responsible for the absence of alkaloids in the rather delicate first-year growth. A similar instance is known to have occurred with other plants. *Hyoscyamus muticus*, a native of Egypt, for instance, is rich in hyoscyamine (1%); but when it was transplanted from the wilderness to the botanical gardens near Cairo, it was found to be free from alkaloids.

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THE ASSAY OF EPHEDRA VULGARIS.*

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Ephedra vulgaris has been variously reported as containing from about 0.2% to about 1% alkaloid. During the past year seven samples of the drug have been assayed by the writer using the method of the U. S. P. X for belladonna, but collecting the final chloroform solution in a tared flask, drying at a temperature of about 50° C. for one or two hours and weighing the alkaloidal residue before titrating. The results obtained in terms of total alkaloid were as follows:

No.	Gravimetric.	Volumetric.	No.	Gravimetric.	Volumetric.	No.	Gravimetric.	Volumetric.
1	1.42	1.25	3	0.93		6	1.21	0.86
	1.43	1.24		0.94			0.74[1]	0.55[1]
	1.42		4	1.17	1.01		0.84	0.66
	1.37[1]	1.15[1]		1.18	1.01		0.87	
							0.73[1]	0.56[1]
2	0.71	0.548	5	1.11	1.04		1.02	0.90
	0.64	0.495		1.13	1.09		0.99	
	0.74		6	0.705[1]	0.502[1]		0.88[1]	0.70[1]
	0.66[1]	0.48[1]		0.655[1]	0.472[1]			

No. 7. Titrated without drying, volumetric only, 1.02, 1.01, 1.15, 1.16.

[1] Alkaloid dried over night; others dried for one or two hours.

The factor used for the volumetric estimations was 1 cc. $N/10$ acid = 0.0165 Gm. alkaloid, using methyl-red indicator. The above results indicate that the alkaloid is quite volatile, although there is also more or less decomposition of the alkaloid in chloroform solution with the formation of hydrochloride. This, however, apparently does not take place under the conditions of the assay, as is indicated by

* Scientific Section, A. PH. A., St. Louis meeting, 1927.

the absence of chlorides in a number of residues tested after titration, although if the chloroform solution is allowed to stand for some time and then evaporated spontaneously, chlorides will be found in the residue. At a temperature of 100° C., the alkaloid is almost completely volatile; 20 cc. of a chloroform solution containing 0.87 Gm. alkaloid by volumetric estimation, after evaporating and drying for one hour at 100° C. weighed 0.0365 Gm., and after 5 hours 0.0045 Gm., and when titrated was found to equal only 0.1 cc. $N/50$ acid = 0.00033 Gm. ephedrine.

The use of ether for the extraction of the drug gives lower results than when ether-chloroform mixture (3–1) is used, and the final extraction of the aqueous solution with ether is much slower than with chloroform, although complete extraction may be obtained with seven or eight 25-cc. portions. The maceration of the drug in the ether-chloroform and ammonia mixture for two hours gives practically the same results as macerating for a longer period (12–48 hrs.). No trouble whatever with emulsions has been experienced.

To overcome the loss through volatility of the alkaloid the following modification of the U. S. P. X method for belladonna is suggested:

Use sodium hydroxide solution instead of ammonia in the extraction of the drug, ether and sodium hydroxide solution in extraction of the aqueous solution and add the volumetric acid solution to the ether solution before evaporating. Owing to the solubility of ephedrine in water it is not advisable to wash the ether solution, so care must be taken that none of the alkaline solution passes with the ether into the flask containing the volumetric acid; but repeated trials have shown that by allowing a thorough separation of the ether and then passing it through absorbent cotton well wet with ether this can be entirely avoided.

A number of other assays using various modifications were made and the results of these and of other work now under way will, it is hoped, appear in a later report.

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EPHEDRINE ASSAYS BY TITRATION.

BY H. O. MORAW.

The standards of the American Medical Association for ephedrine salts, include a gravimetric assay for the alkaloidal content. The only other published information about the assay of ephedrine applies to its determination in the crude drug. The U. S. P. IX assay for Belladonna Root was used in these instances.[1]

More than twelve hours are required for drying the base for the gravimetric determination by the A. M. A. method. It continues to lose weight for a time after that. A white sublimate either the alkaloid itself or a decomposition product continues to settle on the insides of desiccators in which it is dried. It cannot be heated or warmed without loss after the evaporation of the solvents with which it is extracted. It melts at about 40° C.

[1] JOUR. A. PH. A., 15 (1926), 9, 748; also 15 (1926), 12, 1070.