Diheptanol Peroxide	0.2	2	0
	0.5	4	25
	1.0	3	0
	3.0	5	0
	5.0	1	0

These experiments indicate that organic ozonides and diheptanol peroxide generally are toxic to swine ascarids *in vitro* and are valuable in the treatment of dogs infested with ascarids. They show that ozonized cottonseed oil and diheptanol peroxide have a higher therapeutic index than oil of chenopodium in canine ascariasis, although they do not determine the index. The ozonized oil seems superior to the solid peroxide. The experiments suggest that organic ozonides and possibly peroxides may find wide application in the treatment of metazoan and protozoan disease, and offer a new group of organic compounds for exploitation by the experimental therapeutist. This seems timely just now when these diseases are being recognized as important, since the drugs so far put forward for their treatment have been limited to representatives of a few chemical classes, characterized generally by a too high toxicity for the host.

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DRUG EXTRACTION. XI. THE EXTRACTION OF JALAP.*[1]

BY WILLIAM J. HUSA[2] AND PAUL FEHDER.

In an earlier paper (1) a report was made of the effect of solvents in relation to swelling, penetration, imbibition and extraction of jalap. A further study has been made of the extraction of jalap, including such factors as fineness of powder, variation in solvents and methods of assay.

EXPERIMENTAL DATA.

Effect of Fineness of Powder on Percolation.—100-Gm. portions of jalap in Nos. 20, 40, 60 and 80 powders were percolated with alcohol, the percolates being collected in successive fractions of 100 cc., 100 cc. and 300 cc. in each case. Assay results on the percolates indicated that within the limits of No. 20 and No. 80 powder, the fineness of powder has practically no effect on the rate of extraction of resin by percolation.

Effect of Variation in Solvents on Rate of Extraction.—Percolation experiments were conducted using jalap in No. 60 powder and a series of alcohol-water mixtures. The drug used contained 9.4% moisture and the resin content was 7.3% by the U. S. P. X method of assay and 6.0% by Warren's assay method (2). In each case 200 Gm. of drug were moistened with 100 cc. of menstruum, packed in the percolator and menstruum added. After allowing a maceration period of 48 hours, percolation was allowed to proceed at a rate of 10 drops per minute, the percolate being collected in successive fractions of 200 cc., 200 cc. and 600 cc. The various fractions of percolate were assayed for resin and total extractive.

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[1] 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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TABLE I.—PERCOLATION OF JALAP WITH VARIOUS ALCOHOL-WATER MIXTURES.

A. Gm. of Resin in Various Fractions of Percolate (by Warren's Assay Method).

Percolates.	Absolute Alcohol.	95% Alcohol.	Alc. 9 Vol.— Water 1 Vol.	Alc. 4 Vol.— Water 1 Vol.
200 cc.	12.1	12.0	12.3	11.7
200 cc.	0.1	0.2	0.2	0.2
600 cc.	0.2	0.2	0.2	0.1
Total	12.4	12.4	12.7	12.0

B. Gm. of Resin in Various Fractions of Percolate (by U. S. P. X Assay Method).

200 cc.	13.2	13.5	13.7	14.3
200 cc.	0.2	0.5	0.4	0.3
600 cc.	0.1	0.2	0.2	0.2
Total	13.5	14.2	14.3	14.8

C. Gm. Total Extractive in Various Fractions of Percolate.

200 cc.	17.9	19.6	25.0	35.4
200 cc.	2.1	5.0	13.7	21.4
600 cc.	1.9	3.7	13.9	9.7
Total	21.9	28.3	52.6	66.5

The results in Table I show that the amount of resin obtained increases slightly with decreasing alcoholic content of the menstrua by the U. S. P. X assay, but is practically the same with each of the menstrua by Warren's assay method. The greatest portion of the extracted resin is contained in the first 200 cc. of percolate. The amount of total extractive increases with decreasing alcoholic content of the menstrua.

Comparison of U. S. P. X and N. F. VI Methods of Preparing Resin of Jalap.—The U. S. P. X used alcohol as a menstruum for the preparation of resin of jalap while the N. F. VI specifies a mixture of 9 volumes of alcohol and 1 volume of water as the menstruum. In the present study, tests were carried out to determine whether or not the change in official menstruum has any effect on the purity or yield of resin of jalap. Two different lots of resin of jalap were prepared from 500-Gm. portions of jalap in No. 60 powder, using alcohol as the menstruum; each 500-Gm. portion of drug was moistened with 250 cc. of menstruum before packing and there was a maceration period of 48 hours after packing. Percolation was carried out at the rate of 0.6 to 0.8 cc. per minute, 1250 cc. of percolate being collected. The resin was prepared from the percolate as specified in the U. S. P. X. Two portions of resin of jalap were also prepared from 500-Gm. portions of jalap, keeping all experimental details the same as before except that the N. F. VI menstruum of alcohol 9 volumes—water 1 volume was used.

The powdered jalap used was assayed by three different methods and the resin content was found to be as follows: U. S. P. X assay, 7.9%; N. F. VI assay, 7.1%; Warren's assay, 6.3%. The average yield of resin of jalap obtained from the two 500-Gm. portions of jalap by the U. S. P. X method was 6.6%, while by the N. F. VI method the yield was 6.7%.

TABLE II.—ASSAY RESULTS ON RESIN OF JALAP

Percentage of Resin by Various Assay Methods.

Method of Pre aration of Resin of Jalap.	U. S. P. X Assay.	U. S. P. X Assay (Corrected for Citrate Error).	N. F. VI Assay.	N. F. VI Assay (Corrected for Dissolved Solids in Solvents.	Warren's Assay Method.	Average of All the Methods.
U. S. P. X	102.6	94.0	97.7	94.6	92.5	96.3
N. F. VI	103.6	93.5	96.5	94.9	93.0	96.3

Numerous assays were carried out on the resins prepared by the U. S. P. X and N. F. VI methods in order to determine whether the change in official menstruum caused any change in the purity of the resin.

The results in Table II indicate that the U. S. P. X and N. F. VI resins of jalap are of equal purity. It is well known that in the U. S. P. X method of assay some potassium citrate contaminates the resin and is weighed as resin, thus making the results come out over 100%. All the other assay methods gave results below 100%; this would seem to indicate that some of the resin is lost during the assay. In Warren's method, the resin present in the assay sample is washed three successive times with 15-cc. portions of water. It is impracticable to make a direct determination of the amount of resin which dissolves in the wash water along with the impurities which the washing process is intended to remove. However, it was thought that by repeatedly washing the same sample of resin it should be possible to get a clearer idea regarding the possible loss of resin during the washing process. Extensive experiments were carried out on this point. A typical result, based on the average of four determinations, is as follows.

Analysis of a sample of jalap by Warren's method showed it to contain 6.3% resin. After washing the resin again with three 15-cc. portions of water the amount of resin which remained corresponded to 6.0% of the weight of the original drug. After a third, fourth, fifth, sixth and seventh washing, the weight of resin which remained corresponded to 5.8%, 5.6%, 5.5%, 5.3% and 5.2%, respectively, of the weight of the drug. In other words, about 2.5 to 3 per cent of the resin present was lost each time the assay process was repeated. Such a constant loss could hardly be ascribed to removal of impurities and is rather to be viewed as a loss of resin during the assay. Part of this loss appears to be due to a slight solubility of the resin in water.

DISCUSSION OF RESULTS.

It was found that within the limits of No. 20 and No. 80 powder, the fineness of powder has practically no effect on the rate of extraction of jalap by percolation. These results are in accord with the conclusions reached by Husa and Huyck (3) on belladonna root; it was shown that within the limits of No. 20 and No. 80 powder the fineness of powder is of minor importance in the extraction of the alkaloids of belladonna root by percolation.

In the experiment on variation in solvents, it was found that the amount of resin was practically the same for the various menstrua when the assays were made by Warren's method, while the U. S. P. X method of assay seemed to indicate slightly more efficient extraction of resin as the alcoholic content of the four menstrua decreased. It would seem that the apparently higher results in the U. S. P. X assay method may be due to the retention of other constituents of the extractive matter in the resin obtained in the assay. The results in the percolation experiments are in accord with the data obtained previously on the extraction of jalap by a maceration process (1).

The results indicate that the change from the U. S. P. X menstruum (alcohol) to the N. F. VI menstruum (alcohol 9 volumes—water 1 volume) increases the rate of extraction and yield of Resin of Jalap to only a very slight extent, if at all. On the other hand, the use of the N. F. VI menstruum causes a great increase in the total extractive matter in the percolate, thus increasing the bulk of syrupy extract which must be handled and greatly increasing the proportion of impurities to be removed during the precipitation and washing of the resin.

SUMMARY.

Percolation tests show that within the limits of No. 20 and No. 80 powder, the fineness of powder is of minor importance in the extraction of jalap.

The N. F. VI menstruum for Resin of Jalap (alcohol 9 volumes—water 1 volume) has no advantage over the menstruum of the U. S. P. X (alcohol) from the standpoint of rate of extraction, purity and yield of resin. The N. F. VI menstruum has the disadvantage of causing a great increase in total extracted matter, thus increasing the bulk of syrupy extract to be handled and greatly increasing the proportion of impurities to be removed during the precipitation and washing of the resin.

Comparative results on the assay of resin of jalap by several assay methods are presented.

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A STUDY OF PRECIPITATION IN FLUIDEXTRACT OF SENNA I.*[1]

A COMPARATIVE STUDY OF PRECIPITATION IN THE OFFICIAL FLUIDEXTRACTS OF SENNA.

BY KARL L. KAUFMAN[2] AND C. O. LEE.[3]

INTRODUCTION.

Most fluidextracts may be filtered, or decanted, and bottled for the trade within a rather short period of time after they have been made. A few of them, however, continue to sediment over long periods of time, yielding unsightly preparations. Among them is the official fluidextract of senna.

This investigation has been made with the view to finding ways of preventing, or hastening, the completion of sedimentation in fluidextract of senna. A study of the merits of various menstruums has been made together with other factors bearing upon the subject.

HISTORICAL.

Fluidextract of senna was first official in the U. S. Pharmacopœia of 1860 and has been retained in each revision since then. In spite of its unsightly appearance, it has remained official because of its wide use as a valuable medicinal agent.

Soon after their introduction into the Pharmacopœia, fluidextracts became the objects of criticism and study. From the point of view of elegant preparations, many of them are unsatisfactory, even to-day, because of their continued precipitation.

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[1] Based upon a thesis submitted to the Faculty of Purdue University by Karl L. Kaufman in partial fulfilment of the requirements for the Degree of Doctor of Philosophy.

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