THE U. S. P. TEST FOR ACETONE IN ALCOHOL.*

BY JOS. W. E. HARRISSON.

Prohibition has by necessity produced many new denatured alcohols some of which have been termed medicated alcohol. Medicated alcohols, so called because of their adaptability to certain restricted medicinal uses, particularly bathing, generally contain some substance that will produce nausea if imbibed, or alkaloids of an intense bitter taste.

Occasionally acetone is encountered as one of the denaturing agents and during the examination of such a medicated alcohol it was noticed that the U. S. P. test did not give positive results though, undoubtedly, there was at least five percent of acetone present.

Further search showed the presence of a zinc salt as well, which was afterwards proved to be the interfering substance. Antimony and potassium tartrate, which is also a permissible denaturant, affects the test in the same manner, both salts apparently interfering with the action of the sodium nitroprusside which is very unstable.

Numerous tests advocated to establish the presence of acetone were tried in an endeavor to find one unaffected by the interfering metallic salts. Following is a résumé in tabular form of the results obtained:

	U S P. test.	Iodoform.	Vitali.	Chautard.	Denigès.
Alcohol, 95%	negative	negative	false	negative	false
Alcohol, 1% Acetone	positive	positive	false	negative	false
Alcohol, .2% Acetone	positive	positive	false	negative	false
Alcohol, .1% Acetone	negative	positive	false	negative	false
Alcohol, Acetone, Zinc Chloride	negative	positive	false	negative	false
Alcohol, Acetone, Antimony and Potassium Tartrate	negative	positive	false	negative	false

Alcohol containing one percent acetone and the amount of metallic salts required by the Internal Revenue Department was used for the latter experiments.

Upon consulting the table the iodoform test seems to have been the most satisfactory; this is essentially that of Gunning. A small amount of iodine test solution is added to five cc of the sample and then sufficient potassium hydrate test solution or ammonia water to nearly decolorize the iodine. No heat is used or a positive test will be obtained with pure alcohol.

A more satisfactory modification is to add one cc of potassium hydrate T. S. to five cc of the alcohol and then add iodine T. S. until colored slightly. In the presence of at least 0.25 percent acetone there will be an immediate precipitate of iodoform. Pure grain alcohol will gradually give a positive reaction.

The U. S. P. test may be satisfactorily applied if the sample is previously distilled.

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DRUG TOPICS.*

No. 6. Observations on Commercial Star Anise.[1]

BY M. H. CHOW.

Separation of Fruit into Seed, Carpel and Stem.—In order to ascertain the ratio of the several parts of the fruit as found in the drug market, samples of selected, perfect fruit, also of more or less crushed commercial drug, were carefully sorted, the separated parts weighed and the percentages computed. For the sake of a more ready comparison, the results are herewith tabulated.

No.	Weight of original material.	Weight (in grams) of Seed.	Carpel.	Stem.	Percentage of Seed.	Carpel.	Stem.
1	500 grams	80	420	0	16.0	84.0	0
2	500 grams	69	430	0	13.8	86.0	0
3	1200 grams	..	...	150	..	..	12
4	44 grams	6	37.5	0	13.6	86.2	0
5	100 grams	21	79	...	21.0	79.0	..

Samples Nos. 1, 2 and 3 were the ordinary run of the drug obtained from Sing Chong Co., Chinese druggists, 22nd St., Chicago, Ill. Sample No. 4 consisted of perfect fruits, partly from the Drug Cabinet of the University, partly selected from No. 3. No. 5 consisted of perfect fruit obtained from Lehn and Fink, New York City. W. Meissner,[2] who in 1818 made a general chemical survey of the drug, found 21.6 percent of seed and 78.4 percent of carpels (source not stated).

In addition three samples of five hundred grams each from Lehn and Fink were sorted for purposes of chemical study into stems, leaves and fruits. Of stems they yielded 2.4, 3.2 and 2.2 percent, respectively; of leaves 5.1, 2.4 and 1.8 percent, respectively; and of fruits 93, 94 and 95 percent, respectively.[3]

Moisture Determinations.—Because of the volatile oil content[4] the xylene method was given preference, yet the older methods were also tried if for no other reason than to reveal more fully their inaccuracy. With the use of but 10 grams of drug, the amount of water obtained was so small that the percentage of error became too great. Hence 25 grams of No. 80 powder were used in the experiments the results of which are herewitn tabulated.

Expt.	Volume cc. of water collected. Seed.	Carpel.	Stem.	Percentage of moisture. Seed.	Carpel.	Stem.
No. 1	1.10	1.00	1.35	4.4	4.0	5.4
No. 2	0.90	1.40	1.40	3.6	5.6	5.6
No. 3	....	1.35	....	...	5.4	...
Average	1.00	1.25	1.37	4.0	5.0	5.5

In order to reveal the inaccuracy of the older method when applied to a volatile oil-yielding drug, the same materials were subjected to a temperature of from

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* From the Laboratory of Edward Kremers.

[1] Extracts from "A Chemical Investigation of Chinese Star Anise and Its Constituents," by Ming Heng Chow. A thesis submitted for the degree of Master of Science. University of Wisconsin, 1919.

[2] "Almanach oder Taschenbuch fuer Scheidekuenstler und Apotheker," 39, p. 1.

[3] See "Drug Topics" No. 2, JOUR. A. PH. A., 10, 272, 1921.

[4] Errors in gleaning or weighing or both must have crept in for the total of the weights of the parts amounted to 502.5 Gm. (100.10 percent), 498.0 Gm. (99.6 percent), and 495 Gm. (99 percent), respectively, in the three experiments. Mr. Chow has returned to China, hence his original notes could not be consulted conveniently.—E. K.

100 to 110° until of constant weight. The results in grams of the duplicate experiments are herewith tabulated:

	Seed.		Carpel.		Stem.	
	I.	II.	I.	II.	I.	II.
Original wt. of drug	2.4020	2.1164	2.0726	1.8924	3.0611	2.8347
Weight after drying	2.2354	1.9536	1.8946	1.7340	2.7991	2.5765
Loss of weight	0.1666	0.1628	0.1780	0.1584	0.1666	0.1528
Percentage moisture	6.93	7.69	8.05	8.37	5.44	5.39

C. E. Schlegel,[1] who in 1885 subjected the carpels to a proximate analysis by selective solvents, reports a loss of 10.36 percent of "moisture and volatile oil."

A comparison of results by the two methods is brought out in the following table.

	Xylene method, percent.	Drying at 100–110°, percent.	Difference, percent.
Seed	4.00	7.31	+3.31
Carpel	5.00	8.21	+3.21
Stem	5.50	5.41	−0.09

As was to be expected the seeds and carpel afforded appreciable differences according to the method employed, the stems practically none. By way of control it would be of interest to distil a large quantity of stems, if such were available, for volatile oil.

Finally, dehydration over sulphuric acid at room temperature was tried, weighings being made after one month and again after three months. The results of the determinations, in duplicate, on all three materials are again tabulated for ready comparison.

	Carpel.		Seed.		Stem.	
	I.	II.	I.	II.	I.	II.
Orig. wt. of drug	5.2984 Gm.	7.4767 Gm.	3.3700 Gm.	2.8946 Gm.	3.0618 Gm.	4.0192 Gm.
Wt. after 1 mo.	4.9220 Gm.	6.9827 Gm.	3.2126 Gm.	2.7258 Gm.	2.8620 Gm.	3.8206 Gm.
Loss of wt.	0.3764 Gm.	0.4940 Gm.	0.1574 Gm.	0.1688 Gm.	0.1998 Gm.	0.1986 Gm.
Percentage loss	7.10	6.61	4.67	5.83	6.52	4.7
Wt. after 3 mo.	4.9170 Gm.	6.9751 Gm.	3.2260 Gm.	2.7356 Gm.	2.8574 Gm.	3.8104 Gm.
Loss of wt.	0.3814 Gm.	0.5016 Gm.	0.1440 Gm.	0.1590 Gm.	0.2044 Gm.	0.2088 Gm.
Percentage loss	7.19	6.71	4.27	5.81	6.67	5.19

A comparison of the data recorded leads to the following conclusions:

1. That both the drying at about 100° and over sulphuric acid at room temperature yield results that are too high for the reason already indicated.

2. That the results for the seeds, however, differ in this respect from those obtained for both carpels and stems, namely, for the following reasons:

a. That whereas the loss of volatile oil with the water increases the apparent percentage of moisture according to the older methods;

b. The fatty oil content increases the weight of powdered drug (see weights after one and three months, respectively, in the above table) upon prolonged standing thus materially counteracting any loss in weight due to volatility of the substances constituting the so-called volatile oil.

Ash Determination.—In the first series of determination one gram of the powdered material was, in each case, heated in a crucible at a full red heat until the weight of the ash remained constant.

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[1] *Am. Journ. Pharm.*, 57, 426.

In computing the average, the first result, which is apparently too low, was rejected.

Expt.	Weight of ash, Gm. Carpel.	Stem.	Seed.	Percentage of ash. Carpel.	Stem.	Seed.
1	0.0459	0.0652	0.0255	4.59	6.52	2.55
2	0.0452	0.0658	0.0252	4.52	6.58	2.52
3	0.0457	0.0654	0.0252	4.57	6.54	2.52
4	.....	.....	0.0228	....	....	2.28
Average....	.....	.....	.....	4.56	6.55	2.53

In Experiment 4 the entire seed, not the powder, was incinerated. Oswald[1] in 1892 reports an ash content of 2.81 percent for the carpels and 2.46 percent for the seeds. Schlegel[2] reports 3.5 percent for the carpels.

In the second series of determinations the material was charred until it no longer gave off vapors. It was then lixiviated and the solution filtered through a quantitative filter. The filter plus the charred mass were heated to a high temperature until all carbon had disappeared. The residue remaining after this treatment was weighed and computed as water-insoluble ash. To it was then added the filtrate which was carefully evaporated and the residue heated to a dull red heat. The crucible was again weighed and the contents computed as total ash. The difference between total ash and insoluble ash was computed as water-soluble ash. The results of the duplicate determinations are herewith tabulated:

	Carpel. I.	II.	Stem. I.	II.	Seed. I.	II.
Weight of drug, Gm.....	2.0720	1.8924	3.0611	2.8347	2.0244	2.4020
Wt. of insol. ash, Gm.....	0.0595	0.0538	0.1494	0.1394	0.0454	0.0558
Wt. of total ash, Gm.....	0.1045	0.1929	0.2092	0.1908	0.0634	0.0728
Wt. of sol. ash, Gm.....	0.0450	0.0390	0.0598	0.0504	0.0180	0.0270
Percent of total ash.....	5.04	4.91	6.83	6.77	3.12	3.25
Percent of soluble ash.....	2.17	2.06	1.95	1.81	0.89	0.96

Comparison of these figures with the corresponding data of the first series show, as was to be expected, that the results of the total ash of the second series are somewhat higher than those of the first series.

Extractions.—In order to ascertain the extractive (fatty oil, volatile oil, etc.) for such solvents as ether, heptane and petroleum ether (58 to 60°) 30 grams of No. 100 powder were exhausted in a Soxhlet on an electric plate for about 48 hours or until a portion of the extract no longer left a residue upon evaporation of the solvent. The results of the duplicate determinations are likewise tabulated:

Solvent.	Seed. I. Percent.	II. Percent.	Carpel. I. Percent.	II. Percent.	Stem. I. Percent.	II. Percent.
Heptane.....	31.52	32.50	8.46	6.67	....	....
Ether.....	28.39	28.13	7.85	6.23	5.14	5.68
Petrol. ether.....	....	....	2.41	2.11	....	....

SOME CONSTITUENTS OF THE MOUNTAIN BALM.

BY J. W. HOWARD.

The Mountain Balm (*Ceanothus Velutinus*, Douglas) grows commonly on the mountainsides throughout the Rocky Mountain district. Coulter and Nelson[1*] have described is as "a smooth shrub 7–14 dm. high, growing in dense clumps or patches: leaves orbicular-elliptic or elliptical-ovate, obtuse at both ends, coriaceous, shining above (as if varnished) balsamic fragrant, lighter beneath and slightly pubescent, strongly 3-ribbed, 5–10 cm. long, on short stout petioles, persistent: . . "

[1] *Archiv. d. Pharm.*, 229, 84.

[2] *Am. Jour. Pharm.*, 57, 426.

* Numbers refer to Bibliography.