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## DRUG EXTRACTION. V. THE EXTRACTION OF BELLADONNA ROOT WITH GLYCERINIC MENSTRUA.*[1]

BY WILLIAM J. HUSA[2] AND LOUIS MAGID.

Tests were carried out to determine the efficiency of various glycerin-alcohol-water mixtures in the extraction of belladonna root.

### EXPERIMENTAL PART.

100-Gm. portions of belladonna root in No. 40 powder were percolated by Type Process B with four different menstrua, *i. e.*, (A) alcohol 5 vol.—water 1 vol., (B) alcohol 425 vol.—water 100 vol.—glycerin 75 vol., (C) alcohol 325 vol.—water 200 vol.—glycerin 75 vol., and (D) alcohol 200 vol.—water 100 vol.—glycerin 100 vol. The preceding menstrua constituted Menstruum I, 100 cc. being used; a mixture of alcohol 5 vol.—water 1 vol. was used as Menstruum II. In each case the drug was moistened with 60 cc. of Menstruum I, and allowed to macerate for 6 hours before packing. The drug moistened with Menstruum A occupied 250 cc. after packing, while with Menstrua B, C and D the volume after packing was 265 cc. After packing, maceration was allowed to proceed for 24 hours. The percolates were then collected in three fractions, 80 cc., 100 cc. and 300 cc., respectively. The first 80 cc. was collected at the rate of 10 drops per minute, and the remainder of the percolate at 20 drops per minute.

TABLE I.—EFFECT OF VARIOUS GLYCERINIC MENSTRUA ON THE EXTRACTION OF POWDERED BELLADONNA ROOT.

| | Grams of Alkaloid in Various Fractions of Percolate. Menstruum. | | | |
|---|---|---|---|---|
| Percolate. | A. | B. | C. | D. |
| 80 cc. | 0.429 | 0.420 | 0.372 | 0.337 |
| 100 cc. | 0.052 | 0.066 | 0.088 | 0.092 |
| 300 cc. | 0.000 | 0.000 | 0.018 | 0.042 |
| Totals | 0.481 | 0.486 | 0.478 | 0.471 |

### DISCUSSION OF RESULTS.

The results in Table I show that glycerin retards the extraction of the alkaloids of belladonna root. The retardation increases with increasing concentration of glycerin and with decreasing concentration of alcohol. The results of the tests with glycerinic menstrua uphold the general opinion that glycerin does not aid in the extraction of alkaloidal drugs. Firlas (1) has stated that the presence of glycerin in the menstruum does not affect the amount of alkaloids in fluidextracts of cinchona, hydrastis and ergot. Scoville (2) showed that glycerin in the men-

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struum retarded extraction in yellow cinchona and hydrastis, although it aided in the extraction of red cinchona.

In the present experiments, the passage of the glycerinic Menstrua I through the drug could be observed due to the fact that the color became darker than that of Menstruum II. The glycerinic Menstruum B was apparently displaced entirely into the first percolate, while C was displaced into the second percolate, and D was not entirely displaced until the third percolate was being collected. It appears that the retardation of the extraction of the alkaloids of belladonna root with the use of glycerinic menstrua may be related to the difficulty of displacement by the non-glycerinic menstruum which follows.

SUMMARY.

Percolation experiments with glycerinic menstrua indicate that glycerin retards the extraction of alkaloids from belladonna root. The retardation is greater with increasing concentration of glycerin and with decreasing concentration of alcohol.

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## ASSAY FOR PHENOL IN OFFICIAL PREPARATIONS.*[1]

BY GLENN L. JENKINS AND MELVIN F. W. DUNKER.[2]

Numerous colorimetric and volumetric methods have been proposed for the determination of phenols in general (1, 2, 3, 4). The most widely accepted procedure is the well-known iodometric method used in the official assay (5) of phenol and liquefied phenol. Redman and Rhodes (6) have shown that the period of shaking after the addition of the Koppeschaar's Solution and acid, and also after the addition of the potassium iodide may be reduced to one minute if continuous shaking is employed. Olivier (7) had previously pointed out that the time period could be reduced to five minutes. Corfield and Mundy (8) stated that the iodometric method is readily applicable to the assay of phenol containing preparations such as lotions and gargles which do not contain ingredients that react with free bromine. Bauer (9) reported that the official method of assay for phenol could be applied to the assay of Aqua Phenolata N. F. V with accurate results. The assay of preparations containing ingredients that react with bromine necessitates the quantitative separation of the phenol either by distillation or extraction. Corfield and Mundy (8) and Smelt (10) recommend extraction with warm dilute sodium hydroxide solution or preferably distillation for preparations such as phenol ointment, gargle of potassium chlorate with phenol, glycerite of phenol, lozenges and suppositories.

The procedure employed for the estimation of phenol in the study here reported was as follows:

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