SUMMARY.

1. A method has been proposed for the manufacture of a Fluidextract of Uva Ursi in which the crystalline (ellagic acid) precipitate is prevented.

2. Experiments have been made which indicate that the crystalline (ellagic acid) precipitate in Fluidextract of Uva Ursi is due to enzyme activity.

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SOME NOTES ON THE TOLUENE MOISTURE DETERMINATION.*

BY H. GEORGE DEKAY.[1]

The determination of moisture by the toluene method was introduced into the eleventh revision of the United States Pharmacopœia for the first time. The purpose of this work is to compare the oven-drying method of moisture determination with the recommended procedure.

HISTORICAL.

The determination of moisture by distilling the sample with a liquid immiscible with water was first used by Marcusson in 1905 (1). Xylene was used as the immiscible liquid. Rogers (2) proposed the use of toluene to determine the moisture in leather. He used essentially the same procedure as that recommended by Marcusson. Dean and Stark (3) devised an apparatus in which the sample was refluxed with a liquid immiscible with water. The apparatus recommended in the eleventh revision of the U. S. P. is a modification of the Dean-Stark apparatus. Burns (4) used the xylene method in his work on the moisture content of aromatic drugs. Chow (5) found that the results of the xylene method were distinctly lower than those obtained by the oven-drying method. A number of investigators have used the distillation method with varying degrees of success. The collaborative work of Hoch and Prout on the moisture content of crude drugs led to the adoption of toluene as the immiscible liquid in the United States Pharmacopœia XI. They found that the one-hour distillation period recommended by Dean and Stark was not sufficient to obtain all of the moisture from ginger, acacia and asafœtida. The period of distillation is dependent upon the time required to remove all of the moisture from the crude drug.

EXPERIMENTAL.

A number of crude drugs were used in these experiments which had been purchased as official products. They were subjected to the oven-drying and the toluene distillation methods for moisture determination. The drugs selected were grouped into three main divisions, *First*, Drugs containing an official moisture standard; *Second*, Drugs which contained a volatile oil; and *Third*, Drugs containing resins. The drug was placed into an Erlenmeyer flask of 250–500

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cc. capacity and carefully mixed with dry sand. The toluene was added and the distillation carried out, using an electric hot plate as a source of uniform heat.

Satisfactory results were obtained with a three-hour heating period. A blank determination was made on the sand and toluene. The apparatus was calibrated using a bureau of standards calibrated burette.

The following table shows the relationship between the oven-drying and the toluene drying method for the determination of moisture in the first division of drugs listed previously.

TABLE I.

Acacia.

Toluene Method.				Oven-Drying Method.			
No.	Wt. of Drug.	Moisture, Cc.	Per Cent.	No.	Wt. of Drug.	Loss of Weight.	Per Cent.
1	25.000	2.65	10.6	1	20.8830	2.4097	11.54
2	20.000	2.15	10.75	2	12.8694	1.4588	11.33
3	10.000	1.05	10.50	3	6.4485	0.7266	11.26
	Average		10.62		Average		11.38
			Amylum.				
1	25.000	2.05	8.20	1	20.000	1.9219	9.10
2	20.000	1.60	8.00	2	15.000	1.3597	9.06
3	15.000	1.20	8.00	3	10.000	0.9004	9.04
	Average		8.07		Average		9.07
			Gentian.				
1	23.319	1.2	5.15	1	18.3896	1.0837	5.89
2	12.648	0.72	5.69	2	12.7726	0.7655	5.99
3	6.108	0.30	4.91	3	7.0325	0.4285	6.09
	Average		5.03		Average		5.99
			Glycyrrhiza.				
1	12.179	0.60	4.92	1	11.5699	0.7108	6.14
2	8.554	0.44	5.14	2	9.0536	0.5832	6.44
3	5.681	0.30	5.28	3	5.4235	0.3332	6.14
	Average		5.17		Average		6.24

The toluene method consistently gives a lower percentage of moisture than the oven-drying method. There is an apparent difference of approximately one per cent. This corresponds to the experience of different workers using this method (4, 5). The difference may be due to the oven-drying method causing a loss in weight of some constituent which is calculated as moisture.

Table II is a comparison of these same methods on drugs which contain a volatile oil as one of their constituents.

TABLE II.

Anise.

Toluene Method.				Oven-Drying Method.			
No.	Wt. of Drug.	Moisture, Cc.	Per Cent.	No.	Wt. of Drug.	Loss in Weight.	Per Cent.
1	17.100	0.95	5.55	1	12.9326	0.7557	5.84
2	12.883	0.70	5.43	2	10.1427	0.5884	5.80
3	9.304	0.50	5.37	3	6.4110	0.3794	5.87
	Average		5.45		Average		5.84

			Caraway Seed.				
1	13.355	0.72	5.31	1	13.8017	0.9811	7.11
2	10.687	0.55	5.14	2	8.6043	0.6163	7.16
3	7.983	0.45	5.64	3	4.2267	0.3014	7.13
	Average		5.39		Average		7.13
			Cardamom Seed.				
1	10.380	0.90	8.67	1	13.2373	1.3996	10.57
2	8.228	0.70	8.50	2	9.2357	0.9321	10.10
3	4.466	0.40	8.95	3	6.3620	0.6774	10.65
	Average		8.71		Average		10.43
			Black Mustard.				
1	13.091	0.52	3.97	1	13.5598	0.8236	6.07
2	9.147	0.40	4.37	2	8.1341	0.5076	6.24
3	7.895	0.35	4.44	3	5.3667	0.3406	6.22
	Average		4.26		Average		6.22
			Peppermint.				
1	5.248	0.37	7.05	1	8.0947	0.6105	7.54
2	4.207	0.30	7.13	2	8.5201	0.6634	7.61
3	3.033	0.22	7.25	3	5.2689	0.4060	7.70
	Average		7.14		Average		7.62

The volatile oil determination of the tenth revision of the United States Pharmacopœia was used with peppermint and it was found that the average of three determinations gave a yield of 0.32%. When this is subtracted from 7.62% of the oven-drying method, it will still be above that of the toluene determination. The volatile oil determinations on all of these drugs has not been completed but will be reported at a future date.

Table III is a comparison of the same methods as those of Table I and II except it involved drugs having a resin as a constituent.

TABLE III.

Aloe.

Toluene Method.				Oven-Drying Method.			
No.	Wt. of Drug.	Moisture, Cc.	Per Cent	No.	Wt. of Drug.	Loss of Weight.	Per Cent.
1	25.000	0.85	3.40	1	16.000	0.6300	3.93
2	20.000	0.65	3.25	2	12.000	0.4708	3.92
3	14.000	0.45	3.21	3	4.000	0.1550	3.87
	Average		3.29		Average		3.91
			Jalap.				
1	21.033	0.55	2.61	1	20.8739	0.6155	2.94
2	12.2566	0.30	2.45	2	15.6454	0.4625	2.95
3	11.5982	0.28	2.41	3	6.7513	0.2042	2.99
	Average		2.49		Average		2.96

CONCLUSIONS.

The toluene method of moisture determination gives concordant results. This method gives results which are consistently below those obtained by the oven-drying method.

A three-hour distillation period is sufficient for most drugs.

The method can be used for determining moisture in some drugs containing a volatile constituent.

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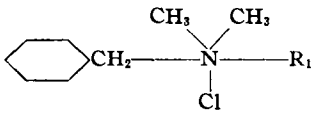
ANTISEPTIC PROPERTIES OF ALKYL-DIMETHYL-BENZYL-AMMONIUM CHLORIDE.*

BY PAUL G. HEINEMAN.[1]

INTRODUCTION.

The preparation investigated is of special interest because it is related to a group of chemical compounds, whose germicidal properties are apparently little understood. The chemical structure of the preparation is



(1)

It represents a mixture of high molecular alkyl-dimethyl-benzyl-ammonium chlorides, in which the high molecular alkyl residue (R_1) is composed of the alkyl radicals C_8H_{17}, $C_{10}H_{21}$, $C_{12}H_{25}$, $C_{14}H_{29}$, $C_{16}H_{33}$ and $C_{18}H_{37}$. The source of these radicals is the mixture of fatty acids of coconut oil in original proportion. Inasmuch as these fatty acids in coconut oil occur in constant proportion, this relationship holds in the final product resulting in a preparation of uniform composition.

The product is freely soluble in water, forming a clear, almost colorless solution. It is soluble also in acetone and in alcohol, but insoluble in ether and only slightly soluble in benzol. The aqueous solution is slightly alkaline to litmus. It possesses an aromatic odor and foams like soap on shaking. Aqueous solutions have an acrid taste, which disappears as dilutions increase.

Studies of antiseptic properties of the ammonium chloride compound have been carried on for a period of more than two years. The following standard procedures were applied:

1 Phenol coefficient determinations.
2 Inhibition and penetration tests.
3 Tests for disinfection of the human skin.
4 Destruction of spores of bacteria and fungi.

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