with an acidified solution. This was treated with tannic acid, Mayer's reagent, picric acid and phosphotungstic acid. Since in no case was a precipitate observed, it may be concluded that ether-soluble alkaloids can be present only in minute quantity.

In spite of extensive experiments for the purpose, no crystalline glucosides could be isolated. As is well known, there is no certain method of establishing the presence or absence of such substances, since they exhibit no uniform properties as a general rule. After rendering any enzyme inert by boiling water and purifying the resulting solution by normal and basic lead acetate, the filtrate was freed from lead and concentrated. Since nothing was obtained in this way, the residue was extracted by ethyl acetate and by other immiscible solvents, but without result. It was found, however, that the aqueous solution contained notable quantities of saponin which was not strongly hemolytic.

Attempts were also made to isolate any choline derivatives. Two pounds of the powdered plant were extracted with boiling, acidified alcohol and the resulting solution was evaporated to dryness under reduced pressure. The residue was purified by exhausting with ether and again dissolved in boiling, absolute alcohol. From this solution any bases were precipitated by treating with alcoholic mercuric chloride. The dark-colored product was then suspended in water and decomposed by hydrogen sulfide. The mixture was then evaporated to dryness, taken up with water and filtered. After evaporating the liquid to a small volume, it was treated with a solution of gold chloride, giving considerable reduction to metallic gold which was removed by filtration. The solution gradually deposited yellow crystals of a compound which answered the description of choline gold chloride, but the amount was too small to purify for analysis.

SUMMARY.

1. A proximate analysis of American mistletoe was made and a table of results is given.

2. Tannin, starch pentosans and saponins are present, probably also some derivatives of choline, but alkaloids were not found.

REFERENCES.

(1) Crawford, *J. Am. Med. Assoc.*, 57, 865 (1911).

(2) Hanzlik and French, *J. Pharmacol.*, 23, 269–306 (1924).

DRUG EXTRACTION. XII. THE EFFECT OF VARIATION IN PROPORTION OF MOISTENING LIQUID ON THE PERCOLATION OF JALAP.[1,2]

BY WILLIAM J. HUSA[3] AND PAUL FEHDER.

Experiments were carried out to determine the effect of varying the proportion of liquid used in moistening jalap on the efficiency of extraction.

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[1] Scientific Section, A. PH. A., Dallas meeting, 1936.

[2] This paper is based on part of a dissertation presented to the Graduate Council of the University of Florida by Paul Fehder, in partial fulfilment of the requirements for the degree of Doctor of Philosophy.

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HISTORICAL REVIEW.

Husa and Yates (1) reviewed the earlier work on the effect of variation in proportion of moistening liquid on the efficiency of percolation and carried out experiments on the percolation of powdered belladonna root which showed that there is a decrease in rate of extraction when the amount of moistening liquid is not kept down to a low proportion.

EXPERIMENTAL DATA.

Comparative percolations were carried out on 1000-Gm. portions of jalap in No. 60 powder, using varying amounts of menstruum for moistening the drug, but keeping all other factors as nearly constant as possible. The menstruum used was U. S. P. alcohol (95 per cent by volume). Four percolations were carried out, the quantities of moistening liquid used being 0, 250 cc., 500 cc. and 700 cc., respectively. A pledget of cotton was placed in the neck of each percolator and on this was placed 100 Gm. of purified sand. The drug was packed in portions. A sheet of filter paper was placed on top of the drug and covered with a layer of purified sand. Each portion of drug was macerated for 48 hours after packing.

The following portions of percolate were collected in each case: Reserve I, 400 cc., at the rate of 10 drops per minute; Reserve II, 400 cc., at the rate of 15 drops per minute; Weak Percolate, 1000 cc., at the rate of 20 drops per minute. The various fractions of percolate were assayed for resin by the N. F. VI method and for total extractive (non-volatile at 100° C.). The drug used contained 6.97 per cent resin by the N. F. VI method of assay.

By using graduated percolators it was possible to determine the volume of the packed drug. The volume of packed drug was 1400 cc. when no moistening liquid was used and 1440 cc., 1250 cc. and 1300 cc., respectively, when moistened with 250 cc., 500 cc. and 700 cc. of liquid.

TABLE I.—EFFECT OF VARIATION IN PROPORTION OF MOISTENING LIQUID.

A. Gm. of Resin in Various Portions of Percolate.

Percolates.	0.*	250 Cc.*	500 Cc.*	700 Cc.*
Reserve I (400 cc.)	45.6	57.1	50.9	36.6
Reserve II (400 cc.)	13.6	6.4	13.2	27.5
Weak Percolate (1000 cc.)	5.3	1.0	1.0	1.8
Total	64.5	64.5	65.1	65.9

B. Gm. of Total Extractive in Various Portions of Percolate.

Percolates.	0.*	250 Cc.*	500 Cc.*	700 Cc.*
Reserve I (400 cc.)	59.2	70.9	63.3	45.9
Reserve II (400 cc.)	22.5	16.6	23.8	37.3
Weak Percolate (1000 cc.)	15.5	10.4	12.4	14.7
Total	97.2	97.9	99.5	97.9

* Quantity of menstruum used in moistening 1000 Gm. of drug before packing.

DISCUSSION OF RESULTS.

The results in Table I indicate that in case of jalap there is more resin and more total extractive in the first portion of percolate when 250 cc. of moistening liquid is used than when no moistening liquid is used; as the quantity of moistening liquid increases from 250 cc. to 500 cc. and 700 cc. the first portion of percolate contains less resin and less total extractive. As far as present results go, it is concluded that jalap is extracted more rapidly if 250 cc. of moistening liquid is used for 1000 Gm. of drug than if 0 or 500 cc. or 700 cc. are used.

Husa and Yates (1) found that too much moistening liquid lowers the rate of

extraction of belladonna root and explained the results as follows: when no moistening liquid is used all of the menstruum must pass through the whole column of drug, while in case of a moistened drug each part of the moistening liquid goes through only that portion of the drug lying between it and the bottom of the percolator.

In comparing the present results on jalap with the previous results on belladonna root a difference is noted. That is, in case of jalap there is more resin in the first portion of percolate when the amount of moistening liquid is increased from 0 to 250 cc.; in case of belladonna root there was no increase in the percentage of alkaloids in the reserve portion when the amount of moistening liquid was similarly increased. In other words, the efficiency of extraction of jalap but not of belladonna root was increased by moistening with a moderate quantity of liquid. As the proportion of moistening liquid was further increased, there was a decrease in efficiency of extraction of both drugs.

The decrease in efficiency of extraction when excessive proportions of moistening liquid are used is readily understandable on the basis of the above-mentioned explanation of Husa and Yates. No definite hypothesis can be offered at this time to explain why small quantities of moistening liquid increase the efficiency of extraction of jalap but not of belladonna root; possibly the more uniform packing which is obtained with a moistened drug is more important for jalap than for belladonna root.

SUMMARY.

Percolation experiments indicate that the efficiency of extraction of jalap is greater when 250 cc. of moistening liquid is used for 1000 Gm. of drug than when the drug is packed in the dry state. As the proportion of moistening liquid is further increased there is a decrease in efficiency of extraction.

REFERENCES.

(1) Husa, W. J., and Yates, S. B., JOUR. A. PH. A., 24, 538 (1935).

THE STABILITY OF DIOTHANE SOLUTIONS. III.*,[1]

BY E. S. COOK AND T. H. RIDER.

It has been shown previously (1), (2) that diothane solutions when subjected to lengthy aging or sterilization undergo a slight alteration characterized by the liberation of aniline. The percentage decomposition was found to be very small—not more than 2.25% under the most stringent conditions—and is insufficient to affect the local anesthetic potency.

An extemporaneously prepared diothane solution shows no physical or chemical alteration or change in local anesthetic potency over a period of a few days if stored in pyrex containers. However, if such a solution is kept for longer periods of time, a slight change in p_H is observed together with the precipitation of the free base of diothane. We have followed the p_H changes of a number of 1% diothane solutions which were stored in pyrex bottles. The variation of p_H with aging of a typical solution is shown in Table I. The p_H determinations were made with the *quinhydrone electrode.*[2]

* Scientific Section, A. PH. A., Dallas meeting, 1936.

[1] From the Research Laboratories of The Wm. S. Merrell Company, Cincinnati, Ohio.

[2] See page 223 for foot-note.