SCIENTIFIC SECTION

STUDIES IN EXTRACTION AS APPLIED TO N. F. PREPARATIONS.* (PRELIMINARY REPORT ABSTRACTED.)

BY EARL GUTH AND H. A. LANGENHAN.¹

A criterion to be used for the selection of the proper menstruum for the preparation of a fluidextract or other extractive galenicals is being sought for. It was suggested that little information could be found in the literature that would be applicable to this problem, viz., that of determining which menstruum should be used for extracting a certain drug and why this particular menstruum is the desirable one. Hence a review of the literature was not undertaken. The first attempt toward possibly simplifying the problem was to tabulate the vegetable drugs used in the preparation of N. F. fluidextracts, together with their constituents, as reported in Wehmer, "Die Pflanzenstoffe," Culbreth, Materia Medica and the National Standard Dispensatory. This compilation containing 100 drugs covered 50 typewritten pages. It had been hoped that the tabulation of the constituents would enable a grouping of this large number of drugs, into alkaloidal, resinous, starchy, etc., so that investigation of a representative of each group might be started. However, with the exception of a few, the percentage content of constituents was not recorded and a grouping was not feasible. It had been suggested that one possible procedure was to extract certain drugs with various alcoholic strength menstrua and based upon the total amount of extractive or the amount of active ingredient, found in the percolate, the desirable menstruum be selected. Hence, the first experiment consisted of percolating 100 Gm. of the drug with 95 per cent, 85 per cent and 75 per cent alcoholic menstrua, respectively, collecting the percolate in fractions of 85 cc., 100 cc. and 100 cc. Then determining the specific gravity and total extractive of each fraction and comparing this with the total extractive of the drug, the latter being determined according to the U.S. P. method for "total extractive." Table I illustrates this type of experimentation. The total extractive column points toward an increase in total extractive with the decrease of alcohol in the menstruum. However, the per cent of total extractive removed by the first 85 cc. of percolate decreases noticeably with the decrease in alcoholic content of the menstrua in Jalap, Angelica and Cocillana. The last column indicates that a total of 285 cc. of percolate was not enough to remove all of the extractive. The first 85-cc. fraction of percolate was collected at the rate of 10 drops per minute and the remaining two fractions at the rate of 20 drops per minute. In each series an attempt was made to introduce similar conditions of procedure.

The second attempt consisted of applying the same procedure of extraction to drugs containing measurable constituents. Jalap, a resinous drug, and Nux Vomica, an alkaloidal drug, were used. The U. S. P. methods of assay were used for both drugs. Early in the experimentation the question arose as to whether some of the potassium citrate used in the U. S. P. method for assay of Jalap, was

 $[\]ast$ This work was aided by a grant received from the American Pharmaceutical Association Research Fund.

¹ University of Washington, College of Pharmacy, Seattle.

Drug.	Menstruum % alcohol.	Total extractive.	1st Fraction. Gm. extd. Sp. gr.		85 cc. % of total ext.	2nd Fraction, Gm. extd. Sp. gr.	action, Sp. gr.	100 cc. % of total ext.	3rd Fraction. Gm. extd. Sp. gr.		100 cc. % of total ext.	% of total extractive extracted.
Jalap No. 1	95	11.85	7.4070	0.8626	62.5	1.1090	0.8296	9.3	0.5491	0.8269	4.6	76.4
Jalap No. 2	95	11.85	8.2322	0.8751	86.9	0.9400	0.8279	8. 8.	0.4790	0.8258	4.0	81.4
Jalap No. 3	85	16.50	9.9344	0.8923	60.2	3.2445	0.8681	19.6	2.0235	0.8625	12.2	92.0
Jalap No. 4	85	16.50	9.0914	0.8911	55.1	3.1855	0.8679	19.4	2.1895	0.8649	13.2	87.9
Jalap No. 5	75	22.68	5.3281	0.9182	23.9	5.6670	0.9025	24.9	2.4300	0.8890	10.5	69.3
Jalap No. 6	75	22.68	8.9271	0.9227	39.2	5.1785	0.9018	22.8	2.8245	0.8910	12.4	74.4
Mandrake No. 1	95	9.56	6.5001	0.8659	68.0	0.9585	0.8245	10.0	0.3930	0.8202	4.1	82.0
Mandrake No. 2	95	9.56	6.8535	0.8689	71.6	0.9540	0.8253	9.9	0.4110	0.8204	4.3	85.9
Mandrake No. 3	85	13.42	8.3963	0.8905	62.6	2.2945	0.8628	17.9	2.2195	0.8741	16.5	97.0
Mandrake No. 4	85	13.42	8.1834	0.8880	61.0	2.5890	0.8651	19.2	2.8330	0.8673	21.1	101.0
Mandrake No. 5	75	15.73	11.0058	0.9194	69.8	3.4390	0.8892	19.6	0.9390	0.9013	5.95	95.0
Mandrake No. 6	75	15.73	11.7013	0.9223	75.0	3.4325	0.8881	19.6	0.9420	0.8776	6.0	101.6
Angelica No. 1	95	8.91	5.2891	0.8463	59.3	2.4655	0.8293	27.9	1.0925	0.8247	12.2	99.4
Angelica No. 2	95	8.91	4.9538	0.8429	55.6	2.5600	0.8295	28.9	1.2635	0.8235	14.2	98.7
Angelica No. 3	85	25.68	7.7902	0.8774	30.3	6.8745	0.8773	26.7	5:0860	0.8729	19.7	76.7
Angelica No. 4	85	25.68	7.9520	0.8773	30.9	6.8365	0.8757	26.5	5.3955	0.8738	21.0	78.4
Angelica No. 5	75	31.97	11.2413	0.9237	35.1	10.8640	0.9156	34.8	5.2445	0.8996	16.4	86.3
Angelica No. 6	75	31.97	11.1027	0.9218	34.7	10.7265	0.9172	34.4	5.2595	0.9020	16.7	85.0
Calendula No. 1	95	18.08	5.6610	0.8507	31.2	4.3040	0.8608	23.8	3.5755	0.8299	19.7	74.7
Calendula No. 2	85	27.57	6.5084	0.8754	23.6	5.9465	0.8746	21.5	3.6710	0.8668	13.8	68.9
Calendula No. 3	75	29.85	9.2833	0.9171	33.8	6.7630	0.9070	22.6	4.1235	0.8883	13.7	70.1
Cocillana No. 1	95	3.61	1.8798	0.8364	52.0	0.6350	0.8213	17.5	0.2785	0.8204	7.7	77.0
Cocillana No. 2	85	4.15	2.2686	0.8608	54.6	1.1275	0.8534	27.1	0.5350	0.8514	12.8	94.5
Cocillana No. 3	75	6.54	2.7548		42.1	1.0550	•	16.1	0.5415	:	8.2	66.4

included in the final residue representing the resin. The residues upon ignition yielded an ash which was alkaline to phenolphthalein. This suggested the presence of water-soluble potassium citrate. The U.S. P. method was modified to the extent of washing the dried and weighed residues with hot water, then drying and again The latter showed a loss in weight. (See Table II.) It must be reweighing. membered that the resin is a complex substance and that water-soluble constituents may have been removed by the alcohol of the alcohol-chloroform mixture during the assay, also that the heat during the drying of the residue may have introduced a change of solubility. However, it is safe to assume that some potassium citrate is carried over and computed as resin. As a check the Jalap was also assayed according to the B. P. method, and the liquid extracts assayed according to the B. P., U. S. P. and U. S. P. modified, methods; the modification consisting of the washing just mentioned. Table No. 3 presents the results of this experiment. Assuming that 285 cc. of percolate did not represent complete extraction (as is indicated in Table No. 1.) the results obtained from the assay of the Jalap percolates offer an interesting condition in that the amount of resin found in the 75 p. c. alcoholic extraction tends to be greater than that found in the original drug. That is it would appear as if the 75 per cent alcoholic menstruum removes more 95 per cent alcoholsoluble constituents than does the 95 per cent alcoholic menstruum.

Table IV illustrates the extraction experiment applied to Nux Vomica. Because of lack of time the drug itself was not assayed.

	,	
% resin U. S. P. method.	% resin after washing.	% lost.
7.26	6.52	0.72
7.45	6.62	0.83
7.37	6.69	0.68
7.47	6.70	0.77
7.22	6.64	0.58
7.16	6.36	0.80
7.54	6.33	1.22
7.21	6.51	0.71
Average 7.33	6.54	0.79
13.53	11.45	2.08
12.40	11.63	0.97
Average 12.96	11.54	1.42
	U. S. P. method. 7.26 7.45 7.37 7.47 7.22 7.16 7.54 7.21 Average 7.33 13.53 12.40	U. S. P. method.after washing. 7.26 6.52 7.45 6.62 7.37 6.69 7.47 6.70 7.22 6.64 7.16 6.36 7.54 6.33 7.21 6.51 Average 7.33 6.54 11.45 12.40 11.63

TABLE II.---ASSAY OF JALAP.

TABLE III.—ASSAY OF JALAP PERCOLATES.

	U. \$	6. P.	U. S. 1	P. Mod.	:	B. P.
	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2
95% alcohol.	р. с.	p. c.	p. c.	p. c.	р. с.	р. с.
85 cc.	11.52	11.68	10.95	10.96	11.13	11.29
100 cc.	0.74	0.67	0.46	0.46	0.55	0.33
100 cc.	0.16	0.14	0.01	0.03	0.08	0.07
Total	12.42	12.49	11.43	11.45	11.76	11.79
85% alcohol.						
85 cc.	10.58	11.57	9.58	10.47	10.45	10.96
100 cc.	0.79	1.99	0.44	0.58	0.46	1.65
100 cc.	0.29	0.32	0.03	0.03	0.05	0.04
Total	11.66	14.12	10.05	11.08	10.96	12.65

Aug. 1931

75% alcohol.						
85 cc.	12.46	11.72	11.08	10.38	11.31	11.26
100 cc.	1.04	1.24	0.18	0.83	0.80	0.80
100 cc.	0.42	0.22	0.03	0.03	0.04	0.03
Total	13.92	13.18	11.29	11.24	12.15	12.09
Assay of drug:						
U. S. P. Me	thod		12.96	% Resin		
U. S. P. Me	thod mod.		11.54°	% Resin		
B. P. Metho	d		11.55	% Resin		

TABLE IV.—ASSAY OF NUX VOMICA PERCOLATES.

	73%	Alcohol.	64% A	lcohol.	49%	Alcohol,
	No. 1.	No. 2.	No. 1.	No. 2.	No. 1.	No. 2.
85 cc.	1.39	1.29	1.31	1.32	1.45	1.51
100 cc.	0.46	0.45	0.38	0.34	0.63	0.55
100 cc.	0.10	0.11	0.14	0.12	0.12	0.12
Total	1.95	1.85	1.83	1.78	2.20	2.18

Attention was now given to the alcoholic strength of menstrua. Tables showing the drugs (U. S. P. & N. F.) extracted with the same alcoholic content menstruum were prepared with the hopes that such a grouping might offer some reason as to the selection of a certain menstruum for a certain drug. Unfortunately no conclusions could be drawn from these compilations.

The menstrua prescribed by the N. F. V for the manufacture of fluidextract, represent 13 different alcoholic concentrations. Some of these vary only by 2 to 3 per cent of alcohol. Scoville (N. F. Revision Bulletin) suggested using a fewer number of concentrations and offered 9 concentrations. Table V represents the N. F. menstrua. The alcohol content is computed based on 95 per cent alcohol, making no allowance for shrinkage. Table VI represents the concentrations suggested by Scoville. The percentage of alcohol was computed based on 95 per cent alcohol and also determined, and an attempt made to account for the difference between the computed and determined by measuring the shrinkage. As the units of volumes used in the shrinkage experiment were small, these results may be considered as approximate.

 TABLE V.—Approximate Percentage of Alcohol in the Menstrua Now Used in the N. F.

 for the Manufacture of Fluidextracts.

Alcohol.		Water.	% of alcohol in menstrua.	Difference in alcoholic, %.
1 Vol.	to	0 Vol.	95	00
5 Vol.	to	1 Vol.	80	15
4 Vol.	to	1 Vol.	76	4
3 Vol.	to	1 Vol.	73	3
2 Vol.	to	1 Vol.	64	9
5 Vol.	to	3 Vol.	60	4
3 Vol.	to	2 Vol.	58	2
1 Vol.	to	1 Vol.	49	9
3 Vol.	to	4 Vol.	41	8
2 Vol.	to	3 Vol.	39	2
1 Vol.	to	2 Vol.	33	6
1 Vol.	to	3 Vol.	24	9
0 Vol.	to	1 Vol.	00	16

The following table shows the actual percentage of alcohol in each of the menstrua as suggested by Scoville. TABLE VI.

Alcohol.		Water.		Sp. gr.	% alcohol corrected for temp.	Scoville's table of percentage.	Computed percentage.
1 Vol.	to	0 Vol.		0.8076 (21° C.)	95.9	95	95.0
9 Vol.	to	1 Vol.		0.8354 (23° C.)	86.5	86	85.5
4 Vol.	to	1 Vol.		0.8611 (23° C.)	78.8	77	76.0
3 Vol.	to	1 Vol.		0.8741 (23° C.)	73.6	73	71.2
2 Vol.	to	1 Vol.		0.8938 (23° C.)	65.5	64	63.3
1 Vol.	to	1 Vol.		0.9289 (23°C.)	49.2	49	47.5
1 Vol.	to	2 Vol.		0.9557 (23° C.)	32.5	33	31.7
1 Vol.	to	3 Vol.		0.9667 (23° C.)	26.1	24	23.7
1 Vol.	to	7 Vol.		0.9799 (23° C.)	13.6	12	13.6
				Shrinkage of Menstru	1a.		
	Alcohol.			Water.		Shrinkage	
	1 Vol.		to	0 Vol.		0.00%	

9 Vol.	to	1 Vol.	
4 Vol.	to	1 Vol.	2.60%
3 Vol.	to	1 Vol.	3.12%
2 Vol.	to	1 Vol.	4.00%
1 Vol.	to	1 Vol.	5.00%
1 Vol.	to	2 Vol.	3.30%
1 Vol.	to	3 Vol.	2.00%
1 Vol.	to	7 Vol.	

1 Vol.

Т me readjustment. Nine of the drugs would be affected by this change. In order to estimate the possible effect of this change, the total extractive of those of the 9 drugs on hand, was determined, using the official menstrua and the proposed menstrua nearest in alcohol content. Table VII contains the results of this experiment.

As a possible criterion the "total solids" of a complete line of N. F. fluidextracts (78 in number) was determined. For comparison the total solids of the commercial fluidextract is included in Table VII.

		TABLE VI	I.		
Drug.	N. F. Per cent alcohol.	Menstrua. Per cent extractive.	Proposed Per cent alcohol.	Menstrua. Per cent extractive.	Com'l fldexts., Gm. extractive in 100 cc.
Angelica	79	31.42	77	30.45	7.25
Apocynum	58	13.41	64	14.36	14.88
Avena Sativa	38	• • •	33		
Cataria	40		49		
Digitalis	79	27.28	77	30.3	20.40
Geranium	58	24.15	64	23.25	23.70
Humulus	61	33.81	64	30.21	
Hydrangea	58	11.86	64	11.87	6.50
Kava	58	9.45	64	9.01	4.25

SWELLING OF DRUGS.

The following experiment was conducted in an attempt to measure the swelling taking place when dried vegetable drugs are allowed to macerate in various alcoholic menstrua. Jalap, Angelica, Ipomea and Colombo were chosen as the drugs to be used.

9 Vol.

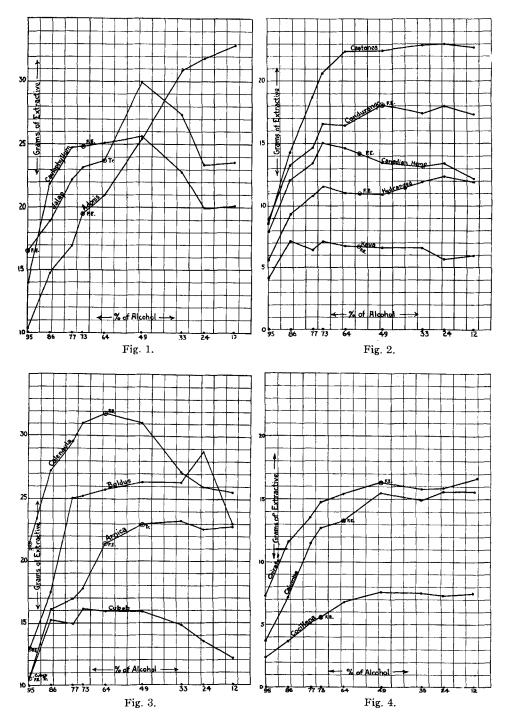
to

Per cent alcohol.	95%.	86%.	77%.	73%.	64%.
Adonis	10.20 Gm.	14.69 Gm.	16.91 Gm.	19.45 Gm.	20.95 Gm.
Arnica	10.38 Gm.	17.04 Gm.	17.91 Gm.	20.75 Gm.	21.38 Gm.
Boldus	12.88 Gm.	17.37 Gm.	25.31 Gm.	25.21 Gm.	25.75 Gm.
Castanea	8.38 Gm.	14.24 Gm.	18.65 Gm.	20.61 Gm.	22.37 Gm.
Chirata	7.30 Gm.	11.74 Gm.	13.66 Gm.	14.75 Gm.	15.43 Gm.
Columba	3.73 Gm.	7.20 Gm.	11.56 Gm.	12.74 Gm.	13.32 Gm.
Calendula	21.33 Gm.	27.18 Gm.	29.62 Gm.	31.00 Gm.	31.86 Gm.
Canadian Hemp	7.86 Gm.	12.05 Gm.	13.39 Gm.	15.08 Gm.	14.62 Gm.
Cubeb	10.42 Gm.	15.22 Gm.	14.91 Gm.	17.15 Gm.	16.90 Gm.
Caulophyllum	13.84 Gm.	21.82 Gm.	24.69 Gm.	24.82 Gm.	25.11 Gm.
Coccillana	2.40 Gm.	3.68 Gm.	5.16 Gm.	5.57 Gm.	6.81 Gm.
Condurango	8.66 Gm.	13.22 Gm.	14.65 Gm.	16.59 Gm.	16.36 Gm.
Guarana	6.09 Gm.	23.27 Gm.	25.72 Gm.	26.03 Gm.	26.28 Gm.
Hydrangea	5.52 Gm.	9.28 Gm.	10.75 Gm.	11.52 Gm.	11.07 Gm.
Jalap	16.55 Gm.	18.85 Gm.	22.17 Gm.	23.14 Gm.	23.72 Gm.
Kava	4.09 Gm.	7.21 Gm.	6.40 Gm.	7.16 Gm.	6.71 Gm.
Per cent alcohol.	49%.	33%.	24%.	12%.	
Per cent alcohol. Adonis	49%. 25.44 Gm.	33%. 30.83 Gm.	24%. 31.84 Gm.	12%. 32.92 Gm.	
				-	
Adonis	25.44 Gm.	30.83 Gm.	31.84 Gm.	32.92 Gm.	
Adonis Arnica	25.44 Gm. 23.00 Gm.	30.83 Gm. 23.23 Gm.	31.84 Gm. 22.62 Gm.	32.92 Gm. 22.88 Gm.	
Adonis Arnica Boldus	25.44 Gm. 23.00 Gm. 26.35 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm.	
Adonis Arnica Boldus Castanea	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm.	
Adonis Arnica Boldus Castanea Chirata	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 15.05 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 16.65 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba Calendula	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 15.05 Gm. 31.10 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm. 27.15 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm. 26.01 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 16.65 Gm. 25.56 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba Calendula Canadian Hemp	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 15.05 Gm. 31.10 Gm. 13.49 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm. 27.15 Gm. 13.15 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm. 26.01 Gm. 13.62 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 16.65 Gm. 25.56 Gm. 12.20 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba Calendula Canadian Hemp Cubeb	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 15.05 Gm. 31.10 Gm. 13.49 Gm. 16.89 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm. 27.15 Gm. 13.15 Gm. 14.83 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm. 26.01 Gm. 13.62 Gm. 13.53 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 16.65 Gm. 25.56 Gm. 12.20 Gm. 12.13 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba Calendula Canadian Hemp Cubeb Caulophyllum	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 15.05 Gm. 31.10 Gm. 13.49 Gm. 16.89 Gm. 25.60 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm. 27.15 Gm. 13.15 Gm. 14.83 Gm. 22.85 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm. 26.01 Gm. 13.62 Gm. 13.53 Gm. 19.97 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 16.65 Gm. 25.56 Gm. 12.20 Gm. 12.13 Gm. 20.26 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba Calendula Canadian Hemp Cubeb Caulophyllum Coccillana	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 15.05 Gm. 31.10 Gm. 13.49 Gm. 16.89 Gm. 25.60 Gm. 7.60 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm. 27.15 Gm. 13.15 Gm. 14.83 Gm. 22.85 Gm. 7.55 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm. 26.01 Gm. 13.62 Gm. 13.53 Gm. 19.97 Gm. 7.32 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 15.63 Gm. 25.56 Gm. 12.20 Gm. 12.13 Gm. 20.26 Gm. 7.52 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba Calendula Canadian Hemp Cubeb Caulophyllum Coccillana Condurango	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 15.05 Gm. 31.10 Gm. 13.49 Gm. 16.89 Gm. 25.60 Gm. 7.60 Gm. 18.10 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm. 27.15 Gm. 13.15 Gm. 14.83 Gm. 22.85 Gm. 7.55 Gm. 17.38 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm. 26.01 Gm. 13.62 Gm. 13.53 Gm. 19.97 Gm. 7.32 Gm. 17.95 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 15.63 Gm. 25.56 Gm. 12.20 Gm. 12.13 Gm. 20.26 Gm. 7.52 Gm. 17.27 Gm.	
Adonis Arnica Boldus Castanea Chirata Columba Calendula Canadian Hemp Cubeb Caulophyllum Coccillana Condurango Guarana	25.44 Gm. 23.00 Gm. 26.35 Gm. 22.45 Gm. 16.35 Gm. 31.10 Gm. 13.49 Gm. 16.89 Gm. 25.60 Gm. 7.60 Gm. 18.10 Gm. 25.72 Gm.	30.83 Gm. 23.23 Gm. 26.38 Gm. 22.91 Gm. 14.89 Gm. 15.80 Gm. 27.15 Gm. 13.15 Gm. 14.83 Gm. 22.85 Gm. 7.55 Gm. 17.38 Gm. 25.30 Gm.	31.84 Gm. 22.62 Gm. 28.55 Gm. 23.06 Gm. 15.67 Gm. 15.85 Gm. 26.01 Gm. 13.62 Gm. 13.53 Gm. 19.97 Gm. 7.32 Gm. 17.95 Gm. 24.46 Gm.	32.92 Gm. 22.88 Gm. 23.07 Gm. 22.75 Gm. 15.63 Gm. 15.63 Gm. 25.56 Gm. 12.20 Gm. 12.13 Gm. 20.26 Gm. 7.52 Gm. 17.27 Gm. 24.19 Gm.	

TABLE VIII.-GRAMS OF TOTAL EXTRACTIVE PER 100 GM. OF DRUG.

The drugs were sifted in a series of sieves until an 80 or 100 powder was obtained. A small portion of each powder was macerated in small well-stoppered vials for 30 minutes with the following menstrua: Absolute, 95%, 85% and 75% alcohol and distilled water. At the end of 30 minutes a small amount of each drug was mounted on a microscope slide in the same menstrua in which it had been macerated, covered with a cover glass and the particles measured with a micrometer ocular. 350 measurements of drug of each maceration were made and an average taken. The measurements represent the average width of the drug particles after maceration. In macerating the drugs in the closed vials only enough menstruum was added in each case to make the drug distinctly moist. The results of the experiment follow:

	Absolute ale	. 95%	Ale.	85%	Ale.	7 5%	h Alc.	Wat	er.
Jalap No. 80 powder	201.8	212.0	5.05	218.3	1.74	222.1	9.72	243.7	27.0
Angelica No. 100	160.0	164.4	2.75	168.1	0.95	169.7	19.26	202.4	26.5
Ipomea No. 80	172.7	168.8	2.31	175.9	6.08	186.6	10.39	206.1	19.3
Colombo No. 80	255.5	258.5	1.17	270.8	1.08	267.9	17.29	314.2	22.9



The Jalap used contained 8.51 p. c "moisture" as determined by the U. S. P. method. A quantity was dried at 105° C. for 24 hours and then treated with the menstrua as above mentioned. Two different samples of this dried lot were used,

195.25

Average

Absolute. 95%. 85%. 75%. Water. Jalap No. 80 powder, (1) 201.6 202.0210.5216.8238.3Oven dried (2)188.9 192.0202.5209.9242.6

and only counts of 100 each were made in place of 350 as before. The results of the air-dried drug measurements are as follows:

Graphs were made representing the increase in the size of the particles.	How-
ever, these failed to offer any additional information The results obtained	so far
would tend to indicate that this method of measurement is unsatisfactory.	

197.4

206.5

313.35

TOTAL EXTRACTIVE EXPERIMENT.

Fifteen vegetable drugs were selected at random for the determination of the total extractive, applying the U.S. P. method and using the nine proposed menstrua already mentioned. The results are presented in tabulated form as well as in graphs. (See Table VIII and charts Nos. 1, 2, 3, 4, 5.) An attempt was made to introduce like laboratory conditions so that the results would be comparable. It must be remembered that time did not permit the checking of these results. Also that the total extractive of vegetable drugs may be influenced by climatic conditions during growth. It was assumed that all samples were dry, *i. e.*, contained the same amount of moisture. Inasmuch as a difference of 2 or 3 p. c. of total extractive is mainly of experimental interest, a difference of 10 p. c. in moisture content would be of similar interest.

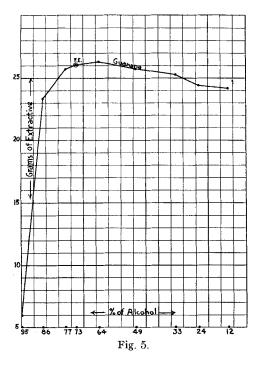
The graphs tend to indicate that the total extractive (of the drugs tested) increases as the alcohol content decreases from 95% to 49%. With concentrations below 49% the total extractive seems to decrease. Taking the average total extractive for all given, with the exception of Adonis, the high point is with the 49% concentration. Also, the 77% and the 12% concentrations represent the same levels.

The following table contains the alcohol per cent of the menstruum used giving the highest total extractive, the lowest total extractive and the alcohol per cent of the menstruum used for preparing the fluidextract and the tincture.

Drug.	High.	Low.	Flex.	Tr.
Adonis	12	95	73	
Arnica	49	95	64	49
Boldus	24	95	95	
Castanea	64 - 12	95	water	
Chirata	49	95	49	
Calumba	12	95	64	58
Cannabis	73	95	58	
Calendula	64	95	64	
Cubeb	73 - 49	95	95	95
Caulophyllum	77-49	95	73	
Coccillana	49-12	95	73	
Condurango	24	95	73	
Guarana	77–33	95	58	
Hydrangea	24	95	58	
Jalap	49	95	95	64
Kava	73	95	58	

240.45

Four of the official menstrua used for preparing fluidextracts represent the high point total extractive menstrua. It must be remembered, however, that an increase in total extractive does not necessarily mean an increase in active constituents. In the experiments already mentioned, an increase in total extractive of Jalap



gave a comparable increase in resin as measured by the U.S.P. assay method. Also the same comparable increase is noticeable in the Nux Vomica results. Much more work must be done before any suggestive interpretations may be offered. Furthermore, the amount of total extractive in the fresh percolate is not always the same as in the stabilized product. As a possible illustration the following may be of interest. A commercial fluidextract of Caulophyllum contained 18.0 Gm. total solids in 100 cc. The fluidextract is prepared with 73%alcohol. This concentration removed 24.8 Gm. of extractive. Again, a commercial fluidextract of Arnica contained 14.5 Gm. of total solids. The official menstruum is 64% alcohol representing 21 Gm. of extractive, and the tincture, made with 49% alcohol represents the highest point in the total extractive column. In contradiction to these results,

a commercial fluidextract of Guarana contained 28.8 Gm. of total solids which is more than the total solids removed by any of the nine menstrua. Also a commercial fluidextract of Adonis (5.73 Gm.) and of Jalap (9.18) falls below the lowest point of extractive obtained. The question arises as to the amount of sediment formed during the aging of the product. Also as to the therapeutic value of this sediment; with this information available selections of menstrua may be made. Until more is known of the therapeutic constituents of many of the vegetable drugs, the total extractive *remaining in solution* may be used as a criterion. This necessitates the preparation and the aging of fluidextracts prepared with the several menstrua. Such experimentation is to be undertaken.

TUNG OIL IMPORTS, JUNE 1931.

Imports of tung oil into the United States in June 1931, amounted to 9,484,708 pounds valued at \$528,388 as compared with 11,538,789 pounds worth \$1,183,470 imported in June 1930, and 10,491,890 pounds valued at \$1,304,289 received during the same month in 1929. Total importations of 37,790,623 pounds valued at \$2,254,487 were recorded during the first six months of 1931 contrasted with 64,711,480 pounds having a value of \$5,969,392 brought in during the corresponding period of 1930.

Cod Liver Oil Imports Greater in 1930.—Imports of cod liver oil into the United States in 1930 amounted to 21,700,000 pounds as compared with 21,450,000 pounds in 1929 and 19,350,000 pounds in 1928.