

saved by selection of proper doses, based upon the results of such tests. Furthermore, these results may often serve as a check upon the animal test.

SUMMARY.

1. The vitameter and antimony trichloride tests give results with a very high percentage of fish liver oils and concentrates which are in close agreement with the biological tests.

2. There are a few cases where the discrepancies are beyond the limits of error of the methods and indicate a lack of specificity of either the vitameter or color test. It is doubtful that either method can serve as an absolute substitute for the biological method.

3. Either the vitameter or color test serves a very useful purpose as a supplement to the biological test.

4. The antimony trichloride test gives results fully as reliable as the vitameter. In general both methods give results which are in close agreement with each other.

REFERENCES.

- (1) Rosenheim, O., and Drummond, J. C., *Biochem. J.*, 19, 753 (1925).
- (2) Carr, F. H., and Price, E. A., *Ibid.*, 20, 497 (1926).
- (3) Wokes, F., and Willimott, S. G., *Analyst*, 52, 515 (1927).
- (4) Norris, E. R., and Danielson, I. S., *J. Biol. Chem.*, 83, 469 (1929).
- (5) Morton, R. A., and Heilbron, I. M., *Nature*, 122, 10 (1928).
- (6) *Hilger Publication*, No 19 $\frac{1}{2}$ (Jan. 1934).
- (7) Army, H. V., and Taub, A., *JOUR. A. PH. A.*, 12, 839 (Oct. 1923).
- (8) Notevart, O., *Biochem. J.*, 29, 1227 (1935).

DRUG EXTRACTION. XVI. THE EFFECT OF THE FORM OF THE PERCOLATOR ON THE EFFICENCY OF EXTRACTION.*¹

BY WILLIAM J. HUSA² AND C. L. HUYCK.

Although a great many forms of percolators have been advocated in more than a century of percolation history there have been surprisingly few published reports of exact comparisons of the efficiencies of the different types. In 1878, J. U. Lloyd (1) measured the effect of varying the diameter and height of percolator tubes in the extraction of *cimicifuga*. He found that when maceration after packing was omitted a tenfold increase in the length of the tubes doubled the yield of extractive matter. Recently Büchi and Feinstein (2) carried out percolations of cinchona in order to determine the relative efficiencies of Oldberg percolators, funnels and cylindrical glass tubes of uniform diameter. The relative efficiencies varied somewhat at different stages of the percolation but from the standpoint of practical percolation the differences were surprisingly small. Hence it was concluded that the Oldberg percolator was as efficient as the other forms and was preferable from the standpoint of ease of packing and general convenience.

* Scientific Section, A. PH. A., New York meeting, 1937.

¹ This paper is based on part of a dissertation presented to the Graduate Council of the University of Florida by C. L. Huyck, in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

² Head Professor of Pharmacy, University of Florida.

In the present study, using powdered belladonna root, percolations were carried out to compare the efficiencies of the Oldberg percolator, funnel and glass tubes of uniform diameter.

EXPERIMENTAL PART.

Except where otherwise specified the drug and menstruum used and the methods of analysis employed were the same as in a previous study (3).

The sizes of the various percolators used are shown in Table I. The tubes of lowest capacity, *i. e.*, the 2.5-cm. tubes, had a capacity of 150 Gm. of powdered belladonna root; hence this quantity of drug was used in all the percolators in order to keep this factor the same. The drug was moistened with 25 cc. of menstruum per 100 Gm. of drug, placed in the percolators in portions with agitation and packed from the top. From preliminary experiments with the narrowest tubes it was found that when the drug was packed tightly from the top about fifteen hours was required for the liquid to reach the lower orifice, and the rate of free flow averaged about 0.079 cc. per minute for the first four hours. To increase the rate of flow, the narrowest tubes were packed more lightly in this experiment so as to allow a free flow of at least 0.4 cc. per minute. The relative tightness of packing is shown by the data on volume of packed drug in Table I. After the liquid had begun to drop from the lower orifice each portion of drug was allowed to macerate for twenty and one-half hours; this time of maceration was adopted mainly for convenience, since in a previous study by the present authors (4) it was found that maceration for forty-eight hours after packing caused only a very slight increase in efficiency of extraction. Percolation was then allowed to proceed at a rate of 0.4 cc. per minute. Two reserve percolates of 60 cc. each were collected from each percolator, the total of these two reserves being equivalent to the 80% reserve usually collected in making fluidextracts by ordinary percolation. In addition to the two portions of reserve, a weak percolate of 60 cc. was collected in each case. The room temperature was about 24° C. during the experiment. Certain experimental details are shown in Table I and the assay results appear in Table II. Two percolators of each type were used and the results given in the tables are the averages of the duplicate percolations.

TABLE I.—EXPERIMENTAL DETAILS.

	Length in Cm.	Inside Diameter at Top in Cm.	Volume of Packed Drug in Cc.	Length of Drug Column in Cm.	Time Required for Liquid to Reach Lower Orifice in Minutes.
Glass tube (2.5 cm.)	90.0	2.5	410	72	220
Glass tube (4 cm.)	64.0	4.0	400	30	114
Oldberg percolator	27.3	5.7	375	17.3	90
Funnel	15.5	17.5	370	9.5	75

TABLE II.—ASSAY RESULTS.

	Reserve I.	Gm. Total Alkaloids.			Reserve I.	Gm. Total Reserve II.	Extractive.	
		Reserve II.	Weak Percolate.	Total.			Reserve I.	Reserve II.
Glass tube (2.5 cm.)	0.55	0.10	0.03	0.68	9.2	8.0	6.4	23.6
Glass tube (4 cm.)	0.48	0.15	0.08	0.71	8.3	8.0	6.8	23.1
Oldberg percolator	0.60	0.15	0.03	0.78	7.8	7.8	7.2	22.8
Funnel	0.58	0.17	0.04	0.79	8.0	8.0	6.0	22.0

Comparison of Oldberg Percolator and Straight Glass Tube of Same Diameter.—Using powdered belladonna root from another shipment, percolations were carried out to compare the efficiency of the Oldberg percolator with that of a straight glass tube of approximately the same diameter. The Oldberg percolator used was the quart size having a length of 45.5 cm. and an inside diameter at the top of 7.5 cm. The glass tube was 70.5 cm. in length and had a uniform inside diameter of 6.5 cm. Portions of 500 Gm. each of belladonna root in No. 40 powder were moistened with the menstruum of 5 volumes of alcohol and 1 volume of water, 300 cc. of liquid being used to moisten each 500-Gm. portion of drug. The volume of the packed drug was approximately

1075 cc. in each case. The length of the drug column was 23 cm. in the Oldberg percolator and 28.5 cm. in the glass tube. Percolation was commenced without maceration before or after packing. In each case a first percolate of 250 cc. was collected in 23 hours and a second percolate of 250 cc. was likewise collected in 23 hours. The temperature was approximately 25° C. during the percolation. Analytical data on the percolates are given in Table III.

TABLE III.—COMPARISON OF OLDBERG PERCOLATOR AND STRAIGHT GLASS TUBE.

	Gm. Total Alkaloids.			Gm. Total Extractive.		
	First Percolate.	Second Percolate.	Total.	First Percolate.	Second Percolate.	Total.
Oldberg percolator	1.4	0.8	2.2	28.9	24.0	52.9
Straight glass tube	1.4	0.7	2.1	29.0	26.0	55.0

DISCUSSION OF RESULTS.

From the data in Table II both the Oldberg percolator and the funnel show more efficient extraction of alkaloids than either of the narrower glass tubes; this is true in each of the reserve portions as well as in the totals. Modern pharmacists may be surprised to note that the funnel gave just as efficient extraction of alkaloids as the Oldberg percolator, but this would not seem so strange to those pharmacists of past generations who advocated the use of funnels. The results on comparative efficiency of the Oldberg percolator, funnel and glass tubes are in general agreement with the results of Büchi and Feinstein (2).

The glass tubes gave a somewhat higher yield of total extractive in the first reserve but on the basis of the totals the difference does not appear to be of any great practical significance. The somewhat greater yield of total extractive given by the longer columns of drug is in agreement with the results of Büchi and Feinstein (2) on cinchona. In Lloyd's work on *cimicifuga* (1), the increased length of drug column apparently increased the yield of extractive matter more than was the case with either belladonna root or cinchona.

In the comparison of the Oldberg percolator with a glass tube of approximately the same diameter, the data in Table III show no appreciable advantage for either type.

SUMMARY.

Using powdered belladonna root, percolations were carried out to compare the efficiencies of the Oldberg percolator, funnel and glass tubes of uniform diameter. The results indicate that the Oldberg percolator and funnel gave more efficient extraction of alkaloids than narrower glass tubes, but that the latter were somewhat more efficient for the extraction of total extractive. In a comparison of the Oldberg percolator with a glass tube of approximately the same diameter, the data show no appreciable advantage for either form of percolator.

REFERENCES.

- (1) Lloyd, J. U., *Am. J. Pharm.*, 50, 1 (1878).
- (2) Büchi, J., and Feinstein, K., *Pharm. Acta Helv.*, 11, 121 (1936).
- (3) Husa, W. J., and Huyck, C. L., "Drug Extraction XV," *Jour. A. Ph. A.*, 27, 105 (1938).
- (4) Husa, W. J., and Huyck, C. L., *Ibid.*, 25, 312 (1936).