RELATIVE VALUE OF SLOW AND FAST PERCOLATION IN PREPARING TINCTURE OF RHUBARB AND AROMATIC TINCTURE OF RHUBARB U. S. P.*

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This work was undertaken at the request of the Revision Committee, U. S. P., to obtain information regarding the extraction of Rhubarb, the principal object being to determine the best method of percolation. In connection with such a problem, the question arose, "How long should the period of maceration extend and how rapidly should the drug be percolated in order to remove the emodin bodies, leaving behind those principles which tend to precipitate on standing."

Tinctures were prepared in order to determine the relative value of long and short maceration along with slow and fast percolation, these preparations consisting of three Tinctures of Rhubard, 1000 cc. each, and the same number and quantity of Aromatic Tincture Rhubarb. The menstrua used in all extractions were U. S. P. X (glycerin, 100 cc., alcohol, 500 cc., water, 400 cc.) as menstruum No. 1 and (alcohol, 1 vol., water, 1 vol.), as menstruum No. 2. The time of maceration and rates of percolation were varied so as to find an effective method of extracting the emodin bodies from the drug, leaving the tannoids behind. The following table shows the schedule carried out in each experiment.

TABLE I.—TINCTURE RHUBARB.

Drug.	Menstruum.	Time of Maceration.	Rate of Percolation.
A (Rhubarb, 200 Gm.)	1—Glycerin, 100 cc.; alcohol, 500 cc.; water, 400 cc. 2—Diluted alcohol	24 hours	0.5 cc. per minute
B (Rhubarb, 200 Gm.)	(Same menstruum as A)	6 hours	2.0 cc. per minute
C (Rhubarb, 200 Gm.)	(Same menstruum as A)	3 hours	4.0 cc. per minute

TABLE II.—AROMATIC TINCTURE RHUBARB.

Drug.	Menstruum.	Time of Maceration.	Rate of Percolation.
D (Rhubarb, 200 Gm.) (Cardamon Seed, 40 Gm.) (Clove, 40 Gm.) (Nutmeg, 20 Gm.)	1—Glycerin, 100 cc.; alcohol, 500 cc.; water, 400 cc. 2—Diluted alcohol	24 hours	0.5 cc. per minute
E (Same drug as D)	(Same menstruum as D)	6 hours	2.0 cc. per minute
F (Same drug as D)	(Same menstruum as D)	3 hours	4.0 cc. per minute

During the percolation of the tinctures, intermediate 5-cc. samples of percolate were taken when 250 cc., 500 cc., 750 cc. and 1000 cc. of percolate had been withdrawn from the drugs. These samples were taken so as to determine the comparative exhaustion (by presence of emodin bodies) of the drugs at different stages of percolation.

An investigation of the literature on the determination of the anthraquinone derivatives in cathartic drugs, revealed that none of the tests was very reliable. It was decided, however, that the method used in conducting the color tests for the presence of emodin bodies in the tinctures was sufficiently accurate to estimate the relative exhaustion of the drugs.

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The test based on the U. S. P. X identity test for Rhubarb was as follows:

To 1 cc. of the intermediate sample of tincture was added 9 cc. of solution potassium hydroxide (1 Gm. of 100% in 100 cc. water). This mixture was shaken, acidulated with HCl, allowed to cool and filtered. (A precipitate formed on adding the HCl, which was insoluble in ether.) The acid filtrate was shaken out with 10 cc. of ether, the ether layer becoming yellow on standing. The ethereal solution was shaken out with 5 cc. of ammonium hydroxide test solution. The ammonium hydroxide layer turned red showing a presence of emodin bodies and the ether layer remained yellow (chrysophanic acid).

TABLE III.—RESULTS OF TESTS FOR EMODIN BODIES IN INTERMEDIATE SAMPLES.

	250-Cc. Stage in Percolation.	500 Cc.	750 Cc.	1000 Cc.
A	Deep red	Light red	Pale red	Faint red
B	Red	Light red	...	...
C*	...	Deeper than A	Light red	Faint red

* Practically no difference in depth of color at corresponding stages.

The test used to determine the emodin bodies in the intermediate samples was also used for the finished tinctures.

TABLE IV.—EMODIN BODIES IN FINISHED TINCTURES.

Tinctures	A, B, C	Same depth of color.
Aromatic tinctures	D, E, F	Same depth of color but lighter than A, B and C.

Tests were also run on portions of the same tincture; one portion was thoroughly shaken to insure a uniform mixture, and the other portion was one from which the precipitate had entirely separated, having been standing undisturbed for one month. Depth of color in each test was practically the same.

TABLE V.—RESULTS OF TOTAL SOLIDS DETERMINATION IN TINCTURES.

Tincture Rhubarb.

A	18.63 Gm. per 100 cc.
B	18.36 Gm. per 100 cc.
C	18.755 Gm. per 100 cc.

Aromatic Tincture Rhubarb.

D	18.84 Gm. per 100 cc.
E	19.26 Gm. per 100 cc.
F	19.655 Gm. per 100 cc.

The following results were obtained as to relative depths of color of the finished tinctures: in the plain tinctures, A was the deepest in color, C next and B the lightest. In the aromatic tinctures, D had the greatest depth of color, F next and E was lightest.

TASTE COMPARISONS.

Results given by four observers, as to strength of tinctures, were not very definite.

TABLE VI.

Tincture.	Observers. 1.	2.	3.	4.	Aromatic Tincture.	Observers. 1.	2.	3.	4.
Strongest	C	C	C	B	Strongest	D	F	D	E
Next strongest	B	B	A	A	Next strongest	E	E	E	D
Weakest	A	A	B	C	Weakest	F	D	F	F

Per Cent Residue in Tinctures after Standing 8 Months.

Tincture Rhubarb	A 0.6 %	*Aromatic Tincture Rhubarb*	D 0.64%
	B 0.4 %		E 0.8 %
	C 0.36%		F 0.7 %

CONCLUSIONS.

1. The color tests (for emodin bodies) on the intermediate samples indicated that long maceration (24 hours) and slow percolation (0.5 cc. per minute) as in Experiment A, gives more rapid extraction of active principles, this percolate showing a greater quantity of emodin bodies at 250 cc. than B and C. Tests for emodin bodies in B (maceration, 6 hours—percolation, 2 cc. per min.) and C (maceration, 3 hours—percolation, 4 cc. per minute) appeared about the same at the corresponding stages of percolation, showing there was no particular advantage of one over the other in regard to time of maceration and rate of percolation.

2. Long maceration followed by slow percolation exhausts the drug with a smaller amount of menstruum than fast percolation, but the quantity of menstruum passed through each drug seemed sufficient to guarantee complete extraction, even with a short maceration period and fast (4 cc. per min.) percolation. This fact is borne out by the results obtained on testing for emodin bodies in the finished tinctures.

3. The results of the color tests were practically identical when performed on portions of the same tincture, one portion having been thoroughly shaken to insure a uniform mixture and the other portion being one from which the precipitate had entirely separated, having been standing undisturbed for one month. In the latter sample the quantity of tincture necessary for the emodin test was taken from the supernatant liquid. This result in the color test comparison indicates that no emodin bodies separate out with the residue.

4. The results in extraction for the Tincture of Rhubarb and the Aromatic Tincture of Rhubarb are practically the same. Little variation was observed in the samples in each series as to depth of color, taste, total solids, active principles in finished tinctures, and amount of residue after aging 8 months.

5. It appears that there is no outstanding value of long maceration and slow percolation over short maceration and fast percolation in preparing Tincture Rhubarb and Aromatic Tincture Rhubarb.

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