

Another sample of fluidextract was assayed by the collaborators by the above method using asbestos or paper as absorbents. The results on both the first and second samples are given in Table IV.

TABLE IV.

| | Sample 1. | Sample 2. | |
|-------------------|-------------------------------------|---------------------------------|-----------|
| | Paper, Gm. Alkaloids/ 100 cc. | Paper, Gm. Alkaloids/100 cc. | Asbestos. |
| R. E. Schoetzow | 2.162 | 1.532 | 1.50 |
| J. W. E. Harrison | | 1.550 | 1.52 |
| | | | 1.61 |
| L. D. Seif | 2.213 | 1.55 | 1.56 |
| | 2.204 | 1.53 | 1.51 |

The average of three assays on Sample 1 is 2.193 with a low of 2.162 (1.4%) and a high of 2.213 (0.91%).

The average of 9 assays on Sample 2 is 1.54 with a low of 1.50 (2.59%) and a high of 1.61 (4.56%).

CONCLUSIONS.

A practical method for Fluidextract of Ipecac is presented which does not yield troublesome emulsions, and by which uniform results may be secured by different analysts.

The value of peroxide-free ether for the assay and the retention of alkaloids by sawdust is confirmed.

REFERENCES.

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MISCIBLE FLUIDEXTRACT OF IPECAC.*

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The chief factors to be considered in the manufacture of fluidextract of ipecac are (1) the choice of a menstruum which will exhaust the drug and (2) the obtaining of a fluidextract which will mix with syrup to form a permanent syrup of ipecac. Seventy-three per cent alcohol is effective as a menstruum but produces a fluid-extract which is not miscible with syrup. Thirty-seven per cent alcohol, as used in U. S. P. X, renders extraction difficult and does not remove the resins which cause clouding when the fluidextract is mixed with syrup.

A process has been suggested to Sub-Committee No. 11 of the Revision Committee of the U. S. P., wherein the drug is exhausted with 73% alcohol, the alcohol

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is then recovered by distillation and the fluidextract finished so as to contain 33% alcohol. The authors claim no credit for the method. This paper merely sets forth the results we obtained in following the method suggested.

METHOD.

| | |
|------------------------|--|
| Ipecac, in fine powder | 1000 Gm. |
| Menstruum | { alcohol 3 volumes { water 1 volume. |

Exhaust the drug by percolating slowly, after macerating 48 hours. Reduce the entire percolate to 1000 cc. by evaporation at a temperature not exceeding 60° C. and add 2000 cc. of water. Filter until brilliantly clear and evaporate the filtrate to 600 cc. To this add 300 cc. of alcohol, mix, assay and adjust with a mixture of 1 volume of alcohol and 2 volumes of water.

EXPERIMENTAL.

Duplicate assays on the powdered ipecac used showed 2.12% and 2.13% of ether-soluble alkaloids. Two 200-Gm. portions of drug were taken and a fluidextract prepared from each by the method previously described. The percolate from 200-Gm. portion No. 1 measured 2250 cc. when the drug was exhausted. That from 200-Gm. portion No. 2 measured 2150 cc. When these percolates were later concentrated, diluted with water, filtered and assayed, the filtrate from No. 1 contained a total of 3.48 Gm. of ether-soluble alkaloids, while that from No. 2 contained 3.49 Gm. This shows an average recovery of alkaloids amounting to 82%. This loss of alkaloids was due, certainly in a large part, to their removal in the dense resinous mass which was filtered off. According to the proposed method this mass is not washed to free it of alkaloids.

Two fluidextracts were previously prepared by this method from another lot of U. S. P. drug and made up to approximate volume without actually assaying them. Results are recorded in the following table.

TABLE OF ASSAYED FLUIDEXTRACTS.

| Sample No. | Age. | Condition. |
|--------------------------|-----------|------------|
| 1 | 6 months | Clear |
| 2 | 6 months | Clear |
| Unassayed Fluidextracts. | | |
| 1 | 14 months | Clear |
| 2 | 14 months | Clear |

Syrup of ipecac was prepared from each of these four fluidextracts. At the end of six months all were clear. The syrups made from the unassayed fluidextracts show a faint deposit at the end of fourteen months. Syrups were made from fluidextracts labeled "U. S. P.," obtained from two reliable manufacturing houses. Voluminous precipitates formed in them in the course of a few days.

Attempts were made to prepare fluidextracts by the U. S. P. X process from the two lots of powdered ipecac used in this work. The menstruum would not pass through the drug, even when no packing in the percolator was employed.

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