

DRUG EXTRACTION. VIII. THE EFFECT OF MACERATION AND RATE OF FLOW ON THE EFFICIENCY OF PERCOLATION.\*<sup>1</sup>

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Percolation experiments using 100-Gm. portions of belladonna root have indicated (1) that maceration causes a slight increase in efficiency of extraction of alkaloids and a definite increase in the proportion of total extractive in the reserve portion. Further experiments have been carried out on belladonna root, cinchona and nux vomica, using quantities of drug more comparable to the 1000 Gm. called for in official formulas.

EXPERIMENTAL PART.

*Experiment I.*—An experiment was carried out to determine whether or not 48 hours' maceration after packing had any influence on the efficiency of percolation either in slow percolation or fast percolation. Four portions of 800 Gm. each of belladonna root in No. 40 powder were percolated with a menstruum of alcohol 5 volumes—water 1 volume, each portion of drug being moistened with 480 cc. of menstruum. In the present experiment "slow percolation" is at a rate of 10 drops per minute for the reserve and 20 drops per minute for the weak percolate, while "fast percolation" is at twice this rate.

The effect of time of maceration and rate of percolation on the quantity of alkaloid and total extractive in the reserve portion of 640 cc. is shown in Table I.

TABLE I.

	Gm. Total Alkaloids in Reserve Portion.		Gm. Total Extractive in Reserve Portion.	
	No Macera- tion.	48 Hours' Maceration after Packing.	No Macera- tion.	48 Hours' Maceration after Packing.
Slow percolation	3.18	3.17	46.5	48.3
Fast percolation	2.96	2.99	47.7	43.6

In this experiment, maceration for 48 hours after packing does not show any advantage in extraction of alkaloids either in slow or fast percolation. In fast percolation the rate of extraction of alkaloids is somewhat lower than in slow percolation.

*Experiment II.*—An experiment was carried out to determine the efficiency of extraction at different rates of flow. Three portions of 800 Gm. each of belladonna root in No. 40 powder were percolated with a menstruum of alcohol 5 volumes—water 1 volume, each portion of drug being moistened with 480 cc. of menstruum and macerated for 15 minutes before packing and 48 hours after packing. The following rates of flow were used; very slow (0.5 cc. per minute); slow (1 cc. per minute); moderate (2 to 3 cc. per minute). The effect of rate of flow on the quantity of alkaloid and total extractive in the reserve portion of 640 cc. is shown in Table II.

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TABLE II.

	Rate of Percolation.		
	Very Slow.	Slow.	Moderate.
Gm. total alkaloids in reserve portion	3.13	3.13	3.12
Gm. total extractive in reserve portion	43.5	49.8	49.1

From this experiment it appears that percolation at the moderate rate of flow gives just as efficient extraction as the slower rates of percolation which were tested.

*Experiment III.*—In order to secure results showing different combinations of the various factors an experiment was carried out in which the rate of flow was kept constant at 2 to 3 cc. per minute in all the percolators and the other factors varied. Four portions of 800 Gm. each of belladonna root in No. 40 powder were percolated with a menstruum of alcohol 5 volumes—water 1 volume, each portion being moistened with 480 cc. of menstruum.

TABLE III.—EFFECT OF VARIATIONS IN MACERATION.

Hours of Maceration.		Gm. Total Alkaloids in Reserve Portion.	Gm. Total Extractive in Reserve Portion
Before Packing.	After Packing.		
0	0	3.3	77.1
6	0	3.3	77.2
0	48	3.4	79.6
6	48	3.4	80.7

The above results indicate that maceration after packing causes a slight increase in efficiency of extraction of alkaloids and total extractive. Maceration before packing seems to be of no importance.

*Experiment IV.*—An experiment was carried out to determine whether or not maceration for 48 hours after packing had any influence on the efficiency of percolation of yellow cinchona, either in slow or fast percolation.

Four portions of 500 Gm. each of yellow cinchona in No. 40 powder (assaying 10.0% alkaloids) were percolated, using the U. S. P. X Type Process B except for variations in maceration and rate of flow. Each portion of drug was moistened with 300 cc. of Menstruum I (glycerin 1 vol.—hydrochloric acid 1 vol.—alcohol 8 vols.) and macerated for 15 minutes before packing.

TABLE IV.—ASSAY RESULTS ON RESERVE PORTION.

Rate of Flow in Cc. per Minute.	Gm. Total Alkaloids in Reserve Portion.		Gm. Total Extractive in Reserve Portion.	
	No Macera- tion.	48 Hours' Maceration after Packing.	No Macera- tion.	48 Hours' Maceration after Packing.
1	24.4	26.2	164.9	175.8
3 to 5	22.6	25.7	147.7	170.8

This experiment shows a slight advantage for the 48-hour maceration period after packing; slow percolation gave slightly better results than fast percolation.

*Experiment V.*—An experiment was carried out to determine whether or not maceration for 48 hours after packing had any influence on the efficiency of percolation of nux vomica in either slow or fast percolation.

Four portions of 500 Gm. each of nux vomica in No. 40 powder (assaying 2.35% alkaloids) were percolated using Type Process B except for variations in maceration and rate of flow. The following menstrua were employed: Menstruum I: acetic acid 100 cc., water 150 cc., alcohol 750 cc.; Menstruum II: alcohol 3 volumes—water 1 volume. Each portion of drug was moistened with 300 cc. of Menstruum I and macerated for 15 minutes before packing.

TABLE V.—ASSAY RESULTS ON RESERVE PORTION.

Rate of Flow.	Gm. Total Alkaloids in Reserve Portion.		Gm. Total Extractive in Reserve Portion.	
	No Macera- tion.	48 Hours' Maceration after Packing.	No Macera- tion.	48 Hours' Maceration after Packing.
0.5 cc. per minute	8.95	9.97	53.0	54.8
1.5 to 2.5 cc. per minute	8.77	9.30	53.7	50.2

The efficiency of extraction of the alkaloids of nux vomica was increased slightly by the 48-hour period of maceration after packing and also by percolating at the slower rate.

## SUMMARY.

Maceration before packing is of no importance in increasing the efficiency of extraction of belladonna root in a percolation process; maceration after packing causes a slight increase in efficiency of extraction of total alkaloids and total extractive. Experiments on yellow cinchona and nux vomica also tend to show that maceration after packing increases the efficiency of extraction to a slight extent.

Slow percolation gave slightly more concentrated percolates than rapid percolation in case of nux vomica and yellow cinchona. Percolation of belladonna root at a moderate rate gave just as efficient extraction as the slower rates tested.

## REFERENCES.

- (1) Husa, William J., and Yates, S. B., *JOUR. A. PH. A.*, 24, 538-543 (1935).

## THE DETERMINATION OF ALCOHOL IN PHARMACEUTICAL LIQUIDS. I. A STUDY OF THE U. S. P. X AND U. S. P. XI METHODS.

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The United States Pharmacopoeia IX (2) was the first edition to carry a method for the determination of alcohol in liquids, and the method stated there was repeated without significant changes in the U. S. P. X (3). The method is based upon the well-known procedure of distilling the alcohol from the sample and determining the specific gravity of the distillate. Then by reference to the proper tables the alcohol percentage of the original sample can be found. Volatile substances other than alcohol and water which would affect the specific gravity are directed to be removed in so far as possible by saturating the distillate with sodium chloride, shaking it with petroleum benzin, and then redistilling the aqueous portion. This procedure is definitely time-consuming and the new U. S. P. XI method (4) is somewhat shorter and simpler, consisting of adding saturated sodium chloride solution to the sample, shaking it with petroleum benzin, and distilling the aqueous portion.