Occasionally a slight odor of acetic acid is observed during this heavy grinding. For this reason it is thought that a 100-mesh powder represents the optimum of uniformity to be sought.

As a further test of the possibility of decomposition of acetylsalicylic acid during melting, an analysis, according to the saponification method of the U. S. P. X, was made of the four acids after melting. Melting-point tubes were weighed, charged with the acid, ground to 200 mesh and dried, and weighed again. The melting point was then determined, using an initial bath temperature of 130° C. and a rate of heating of three degrees per minute. After the samples had cooled and solidified, the tubes were freed from adherent bath liquid and weighed a third time. The combined tubes containing each sample were ground in a mortar and the acid dissolved in alcohol. Fifty cc. of half-normal sodium hydroxide were added to the solution and the assay completed according to the official method. The results follow:

TABLE VIII.

Sample.	Fineness.	Purity before Melting.	Purity after Melting.
A	200	99.05	98.40
B	200	99.20	98.27
C	200	99.78	98.40
D	200	99.75	98.80

Samples were also melted as recommended here, with a fine strip of blue litmus paper placed in the top of the tube. The paper became red as the sample melted, showing the evolution of acetic acid, also detectable by its odor.

CONCLUSION.

In the opinion of the authors, the melting point rubric of U. S. P. XI should read as follows:

Acetylsalicylic acid has a melting point not below 135° C. when determined by the following method. Crush the acid to a No. 100 powder and dry in a desiccator over sulphuric acid for twelve hours. Place a column of the powder 2.5 to 3.0 cm. in length in a capillary melting-point tube having an internal diameter of 0.8 to 1.0 mm., sealed at one end. Place the tube in a bath previously heated to 130° C. and continue the heating at the rate of 3° per minute until the acid melts.

PITTSBURGH, PA.,
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TINCTURE NUX VOMICA.*

BY V. L. DICKEY AND F. W. NITARDY.

INVESTIGATION OF THE USE OF HYDROCHLORIC ACID AND ACETIC ACID FOR ACIDIFYING THE MENSTRUUM AND THE DETERMINATION OF THE EFFECT OF SLOW AND FAST PERCOLATION.

Experiments were carried out in an attempt to find a more satisfactory method of extracting Nux Vomica. The object was to determine the advantages and disadvantages of menstrua containing hydrochloric acid and acetic acid, respectively,

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and also whether any improvement resulted by slow or rapid extraction. Using 100-Gm. quantities of the same lot of drug in each case, tinctures were prepared with the following variations. The drug used assayed 2.984% alkaloids.

TABLE I.

Experiment.	Menstruum.	Time of Maceration.	Rate of Percolation.
A	1. Alcohol 750 cc.; water 240 cc. Acetic acid 10 cc. 2. Alcohol 3 vol.; water 1 vol.	24 hours	0.5 cc. per min.
B	1. Alcohol 750 cc.; water 245 cc. Hydrochloric acid U. S. P. X 5 cc. 2. Alcohol 3 vol.; water 1 vol.	24 hours	0.5 cc. per min.
C	1. Alcohol 750 cc.; water 242.5 cc. Hydrochloric acid U. S. P. X 7.5 cc. 2. Alcohol 3 vol.; water 1 vol.	24 hours	0.5 cc. per min.
D	(Same menstruum as A)	6 hours	2.0 cc. per min.
E	(Same menstruum as A)	3 hours	4 cc. per min.

On each reserve portion (950 cc.), the assay, acidity and p_H were determined and completeness of extraction was calculated. The results obtained were as follows:

Tincture.	Average of Two Assays.	Degree of Extraction.	Acidity Determination on 10 Cc. of the Tr. Expressed as Cc. $N/10$ NaOH Required. Phenolphthalein Indicator.	p_H.
A	0.2806 Gm. in 100 cc.	94.03%	7.55 cc.	5.32
B	0.2519 Gm. in 100 cc.	84.42%	5.55 cc.	1.99
C	0.2882 Gm. in 100 cc.	96.58%	8.45 cc.	1.55
D	0.1958 Gm. in 100 cc.	65.62%	7.60 cc.	5.19
E	0.1892 Gm. in 100 cc.	63.4 %	7.49 cc.	5.26

Tinctures A and C being high in assay were adjusted by using a menstruum of alcohol 3 vol.—water 1 vol., making the finished tincture conform to the required U. S. P. standard. Each of the five prepared tinctures was defatted by cooling to 5° C. and filtering. About the same amount of fat appeared to separate from each. Tinctures B, D and E were defatted without adjustment since the alkaloidal content on B was within the U. S. P. range and that of D and E below this range. Samples A and C were re-assayed, the results being as given in the following table:

TABLE II.

Tincture.	Average of Two Assays.	Acidity Determination on 10 Cc. of the Tr. Expressed as Cc. $N/10$ NaOH Required. Phenolphthalein Indicator.	p_H.
A	0.2475 Gm. per 100 cc.	7.14 cc.	5.45, check 5.43
C	0.2552 Gm. per 100 cc.	7.59 cc.	1.76, check 1.74

Extraction efficiency and completeness seem to depend on adequate maceration and slow percolation combined with a sufficient amount of available acid. The degree to which the acid ionizes seems less important. Tincture A was nearly equal in available acid to tincture C but of much higher p_H. However, extraction was nearly as good and when compared to tincture B which differed from C, only

in the amount of acid used, it would appear that with equal normality in acid present in the menstruum, there would probably be no difference in extraction efficiency between the use of acetic and hydrochloric acids. It also appears that 10 cc. of acetic acid or 7.5 cc. hydrochloric acid represent the minimum amounts per liter of menstruum that should be used and slightly greater amounts may prove still more effective.

Observations were begun on the tinctures two months after preparation, being kept in amber bottles at room temperature in the absence of light.

TABLE III.—SEDIMENTATION OF TINCTURES.

	Age of Tinctures at Observation.		
	2 Months.	3 Months.	7 Months.
Tincture A	−	−	+
Tincture B	+	+	++
Tincture C	+	+	++
Tincture D	−	−	++++
Tincture E	+	+	++

— represents no sediment.
+ represents slight sediment indicating the least.
++++ indicating the largest amount with no great difference between the two extremes.

A close examination was necessary to detect the precipitate in the tinctures as no great quantity was present in any of them. The sediment which does appear may be a trace of fat not removed in the defatting process. The supernatant liquid was clear in all samples.

CONCLUSIONS.

1. Adequate maceration and slow percolation are necessary for complete extraction.

2. Not less than 10 cc. of acetic acid or less than 7.5 cc. of hydrochloric acid per liter of menstruum should be used. Smaller amounts of acid yield less complete extraction.

3. Defatting does not remove alkaloids from the tincture, and aids in avoiding precipitation as the tincture ages.

4. Complete extraction resulting from longer maceration and slower percolation apparently does not lead to greater precipitation on aging. Acetic acid has a slight advantage over hydrochloric acid from standpoint of freedom from sedimentation of the finished tincture.

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Perhaps everyone will admit that if pharmacists magnified their profession to the fullest there would be developed a relatively greater appreciation of it; and, as a result, its opportunities would be developed to a greater extent, and the public could have a better understanding of its worth. There are few industries to which pharmacy has not directly or indirectly contributed; in most of the divisions of science and art, pharmacy has a part and has contributed very largely to the achievements of medicine.