric acid until the alkaloids are completely removed. Collect the acid washings in a separator, add ammonia water until the solution is decidedly alkaline to litmus, and completely extract the alkaloids by shaking out repeatedly with chloroform. Evaporate the combined chloroform washings to dryness, dissolve the alkaloids from the residue in exactly 5 mils of tenth-normal sulphuric acid V. S. and titrate the excess of acid with fiftieth-normal potassium hydroxide V. S., using cochineal T. S. as indicator.

Each mil of tenth-normal sulphuric acid V. S. consumed corresponds to 28.92 milligrams of the alkaloids of stramonium.

EXPERIMENTAL DATA.

The Samples.—Samples of Ointment of Stramonium were prepared by accurately weighing 3 Gm. portions of Pilular Extract of Stramonium in 250-mil centrifuge flasks, warming on a water-bath until the extract was soft, adding 1.7 mils of diluted alcohol and working the alcohol into the extract with a glass stirring rod. Then 7 Gm. of melted hydrous wool fat were added to the contents of each flask and thoroughly mixed; and finally 21 Gm. of melted benzoinated lard were added to each flask in the same manner. The stirring rods were then removed, carefully wiped with small pieces of filter paper, and the pieces of filter paper then placed in the proper flasks. The 150-mil portions of the ether-chloroform mixture were then added, and the above assay carried. At the same time, samples of the same Pilular Extract of Stramonium (2 Gm.) were assayed. The results follow.

* Scientific Section, A. Ph. A., Cleveland meeting, 1922.

† The method may be hastened by centrifuging:

Sample Number.	Assay of ointment, Per cent.	Assay of extract, Per cent.
1	0.843	0.832
2	0.829	0.820
3	0.881	0.835
4	0.857	0.829
5	0.849	0.836
6	0.838	0.822
Average	0.8495	0.829

BIBLIOGRAPHY.

[1] U. S. P. IX, 1916, p. 482.
[2] U. S. P. IX, 1916, p. 161.
[3] U. S. P. IX, 1916, p. 161.
[4] U. S. P. IX, 1916, p. 144.
[5] U. S. P. IX, 1916, p. 178.

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HOT EXTRACTION OF DRUGS.*

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The problem of economical extraction of drugs is serious to the drug manufacturer. The increasing use of standardized preparations calls for a nearly complete extraction of the active or soluble constituents of a drug, and the amount of menstruum needed for this as well as the time involved and cost of concentrating the weaker percolate are all factors in the cost of the product. Any method of procedure which will hasten the exhaustion, or reduce the amount of percolate needed, will be likely to materially reduce the cost of manufacture and also produce a more stable product. For precipitation in galenical preparations depends in large measure upon exposure to light and air, and the less the volume of percolate the less is the exposure, and the smaller the precipitation.

Since heat favors the solution of plant constituents as it does of synthetic or natural chemicals, hot extraction has often been advocated for the more slowly exhausted drugs. But in drugs we have other factors beside the soluble or active constituents. It is desirable to extract the soluble or active constituents with as little of the inert materials as possible so that stability in the finished preparation may be promoted. Moreover, many drugs contain enzymes which act upon other constituents and either produce the desired properties, as in the case of cyanogen products in wild cherry, or of the volatile oil in the case of mustard, or else may destroy the active constituent as in strophanthus. The question of the influence of heat on these enzymes is therefore important. Then there is to be considered the action of heat on both the active constituents and on the inert. Few of the active constituents of drugs are injured by a moderate heat, if not too long continued and if protected, in part, from the action of air while hot. On the other hand, it was shown in the case of licorice[1] that the albuminoid principles of this drug are coagulated by

* Scientific Section, A. Ph. A., Cleveland meeting, 1922.

[1] "The Extraction of Licorice," JOUR. A. PH. A., 10, 688, 1921.