wherein R-alkyl radical may be an allyl, β-brom-allyl, phenyl, cyclopentyl, a primary, or secondary alkyl radical with 2 to 6 C-atoms; and R′, a methallyl radical (2-methyl-allyl). Some of these compounds have been prepared by Tabern and Volwiler (4).

Albino rats weighing 77 to 128 Gm. (average, 98 Gm.) were used in this study. Solutions of the sodium salts of the compounds were injected intraperitoneally. The M. A. D., duration of action and the M. L. D. were determined by using 5 animals for each dose level.

As shown in Table I, these barbituric acid derivatives have a distinctly shorter duration. Thus, the substitution of a methallyl (2-methyl-allyl) radical in barbituric acid derivatives, similar to the nitrogen alkyl barbituric acid compounds, produces a change (shorter) in the duration of action. Compounds numbered 19, 20, 21 and 22 (Table I), derivatives of methallyl thiobarbituric acid, cause convulsions with little or no hypnotic or anesthetic properties. There is evidence (5) that certain thiobarbituric acid compounds show pathological changes, and when administered by vein in man (6), thrombosis and soreness of the arm may result.

CONCLUSIONS.

1. A number of methallyl (2-methyl-allyl) barbituric acid derivatives have been synthesized and studied pharmacologically.

2. The substitution of a methallyl radical in barbituric acid derivatives distinctly shortens the duration of action.

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DRUG EXTRACTION. XIII. THE EXTRACTION OF IPOMEA.*[1]

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

The U. S. P. X specified alcohol as the menstruum for the preparation of resin of ipomea. The N. F. VI changed the menstruum to a mixture of 9 volumes of alcohol and 1 volume of water. In the present paper a report is made of experiments which were carried out to determine the relative value of the U. S. P. X and N. F. VI menstrua from the standpoint of rate of extraction, purity and yield of resin.

EXPERIMENTAL DATA.

Effect of Variation in Solvents on Rate of Extraction.—Percolation experiments were conducted using ipomea, in moderately coarse powder, containing 21.1 per cent of resin by the N. F.

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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. [2] Head Professor of Pharmacy, University of Florida.

VI assay. Two 500-Gm. portions of ipomea were extracted with alcohol, using 300 cc. of moistening liquid and with a maceration period of 48 hours after packing. Two other percolations were carried out in the same manner with the exception that the menstruum was a mixture of alcohol 9 volumes—water 1 volume. Percolation was conducted at the rate of about 0.5 cc. per minute, the percolates being collected in successive fractions of 250 cc., 250 cc., 500 cc. and 500 cc. The various fractions of percolate were assayed for resin by the N. F. VI assay and for total extractive. The results are shown in Table I, the figures reported being the averages of duplicate experiments.

TABLE I.—PERCOLATION OF IPOMEA WITH DIFFERENT MENSTRUA.

	Gm. of Resin in Various Fractions of Percolate.		Gm. of Total Extractive in Various Fractions of Percolate.	
Percolates.	Alcohol.	Alc. 9 Vol.—Water 1 Vol.	Alcohol.	Alc. 9 Vol.—Water 1 Vol.
250 cc.	60.5	63.7	65.3	70.3
250 cc.	33.1	32.9	35.6	38.1
500 cc.	7.8	6.9	10.3	14.3
500 cc.	1.1	1.5	2.6	6.3
Total	102.5	105.0	113.8	129.0

The results in Table I indicate that the rate of extraction of resin from ipomea is practically the same with alcohol as with a mixture of 9 volumes of alcohol and 1 volume of water. From 500 Gm. of drug, the more aqueous menstruum removed 2.5 Gm. more resin and 15.2 Gm. more total extractive.

Comparison of U. S. P. X and N. F. VI Methods of Preparing Resin of Ipomea.—Experiments were carried out to determine whether or not the change in official menstruum has any effect on the purity or yield of resin of ipomea. Two different lots of resin of ipomea were prepared from 500-Gm. portions of ipomea in moderately coarse powder, using alcohol as the menstruum; each 500-Gm. portion of drug was moistened with 300 cc. of menstruum before packing and there was a maceration period of 48 hours after packing. Percolation was carried out at an average rate of about 0.6 cc. per minute, 1500 cc. of percolate being collected from each 500-Gm. portion of drug. Each 1500-cc. portion of percolate was evaporated on a water-bath to a thin syrup, which was poured in a thin stream, with constant stirring, into 700 cc. of hot distilled water. The precipitated resin was allowed to settle, the supernatant liquid was decanted and the precipitate washed twice, by decantation, with 700-cc. portions of hot distilled water. Each lot of resin was dried to constant weight in a vacuum oven at 75–80° C.

Two portions of resin of ipomea were also prepared from 500-Gm. portions of ipomea, 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 average yield of resin of ipomea obtained from the two 500-Gm. portions of ipomea by the U. S. P. X method was 19.5%, while by the N. F. VI method the yield was 20.3%.

The various lots of resin were assayed by the N. F. VI assay method which is specified for determining the percentage of resin in ipomea. Taking the average of several determinations, the resin prepared by the U. S. P. X method was found to assay 98.2% resin while the resin prepared by the N. F. VI method assayed 96.7%.

DISCUSSION OF RESULTS.

It was found that the rate of extraction of resin of ipomea is practically the same with the U. S. P. X menstruum (alcohol) as with the N. F. VI menstruum (alcohol 9 volumes—water 1 volume). The N. F. VI menstruum gives a slightly higher yield of resin, but the product is not as pure as that obtained by the U. S. P. X method. On the basis of the purity of the product, it is concluded that the U. S. P. X menstruum is preferable to the N. F. VI menstruum.

The results of the present study are in accord with the results obtained by Husa and Fehder (1) in a similar investigation of the preparation of resin of jalap in which it was found that the more aqueous menstruum had 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.

SUMMARY.

The N. F. VI menstruum for Resin of Ipomea (alcohol 9 volumes—water 1 volume) gives a slightly higher yield than that obtained with the U. S. P. X menstruum (alcohol). However, the N. F. VI product is less pure than that obtained by the U. S. P. X method. On the basis of the purity of the product, it is concluded that the U. S. P. X menstruum is preferable to the N. F. VI menstruum.

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METHODS USED IN THE DETERMINATION OF THE ALKALINITY IMPARTED TO WATER BY AMPUL GLASS.

BY R. K. SNYDER AND E. N. GATHERCOAL.*,[1]

INTRODUCTION.

Chemists, perhaps, first used hermetically sealed glass containers for the preservation of materials sensitive to air and moisture.

Needham, 1759, and Spallanzani, 1769, mention the preservation of sterile putrescible liquids in flasks hermetically sealed by drawing out the necks of the flasks.

Limousin, inventor of the cachet, was the first to propose (1886) the method of dispensing sterile fluids in ampuls. French pharmacists were the first to offer injectable medicaments in such containers. Little change has been made during the subsequent years in the shape and appearance of ampuls.

The fact was noted rather early in the use of ampuls that certain solutions, notably the alkaloidal solutions, were visibly affected by the alkalinity produced by the glass during the sterilization and aging of the ampul solution.

AMPUL GLASSES.

The glass first used in ampuls was of the usual soda-lime type containing about 70% of SiO_2, 14% of Na_2O and 13% of CaO. Rather early in the present century glass was produced containing boric oxide. This Jena glass was found to be much more resistant to corrosion by reagents than was the ordinary soda-lime glass. Jena glass contained about 4% of B_2O_3, but in 1911 a new Jena glass was produced containing about 65% of SiO_2, 11% of B_2O_3, 4% of Al_2O_3, 11% of ZnO and 7.5% of Na_2O. Jena glass is harder to fuse than soda-lime glass and yet it can very nicely be used for ampuls, and has been widely introduced for this purpose.

A few years later, Pyrex glass was introduced for laboratory use. This glass contains about 80% of SiO_2, 12% of B_2O_3, 2% of Al_2O_3 and 4% of Na_2O. This glass is difficult to fuse, due to its low alkalinity and high content of silica. It is of high resistance to corrosion due to

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