

BIBLIOGRAPHY OF PHARMACEUTICAL RESEARCH

Compiled by A. G. DuMez, Reporter on the Progress of Pharmacy.

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AN ASSAY METHOD FOR FLUIDEXTRACT OF IPECAC, U. S. P.*

BY MORTIMER BYE,¹ R. E. SCHOETZOW,² J. W. E. HARRISSON³ AND L. DALE SEIF.⁴

INTRODUCTION.

Several different methods for the assay of Fluidextract of Ipecac have been proposed since the method of the U. S. P. X has been found to yield troublesome emulsions. The methods of Palkin and Watkins (1), the automatic extractor method of Palkin, Murray and Watkins (2) and a modified U. S. P. IX method were reported by Bliss and collaborators (3). Other methods considered were those of Leger (4) and the British Pharmacopœia method (5).

After careful study of these methods two were thought to be worthy of further work by collaborators and a third method which is essentially the U. S. P. Type C Assay Method with certain modifications were proposed. These three methods were compared with the present U. S. P. X method.

METHODS.

1. *U. S. P. X Method.*—Follow U. S. P. Type Process D, using plenty of ether in the first shake-outs, continue extractions until no cloudiness results when tested with mercuric potassium iodide, if possible. Do not allow final ether solution to go to dryness on the steam-bath, but add 2 or 3 cc. of alcohol or ether to the last few cc. remaining; again evaporate to 2 or 3 cc. and add 0.1*N* H₂SO₄ before evaporating off last traces of solvent.

2. *Palkin and Watkins Method (Hand Extraction).*—Measure 20-cc. sample into a 100-cc. volumetric flask, add about 5 cc. *N* sulphuric acid and evaporate on a steam-bath with the aid of a current of air to about 10 cc. Then, while rotating the flask, add about 30 cc. of water; cool and make up to the mark with water. Mix well and allow to stand over night. Decant the supernatant liquid through a dry filter, rejecting the first few cc. Then proceed by either Method 2 or 3. Pipette 20-cc. filtrate (representing 4-cc. original sample) into a separatory funnel. Add 2 cc. of ammonia T.S. and completely extract the alkaloids with peroxide-free ether until no cloudiness results with potassium mercuric iodide. Evaporate the combined ether solutions on steam-bath and finish as directed in U. S. P. X, using precautions under No. 1 about evaporating last traces of solvent.

3. *Palkin, Murray and Watkins (Automatic Extractor).*—Pipette 20 cc. of filtrate (in Method 2) into an automatic extractor for liquids lighter than water which has been fitted to a 200-cc. flask. Add 50 cc. of water, 2 cc. of ammonia T.S. and 50.0 cc. of peroxide-free ether, shake gently to prevent deposition of solid matter on bottom of extractor; then add ether until about 75 cc. have passed into the flask. Extract on a steam-bath for about two hours. Separate

* Permission granted to publish by Chairman E. Fullerton Cook, Revision Committee, U. S. P.

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