

Contributed and Selected

SOME SUGGESTED MODIFICATIONS OF THE PHARMACOPŒIA METHOD FOR THE ASSAY OF BELLADONNA LEAVES.¹

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The present Pharmacopœia method for the assay of mydriatic alkaloids, while very practical in principle, has several defects in the details of its manipulation which could be easily remedied and which would add much to the accuracy of the method in the hands of the average analyst. In a previous article² the writer described the use of a separatory funnel for both macerating and percolating the drug, thus doing away with the cumbersome and tedious operation of transferring the marc from a flask to a percolator. This transfer of the drug is undoubtedly one of the principal sources of error, especially when in the hands of an inexperienced analyst. The writer has used the long, narrow, Squibbs separatory funnel in hundreds of assays and has found it admirably adapted to the work.

It is desired in this article to draw special attention to the question of menstruum. The amount specified in the present Pharmacopœia is not sufficient to exhaust the drug completely except perhaps with exceedingly slow and careful percolation. It has been found by experience that the maceration liquid does not extract the bulk of the alkaloids as would be expected but that a large quantity is extracted by the percolation. In view of this fact it is essential that the percolation be very thorough and the quantity of menstruum specified in the official method is not sufficient to assure complete exhaustion in the majority of cases.

During the course of some studies on the cultivation of belladonna the writer found it necessary to devise an assay method which would be applicable to very small samples of drug. It was frequently necessary to work on quantities as small as two grams, while the maximum was never more than five grams. The Pharmacopœia method was followed in principal but several modifications were introduced. In order to insure complete exhaustion of the drug a large amount of menstruum was used, the amount decided upon being 50 cc. for maceration for a sample of drug weighing between two and five grams and 60 cc. for the percolation. This is slightly more menstruum than is specified for 10 grams of drug in the official method. The amount of ammonia water used in the extraction was in the proportion of 1 cc. to every gram of drug used. This is about twice the amount used in the official method and was selected because it causes the proper agglutination of the drug and also insures the presence of sufficient alkalinity in samples which contain far above the average percentage of alkaloids. The shaking out process with acidulated water and later with chloroform was used as in the official method. In titrating hematoxylin was used as indicator.

The following table shows the amount of menstruum and ammonia water used

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²Merck's Report, August, 1910, p. 215.

in the extraction and the percentage of alkaloids found in a sample of *Belladonna* leaves and a sample *Datura Tatula* leaves.

DRUG.	Weight of Sample (grams).	Amount of Menstruum (c.c.)		Amount of Ammonia Water (cc.)	Percentage Total Alkaloids.
		Maceration.	Percolation.		
Belladonna Leaves	5	50	60	5	0.623
	4	50	60	4	0.621
	3	50	60	3	0.618
	2	50	60	2	0.621
Datura Tatula Leaves	5	50	60	5	0.481
	4	50	60	4	0.481
	3	50	60	3	0.490
	2	50	60	2	0.488

The above table shows that the method as modified is well adapted to assaying small samples. While the amount of menstruum used is probably considerably larger than actually necessary to insure complete exhaustion there is no harm in such excess and it is a very convenient quantity to work with. The menstruum can readily be recovered and by means of its specific gravity adjusted to its proper proportion of ether and chloroform so that it may be used indefinitely.

The method described has been used with equal success on *Belladonna* Root and *Stramonium* Leaves. When used on *Hyoscyamus* Herb, the proportion of ammonia water could no doubt be reduced considerably although the method has given very good and concordant results without any modification.

In view of what has been written it would seem then that the official process could be improved by the following several changes:

1. The introduction of a suitable vessel or apparatus such as a Squibb separatory funnel for the combined maceration and percolation. This would save time and insure against loss of material through transference to a percolator.
2. An increase in the quantity of menstruum both for maceration and percolation. This would insure a more thorough exhaustion.
3. An increase in the quantity of ammonia water used, when extracting the drug. This would aid in the complete liberation of the alkaloids from their salts.

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THE PHARMACOPŒIAL REQUIREMENTS FOR *CANNABIS SATIVA*.*

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The United States Pharmacopœia, Eighth Revision, specifies that "*Cannabis Indica* shall consist of the dried prepared tops of the pistillate plant of *Cannabis sativa*, grown in the East Indies and gathered while the fruits are yet undeveloped,

*Presented to the Division of Pharmaceutical Chemistry of the American Chemical Society.