(1)	Calcium chloride	0.00082425
(2)	Inosite hexacalcium gluconate	0.00120096
(3)	Calcium diphosphate	0.00129375
(4)	Calcium glycerophosphate	0.00161150
(5)	Calcium lactate	0.00163635
(6)	Calcium gluconate	0.00336241

The foregoing quantities represent equivalent amounts of calcium.

Attention is called to the fact that this preliminary study leaves relatively wide gaps between the various quantities of magnesium sulphate used to neutralize the absorbed calcium (gaps between 1.0 and 1.25, 1.25 and 1.50, 1.50 and 2.00); and also that but one time interval (2 hours) was involved. Consequently further studies are underway in which the effects of longer time intervals between the doses of the calcium compounds and the doses of the magnesium sulphate are being recorded, as well as the effects of increases of 0.1 mg. of magnesium sulphate per Gm. of body weight of mouse between the values (1.0 and 1.25, 1.25 and 1.50, 1.50 and 2.00) which appear in the present tables.

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THE SOLUBILITY OF CALCIUM LEVULINATE IN WATER.

BY GERALD J. COX,[1] MARY L. DODDS[2] AND CLARENCE CLASPER.[3]

Calcium levulinate has been shown to be more satisfactory for parenteral administration of calcium than any other known salt (1, 2). Anticipating the prob-

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able early adoption of this compound in therapeutics, we have determined the solubility of calcium levulinate in water over a range of temperature as an aid in the technology of both the preparation of solutions and the purification of the salt by crystallization.

The calcium levulinate samples for this study were purified by repeated crystallization from water and alcohol mixtures and dried in air at room temperature. The criteria of purity were the absence of color in concentrated solutions and the calcium content. The latter was determined by permanganate titration of the precipitated calcium oxalate from nine samples and found to average 13.33% calcium compared with the theoretical value of 13.09% from $Ca(C_5H_7O_3)_2.2H_2O$.

Saturated solutions of calcium levulinate in distilled water were prepared by immersion in appropriate baths at seven different temperatures of (1) water and excess calcium levulinate, and (2) solutions of calcium levulinate saturated at higher temperatures. After about six hours, samples were withdrawn isothermically and weighed. Calcium was determined on aliquots of the samples by permanganate titration of the precipitated oxalate. The temperature of saturation was determined by means of short stem immersion thermometers calibrated by the Bureau of Standards.

The means of at least four determinations at each temperature were used in the calculation of an equation which shows the composition of a saturated solution at temperatures from 0° to 55.4° C. This equation is

$$p = 27.58 + 0.173t + 0.0031t^2$$

in which "p" is the number of grams of calcium levulinate, $Ca(C_5H_7O_3)_2.2H_2O$ per hundred grams of solution and "t" is temperature in centigrade degrees.

Comparison of the observed solubilities with those calculated from the equation are shown in Table I.

TABLE I.

Temp., °C.	Mean p., Observed.	p., Calc'd.
0	27.6	27.58
15.8	31.1	31.08
25.0	34.0	33.81
30.0	35.5	35.56
37.0	38.7	38.32
45.3	41.4	41.48
55.4	47.0	46.67

For "p" = 100, "t" is found to be 127.4° C. The temperature, 127.4° C. therefore is the calculated point at which calcium levulinate dissolves in its water of crystallization. Melting points for the material used were found to range from 108° to 125° C. depending on the rate of heating. The latter melting point was obtained by immersion of open capillary melting point tubes in a bath at 125° C. The time rate of melting was approximated to those of benzoic acid in a bath at 122° C. and of urea at 132° C. As the observed melting point is in fair agreement with the calculated, the equation can be applied safely for temperatures above 55.4° C., the maximum used in this study.

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PHYTOCHEMICAL NOTES.*[1]

No. 110. DIGITONIN AND PHYTOSTEROL FROM THE SEED OF DIGITALIS PURPUREA.

BY OLE GISVOLD.

A kilo of seed, not comminuted, was extracted with a liter of alcohol in a continuous extraction apparatus for 8 hours and the extract tested for digitonin with cholesterol. The extraction was repeated with a like amount of solvent for 10 hours and a third time for 12 hours. The third extract gave but a slight precipitate with cholesterol.

The same process was repeated with comminuted seed. So far as the digitonin was concerned, the results appeared to be about the same. However, the comminuted seed gave up more of its fatty oil. These results might have been anticipated, since the digitonin is located in the seed coating, whereas the fatty oil is located in the cotyledons.

The balance of the available seed, 25 pounds, all of which had been raised in the Garden of the Pharmaceutical Experiment Station under the direction of Prof. W. O. Richtmann, was ground and extracted in the Lloyd extractor.

The first alcoholic concentrates removed had separated a considerable amount of fat with which was mixed a solid substance that was removed by force filter. Hence the alcoholic concentrate yielded two products, *viz.:*

1. The solid that remained in the filter and which was shown to be a digitonide, and digitonin.

2. The filtrate which consisted of the alcoholic extract plus dissolved and separated fat.

1. Purification and identification of the digitonide. The greenish material which had remained in the filter was several times suspended in hot petroleum ether to remove fat which was added to the other petroleum-ether extracts. After that it was suspended several times in hot alcohol to remove any alcohol-soluble material. The substance thus purified was designated "A;" the combined alcoholic filtrates were designated "B."

(A) The purified and pulverized solid weighed 18.5 Gm. It was suspected to be digitonide, since the digitonin from the seed coats and the sterol from the cotyledons would naturally be brought together in the alcoholic extract. In so far as the two dissolved substances met within the tissue of the seed, the insoluble digitonide resulting should be found in the extracted marc. In so far as the two substances reacted in the percolate, they would precipitate each other.

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[1] Scientific Section, A. PH. A., Madison meeting, 1933.