readily obtained. A typical procedure is as follows, the quantities given yielding a solution containing about 12 Gm. Bi_2O_3 in 100 cc.:

Bismuth Subcarbonate	15 Gm.
Water	25 cc.
Conc. nitric acid	25 cc.

Heat, but do not boil, until solution is complete and all carbon dioxide is expelled. Dilute to about 600 cc. but not to the precipitation point. Add ammonia water in slight excess. Use litmus paper and have the mixture alkaline throughout. Collect the precipitate on a Büchner filter by suction, washing well to remove soluble salts.

Dissolve 10 Gm. Rochelle salt in water to make about 25 cc. When the Rochelle salt is dissolved add 20 cc. of glycerin and heat to about 100° C. Do not boil.

To the hot mixture add the bismuth precipitate and mix well. To this mixture add a 50% solution of sodium hydroxide, a few drops at a time, stirring continuously until solution is complete; to this solution add a pasty mixture of tartaric acid and water, a little at a time, until it just turns blue litmus red. No precipitation should occur at this point, but if it does—a drop or two of ammonia will usually clear up the precipitate. Add water to bring the volume up to 100 cc.

The above has been varied by increasing or decreasing the glycerin, substituting sodium tartrate or potassium tartrate for the Rochelle salt, potassium hydroxide for the sodium hydroxide, etc. It has been found that within a quite liberal range all these factors may be varied and solutions obtained that seem to be permanent.

Experiments are under way to determine whether or not a solid bismuth compound that is permanent and soluble in water can be separated from such solutions.

DRUG EXTRACTION. VI. DETERMINATION OF THE PRESSURE EXERTED BY A DRUG DURING PERCOLATION.*[1]

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

Previous experiments (1) seem to indicate that maximum swelling of powdered drugs is not attained with the proportions of liquid ordinarily used in moistening drug powders preparatory to packing in a percolator. From this it would follow that a certain amount of pressure would be developed in the percolator after the drug has been packed and excess menstruum added. This pressure might affect the imbibition of the menstruum, the solvent power of the menstruum, or result in a slowing or even complete stopping of the process of percolation. Because of the fundamental importance of this question, an apparatus and a method have been developed for the determination of the pressure exerted by a drug during percolation. Using this new apparatus, measurements have been made on powdered belladonna root, rhubarb, senna and red cinchona.

APPARATUS FOR DETERMINATION OF PRESSURE.

An apparatus (Fig. 1) has been developed, whereby the pressure exerted by a powdered drug during percolation can be measured. A rubber tube, closed at one end and connected to

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

a glass tube at the other end, is placed in a percolator in such a manner that the powdered drug may be packed entirely around it. The rubber tube is filled with water as is also the connecting glass tube down to a point exactly opposite the bottom of the rubber tube. Mercury then fills the remainder of the glass tube, bent in the form of a U, until the mercury in the two arms is at the same level. The powdered drug is packed firmly around the rubber tube, using moderate pressure, and taking care not to displace the levels of the mercury arms. As the percolation proceeds, the volume of the rubber tube is kept constant by maintaining the level of the left mercury arm. As more menstruum is added, any pressure caused by further swelling of the drug tends to force the mercury in the left arm downward, but this force is neutralized by the addition of mercury to the right arm by means of an extension filled with mercury and connected to the bottom of the U-tube. By measuring the height of mercury in the right arm from the point designated as zero, the increase or decrease in pressure exerted by the drug can be determined. The volume of the drug in the percolator is kept constant by means of a perforated brass plate, which is placed on the surface of the powdered drug and kept in place by means of rods clamped in a fixed position.

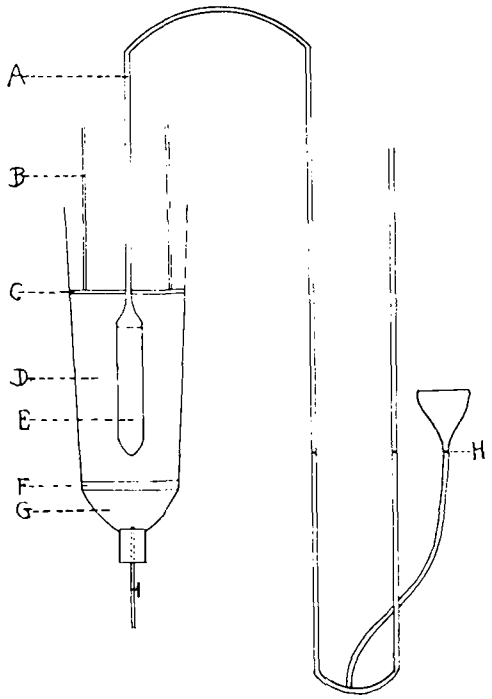

Fig. 1.—Apparatus for determination of pressure in percolator. A—Glass Tube; B—Iron Rod; C—Perforated Brass Plate; D—Drug; E—Rubber Tube; F—Sand; G—Cotton; H—Level of Mercury.

The authors are indebted to Dr. R. C. Williamson, Head of the Department of Physics, for helpful suggestions in connection with the development of the apparatus.

EXPERIMENTAL DATA.

Pressure in Percolator, Rate of Free Flow and Percentage Swelling of Powdered Belladonna Root Moistened with Varying Proportions of Water.—Four 100-Gm. portions of powdered belladonna root (moisture content of 9.60%) were moistened with 0, 25, 50 and 100 cc., respectively, of water. No time was allowed for maceration of the moistened powders, each portion being packed in a percolator fitted with the apparatus for determination of pressure, and percolation started within fifteen minutes after moistening, using water as the menstruum.

The percolators used being graduated, it was possible to ascertain the volume of the packed drugs by correcting the apparent volume for the volume occupied by the apparatus for pressure determination. Data were thus obtained as to the swelling of belladonna root in No. 40 powder when moistened with varying quantities of water.

TABLE I.—SWELLING OF BELLADONNA ROOT IN NO. 40 POWDER WITH VARIOUS AMOUNTS OF MOISTENING LIQUID.

Cc. of Water Used for Moistening.	Volume of Powder.	Percentage Swelling.
Dry	188 cc.	..
25 cc.	211 cc.	12
50 cc.	225 cc.	20
100 cc.	245 cc.	30

The results in Table I indicate that the percentage swelling increases as the proportion of moistening liquid is increased.

The lower orifice of the percolator was left open so that the volume of percolate collected after various time intervals gives a measure of the rate of free flow. In Table II, data are given as to the rate of flow and pressure exerted in the four percolators.

TABLE II.—PRESSURE DEVELOPED AND RATE OF FREE FLOW FOR BELLADONNA ROOT MOISTENED WITH VARYING QUANTITIES OF LIQUID, USING WATER AS THE MENSTRUUM.

Time in Hours.	Pressure in Mm. of Mercury.				Number of Cc. of Percolate Collected.			
	0.*	25.*	50.*	100.*	0.*	25.*	50.*	100.*
1	64	9	13	10	0	0	0	25
7	69	19	26	19	0	0	15	205
10	69	30	29	17	0	10	40	300
14	69	34	34	15	0	31	92	425
24	69	42	31	11	0	84	200	665
38	99	60	29	7	5	151	328	765
48	108	59	29	6	18	167	360	796
72	123	46	21	4	34	192	420	813
96	119	39	..	1	49	219	...	835
120	83	35	..	..	53	239	...	...
144	77	29	..	..	57	254	...	...

* Number of cc. of moistening liquid used for 100 Gm. of drug.

The results in Table II indicate that as the proportion of water used for moistening increases, the pressure exerted decreases. In each percolator the pressure rises to a maximum and then decreases. The rate of free flow increases rapidly with increasing amounts of moistening liquid. The menstruum had penetrated completely down through the column of packed drug after 26 hours when the powder was packed dry, after 7 hours when the powder was moistened with 25 cc. of water before packing, after 3 hours when 50 cc. of moistening liquid was used and in 25 minutes when 100 cc. of moistening liquid was used.

The decrease in pressure during the later stages of percolation is probably due to the extraction of soluble constituents, thus decreasing the quantity of solid material present. In the case of belladonna root, the decrease in pressure does not bring about an increased rate of flow, but conversely there is a decreased rate of free flow in the later stages, which occurs later than the decrease in pressure. Apparently some factor comes into play to reduce the rate of flow; possibly this is due to swelling of starch grains or other material in such a manner as to decrease the permeability of the cells, the swelling perhaps taking place in such a way as to fill the open spaces in the drug without increasing the total volume (and pressure).

Pressure in Percolator, Rate of Free Flow and Percentage Swelling of Drugs in Hydro-Alcoholic Menstrua.—Data from experiments on various drugs and menstrua are given in Table III.

Time Factor in Imbibition.—An experiment was performed, using the centrifuge method, to determine the amount of imbibition of water by belladonna root in No. 40 powder with 10 minutes, 24 hours and 48 hours of maceration. The pur-

pose of the experiment was to discover whether appreciable additional imbibition took place between 24 and 48 hours, since data on this point had not previously been obtained. The results of the experiments are given in Table IV.

TABLE III.—A. DETERMINATION OF PRESSURE IN PERCOLATOR AND RATE OF FREE FLOW USING 100-GM. PORTIONS OF BELLADONNA ROOT IN NO. 40 POWDER.

Menstruum.	Cc. of Moistening Liquid.	% Swelling.	Hours Maceration. Before Packing.	Hours Maceration. After Packing.	Hours for Flow to Commence.	Time of Readings in Hours after Flow Started.	Cc. Percolate.	Pressure in Mm. Mercury.
Water	50	36	6	0	2.08	0.50	15	+ 2
						2.33	190	+ 2
						12.33	480	− 4
						17.75	780	− 8
						21.66	1000	− 9
						0.00	0	+13
Water	50	36	6	48	2.25	4.33	50	+17
						15.41	140	+14
						52.00	300	+ 8
						72.75	330	− 2
Alc. 5 vol.— water 1 vol.	0	..	0	0	1.50	1.58	30	+ 7
						2.75	50	+ 1
						5.58	140	0
						16.83	450	+18
						30.83	950	+15
						46.00	1430	+12
Alc. 5 vol.— water 1 vol.	25	24	0	0	0.41	0.25	30	− 2
						1.92	330	− 4
						4.00	700	0
						4.58	800	+ 2
						5.58	1000	+ 5
Alc. 5 vol.— water 1 vol.	50	24	0	0	0.16	0.25	50	− 2
						1.41	350	− 3
						1.75	480	− 3
						2.75	780	− 2
						3.50	1000	− 2
Alc. 5 vol.— water 1 vol.	50	27	6	0	0.16	0.33	90	− 9
						1.08	350	−10
						1.41	500	−11
						2.33	900	−11
						2.50	1000	−11
Alc. 5 vol.— water 1 vol.	50	24	6	48	0.16	0.08	28	+ 1
						0.25	87	0
						1.25	480	0
						2.83	1000	− 1
Alcohol	0	..	0	0	0.66	0.25	10	+ 5
						3.66	120	+ 7
						15.58	470	+14
						20.08	600	+18
						40.08	1100	+20
Alcohol	50	21	0	0	0.08	0.20	90	− 5
						0.45	240	− 6
						0.86	420	− 5
						1.11	1000	− 6
B. Using 100 Gm. Rhubarb in No. 40 Powder.								
Alc. 4 vol.— water 1 vol.	50	16	6	48	0.10	0.13	30	+ 1
						0.33	290	0
						0.68	780	− 2
						0.83	1000	− 2
C. Using 100 Gm. Red Cinchona in No. 40 Powder.								
U. S. P. X Menstruum I and II	50	38	6	48	0.20	0.08	28	+19
						0.16	100	+15
						0.35	300	+ 9
						0.86	880	+ 6
						1.00	1000	+ 5

	D. Using 100 Gm. Alexandria Senna in No. 20 Powder.							
Alc. 1 vol.— water 2 vol.	50	18	6	48	0.10	0.05	30	+ 1
						0.10	50	0
						0.18	100	− 2
						0.66	400	−10
						1.68	1000	−18

TABLE IV.—WEIGHT IN GM. OF WATER IMBIBED BY BELLADONNA ROOT IN NO. 40 POWDER.

For 5 Gm. of Drug (by Experiment), Time of Maceration.			For 100 Gm. of Drug. (Calculated), Time of Maceration.		
10 Min.	24 Hrs.	48 Hrs.	10 Min.	24 Hrs.	48 Hrs.
11.74	13.70	14.42	234.8	274.0	288.4
12.02	13.60	14.24	240.4	272.0	284.8
(Av.) 11.88	13.65	14.33	237.6	273.0	286.6

The results indicate that imbibition increases somewhat with time.

DISCUSSION OF RESULTS.

Percolation of Belladonna Root with Water.—Moistening of 100 Gm. of powdered belladonna root with 50 cc. of water resulted in 20% swelling if no maceration were allowed and 36% if 6 hours' maceration before packing was employed. The swelling results account for the fact that the rate of free flow was greater and the pressure less in the latter case. When 48 hours' maceration after packing is used, the rate of free flow is smaller than when no maceration after packing is allowed. Using 6 hours' maceration before packing and 48 hours' maceration after packing, the pressure in the percolator is greater than when only six hours' maceration before packing is employed but less than when no maceration whatever is used.

Percolation of Powdered Belladonna Root with Hydro-Alcoholic Menstruum.—Using a menstruum of alcohol 5 vol.—water 1 vol., data were obtained on the effects of using varying proportions for moistening, and the effects of the 6-hour period of maceration before packing and the 48-hour period after packing. Allowing no time for maceration, increased amounts of moistening liquid increase the rate of free flow and decrease the pressure. Maceration for 6 hours before packing increased the rate of free flow and decreased the pressure; the pressure in the percolator was increased by maceration for 48 hours after packing. Using a menstruum of alcohol, the rate of free flow is likewise increased and the pressure decreased by use of 50 cc. of moistening liquid for 100 Gm. of drug.

Percolation of Other Drugs.—Using the official menstrua and the official processes for the preparation of the respective fluidextracts, percolation experiments were carried out using rhubarb, red cinchona and Alexandria senna. In each case the rates of free flow were very rapid and the pressures exerted decreased during the percolation. The greatest pressure was observed during the percolation of red cinchona, while the greatest decrease in pressure during percolation was observed with senna. The rates of free flow increased during the percolation, showing that as soluble constituents were removed, a more rapid passage of the menstrua was allowed.

SUMMARY.

An apparatus has been developed for determining the pressure exerted by a drug during percolation. Using this new apparatus, measurements have been made on belladonna root, rhubarb, senna and red cinchona.

In case of belladonna root the rate of free flow decreases after a time, even though the pressure in the percolator decreases; with the other drugs there is a decreased pressure accompanied by an increased rate of flow as percolation proceeds. In general, the use of increasing quantities of moistening liquid decreases the pressure and increases the rate of free flow. Maceration after packing increases the pressure in the percolator. The greatest pressure was observed during the percolation of red cinchona, while the greatest decrease in pressure during percolation was observed with senna.

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A STUDY OF *LACINARIA* SPECIES.*

BY B. V. CHRISTENSEN[1] AND G. M. HOCKING.

Lacinaria or *Laciniaria*[2] is the name generally used to-day by taxonomists for the genus formerly known as *Liatris*, although this latter name still persists on continental Europe. Under the name "Liatris," the corm of several species of the genus has been used in American medical practice for over a century, particularly by the Eclectics and Shakers.

In the present work, attention was confined chiefly to the species *L. spicata* (L.) Kuntze and *L. tenuifolia* (Nuttall) Kuntze, which grow commonly in the region of Gainesville, Florida. The former species was particularly valuable for study because it is the subterranean parts of this member of the genus which have been used most frequently as a drug.

LACINARIA SPICATA.

Lacinaria spicata has been known by at least thirty-five common English names, of which the most important are (Blue) Blazing Star, (Ohio) Devil's Bite, Rattlesnake's Master, (Spike) Gayfeather, and (Spiked) Button Snakeroot. Some of these names indicate medicinal uses of the plant, and even the scientific name, *Liatris*, is said by some to be derived from a Greek word meaning "invulnerable," referring perhaps to its supposed value in treating (snake) wounds. The species differs from all but two or three others of the same genus in preferring a thinly wooded *moist* humid soil. It is said to grow throughout eastern North America in the area bounded by the Atlantic Ocean on the east, the Gulf of Mexico in the south, Arizona and Colorado in the west, and Minnesota and Ontario on the north. It is perhaps the most widely distributed *Lacinaria* species.

The underground portions used constitute the corm with attached fibrous roots. The corm is irregularly globoid, tapering to the stem above, often laterally lobed, and measuring 1.5 to 2.5 cm. in vertical diameter and 1.5 to 3 cm. in horizontal diameter. Externally, it is pale reddish brown to brownish gray, with roughly furrowed surface; the upper portion covered with adherent fibrous leaf bases, the lower bearing numerous small fibrous roots which originate largely at the tips of the corm protrusions. Fracture tough, woody, showing dirty yellowish internally; when cut, showing waxy sheen. In younger specimens one or two, in older several, woody rings

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* Scientific Section, A. PH. A., Portland meeting, 1935.

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[2] The authors consider *Laciniaria* to be somewhat less preferable by reason that the same name is given to a molluscan genus.