(11) Glasstone, "Electrochemistry of Solutions," page 77.
(12) F. Hahn and H. Clos, *Z. anal. Chem.*, 79 (1929), 26–37.

COLLEGE OF PHARMACY,
UNIVERSITY OF FLORIDA,
GAINESVILLE, FLA.

A STUDY OF THE U. S. P. X METHOD AND A MODIFIED METHOD FOR THE ASSAY OF OLEORESIN OF ASPIDIUM.*

BY A. RICHARD BLISS, JR.,[1] M. L. PABST,[2] R. W. MORRISON[3] AND E. O. PRATHER, JR.[1]

INTRODUCTION.

In their investigations of the chemical and the biological methods for the standardization of Oleoresin of Aspidium, Pabst and Bliss (1) found certain factors which they believe may lead to varying results when the method of the U. S. Pharmacopœia X (2), the British Pharmacopœia method (3), the Swiss Pharmacopœia method (4), the Fluck modification of the Swiss procedure (5), and the Swiss method as modified by Lyons (6) are employed. They presented a modification of the U. S. Pharmacopœia X method which is more reliable, more accurate and more expeditious. The investigation herewith reported concerned itself with a comparative study of the U. S. Pharmacopœia method and the Pabst-Bliss modification of the U. S. Pharmacopœia X method.

MATERIALS USED.

Four samples of Oleoresin of Aspidium were purchased. Chemical and physical examinations proved the absence of adulterants.

THE PROCEDURES FOLLOWED.

Procedure I: The U. S. Pharmacopœia X Method (2).—"Warm the oleoresin ôn a water-bath and stir until it is thoroughly mixed. Transfer about 5 Gm., accurately weighed, to a 200-cc. flask, dissolve in 40 Gm. of ether and add 100 Gm. of aqueous solution of barium hydroxide (3 to 100), and shake vigorously for five minutes. Allow the liquids to separate and filter off 86 Gm. of the aqueous fluid Transfer this to a separator, add sufficient hydrochloric acid to produce a distinctly acid reaction, and extract with three successive portions of 30 cc., 20 cc. and 15 cc. of ether. Draw off and combine the ethereal solutions, filter, wash the filter with ether, evaporate and dry the residue to a constant weight of 100° C. This residue weighs not less than 0.96 Gm., corresponding to not less than 24 per cent of crude filicin."

Procedure II: The Pabst-Bliss Modification of the U. S. P. Method (2).—Warm the oleoresin on a water-bath and stir until it is thoroughly mixed. Transfer about *3 Gm.*, accurately weighed to a 250-cc. flask, dissolve in *40 cc.* of ether, add *75 cc.* of a three per cent aqueous solution of barium hydroxide, and shake vigorously for 5 minutes. Transfer this mixture to a separator. Allow the liquids to completely separate, draw off and filter the barium hydroxide layer. Rinse the 250-cc. flask with two 25-cc. portions of a 3 per cent aqueous barium hydroxide solution. After each rinsing, transfer the barium hydroxide solution to a separator, shake for one minute, allow the liquids to separate completely, draw off and filter the barium hydroxide layer. Transfer the combined filtered barium hydroxide solutions to a separator, make distinctly acid (to litmus) with concentrated hydrochloric acid, and extract with three successive portions of 30 cc., 20 cc.

---

* Scientific Section, A. PH. A., Toronto meeting, 1932.

[1] University of Tennessee (Memphis).

[2] University of Buffalo.

[3] University of South Carolina.

and 15 cc. of ether. Draw off the combined ethereal solutions, filter, wash the filter with ether, evaporate and dry the residue to a constant weight at 100° C.

COMPARATIVE RESULTS.

The collaborators' results are assembled in the table below.

TABLE I.—ANALYSES OF OLEORESIN OF ASPIDIUM.

Analyst and Methods.	Sample I.	Sample II.	Sample III.	Sample IV.
Pabst, M. L.				
Mod. Meth.:	23.54*	24.50	23.52	24.42
U. S. P.:	24.76	25.71	24.59	23.61
Morrison, R. W.				
Mod. Meth.:	23.49	24.59	23.49	24.40
U. S. P.:	24.99	25.94	24.30	24.14
Prather, E. O., Jr.				
Mod. Meth.:	23.46	24.57	23.47	24.46
U. S. P.:	24.88	25.62	24.20	23.90
Bliss, A. R., Jr.				
Mod. Meth.:	23.53	24.53	23.52	24.39
U. S. P.:	24.69	25.80	24.46	23.88
Averages:				
Mod. Meth :	23.505	24.55	23.50	24.42
U. S. P.:	24.830	25.77	24.39	23.88
Variation:				
Mod. Meth.:	0.08	0.09	0.05	0.07
U. S. P.:	0.30	0.32	0.39	0.53

* Figures represent the per cent of filicin.

CONCLUSIONS.

The Pabst-Bliss modification of the U. S. Pharmacopœia X method is more accurate, more rapid, less complicated and more expeditious than the U. S. Pharmacopœia method.

The possibilities for error are reduced since the entire aqueous fraction is used instead of an aliquot portion. Less time is required in carrying out the modified method since it is not necessary to determine the specific gravity, or to weigh the barium hydroxide solution, the ether and the portion of the aqueous layer. The use of a smaller quantity of oleoresin results in a smaller amount of residue which requires less time for drying to a constant weight.

BIBLIOGRAPHY.

(1) M. L. Pabst and A. R. Bliss, Jr., JOUR. A. PH. A., 21 (1932), 431.

(2) *Pharmacopœia of the United States, Tenth Revision*, J. B. Lippincott Company, Philadelphia, Pa., 68, 247, 248.

(3) *British Pharmacopœia*, Constable & Company, Ltd., 10 Orange Street, Leicester Square, W. C. (1914), 156.

(4) *Swiss Pharmacopœia*, Fifth Edition, 281.

(5) H. Fluck, *Bull. sci. pharmacol.*, 34 (1927), 268–272.

(6) A. B. Lyons, "Practical Standardization by Chemical Assay of Organic Drugs and Galenicals," Nelson, Baker & Co., Detroit, Mich. (1920), 235–236.
