

TABLE I.—CRUDE FIBER DETERMINATIONS ON THE SAME SAMPLES BY DIFFERENT ANALYSTS.

| Analysts. | Gum A. | Gum B. |
|-----------|--------|--------|
| 1..... | 0.32% | 2.10% |
| | 0.46% | 2.06% |
| 2..... | 0.48% | 2.05% |
| | 0.57% | 2.35% |
| 3..... | 0.50% | 2.50% |
| | 0.52% | 2.67% |
| 4..... | 0.60% | 2.10% |
| 5..... | | 2.02% |

TABLE II.—CRUDE FIBER DETERMINATION IN COMMERCIAL SHIPMENTS AS IMPORTED.

| Laboratory No. | Crude fiber %. | Description of sample. |
|----------------|----------------|-------------------------------------------------------------------------------------------------------------|
| 73264 | 0.54 | Bark on 1/4 pieces; much soil; cherry-red; average sized pieces. |
| ... | 0.52 | Selected sample; barely passable grade; bark on 1/2 pieces; transparent; little soil; average sized pieces. |
| ... | 0.00 | Selected sample; best grade; no bark present; color transparent; no soil; pieces average size. |
| 71917 | 0.24 | Very little bark; transparent; no soil; pieces average size. |
| 73265 | 1.07 | Much bark; color red and yellow; much soil; size No. 5 sieve. |
| ... | 0.28 | Selected; bark small amount on 1/2 of pieces; transparent; little soil; average size. |
| ... | 0.65 | A little bark on every piece; color mixed; little soil; average size. |
| 74416 | 0.70 | A little bark on every piece; color mixed; little soil; average size. |
| 74485 | 1.23 | Much bark; dark color; little soil; pieces average size. |
| 74781 | 1.60 | Bark in excessive amount; shipments being cleaned (Nov. 2/18). |
| 74779 | 0.95 | Bark in excessive amount; shipments being cleaned (Nov. 2/18). |
| 74879 | 0.98 | Bark in excessive amount; shipments being cleaned (Nov. 2/18). |
| 74878 | 1.30 | Bark in excessive amount; shipments being cleaned (Nov. 2/18). |
| 74923 | 1.30 | Bark in excessive amount; shipments being cleaned (Nov. 2/18). |

TABLE III.—REDUCTION OF CRUDE FIBER BY COMMERCIAL METHODS OF CLEANING.

| Laboratory No. | Before cleaning. | After cleaning. |
|----------------|------------------|-----------------|
| 74485 | 1.23% | 0.78% |
| 74779 | 0.95% | 0.73% |
| 74781 | 1.60% | 0.51% |
| 74878 | 1.30% | 0.50% |
| 74879 | 0.98% | 0.46% |
| 75607 | 1.35% | 0.54% |
| 76971 | 1.07% | 0.96% |

DRUG TOPICS.*¹No. 2. The Moisture Content of Aromatic Drugs.²

BY WALTER C. BURNS.

The loss in weight which a drug suffers at 100° or thereabouts has customarily been computed as its moisture content. That such results may be as inaccurate

* From the Laboratory of Edward Kremers.

¹ The first note to be published under this general heading was the one by Raymond C. Schulz, "On the Ash of Kamala," which appeared in the *Pharmaceutical Review*, 25, 129 (1907). There has been no lack of material, but the time to edit laboratory notes seemed to be wanting.—E. K.

² Extracts from a thesis on "The Moisture Content of Aromatic and Other Drugs." University of Wisconsin, 1912.

as the method is unscientific in certain cases, may readily be demonstrated in connection with such aromatic drugs as cloves and cinnamon. Accurate moisture determinations of such drugs can, however, be made by employing the so-called xylene method worked out in the Forest Service:¹

Cloves.

A. The entire cloves, as found in the market, were ground and 10 grammes used for an experiment. Two experiments yielded 0.5 Cc. and 0.5 Cc., respectively, of water, corresponding to a moisture content of 5.0 p. c. and 5.0 p. c., respectively.

B. Penang cloves in very fine powder, of which 10 grammes were used in each case. Four experiments yielded 0.55 Cc., 0.55 Cc., 0.65 Cc. and 0.55 Cc., respectively, of water, corresponding to 5.5 p. c., 5.5 p. c., 6.5 p. c. and 5.5 p. c., respectively.

In order to compare these results with those obtained by the ordinary method of drying, 10 grammes were heated in a drying oven to a temperature of from 100 to 110° until of constant weight. The loss of weight in three experiments amounted to 1.92 Gm., 2.01 Gm. and 1.98 Gm., respectively, corresponding to 19.2 p. c., 20.1 p. c. and 19.8 p. c., respectively.

As was to be expected, the loss due to the large amount of volatile oil present was considerable and, if computed as moisture, would give results 300 to 400 percent out of the way.

The true moisture content has also been determined by H. H. Holmes,² who, in a commercial sample, found 0.35 Cc., 0.37 Cc. and 0.38 Cc. of water, respectively, in 10 Gm. amounts of drug, using the xylene method. These figures correspond to 3.5 p. c., 3.7 p. c. and 3.8 p. c., respectively, of moisture.

Saigon Cinnamon.

A. The whole drug, as ordinarily found in the market, was ground to a powder so as to cause complete exhaustion of the water by the xylene. 10 grammes of this powdered drug were used for each experiment. Four experiments yielded 0.35 Cc., 0.4 Cc., 0.5 Cc. and 0.6 Cc., respectively, of water, corresponding to a moisture content of 3.5 p. c., 4.0 p. c., 5.0 p. c. and 6.0 p. c., respectively.

In one experiment the 10 Gm. of powdered drug were allowed to stand in the xylene for three weeks before distilling the mixture. The distillate separated 0.4 Cc. of water corresponding to 4 p. c.

B. Finely ground cinnamon, such as is sold as spice, was used, 10 grammes being taken in each case. Five experiments yielded 0.6 Cc., 0.58 Cc., 0.6 Cc., 0.62 Cc. and 0.6 Cc., respectively, of water, corresponding to 6.0 p. c., 5.8 p. c., 6.0 p. c., 6.2 p. c., and 6.0 p. c., respectively.

In order to compare these results with those obtained by the ordinary method, 10 grammes were heated, in a drying oven, to a temperature of from 100 to 110° until of constant weight. In three experiments the loss in weight amounted to 0.95 Gm., 0.95 Gm. and 1.00 Gm., respectively, corresponding to 9.5 p. c., 9.5 p. c., and 10 p. c., respectively.

¹ Arthur L. Dean, "The Estimation of Moisture in Creosoted Wood," U. S. Department of Agriculture, Forest Service, *Circ.* 134 (1908).

² H. H. Holmes, "Determination of the Moisture Content of Vegetable Drugs," U. W. Thesis, 1911.

Here again the difference in the results for the two methods is great, though not as great as in the case of cloves. Yet a difference of about 100 p. c., due, no doubt, to the volatile oil content of the drug, renders the results by the older method practically valueless.

Peppermint.

The volatile oil content of such a drug as peppermint is much lower than that of such spices as cinnamon and particularly cloves. Yet there also it seems desirable to employ the xylene method or some modification thereof. In two experiments by Holmes,¹ 10 Gm. of powdered peppermint leaves yielded 0.4 Cc. and 0.4 Cc., respectively, of water, corresponding to 4 p. c. and 4 p. c. of moisture in the air-dried drug as taken from the container.

Gaultheria.

Wintergreen is not an aromatic drug in the same sense in which peppermint and the spices are aromatic drugs, for the methyl salicylate is not preformed in the fresh leaves and should not be noticeable in properly cured leaves. However, a close relation exists between the moisture content of improperly cured leaves and any odor of methyl salicylate observable in such a drug. In order to ascertain the true moisture content of such an improperly cured drug, the volatile methyl salicylate that has resulted upon hydrolysis should not be computed as moisture. Whether the older method be used or the xylene method, in both cases the temperature employed is above the thermal death point of the hydrolytic enzyme. It does not seem probable either that, if any slight reaction should take place before the thermal death point of the enzyme is reached, the amount of water thus taken from the drug in the hydrolysis will be sufficient to affect the results. The amount thus withdrawn would seem to be entirely negligible, certainly within the limits of error of the assay.

Holmes¹ assayed leaves that had been gathered and cured in June 1910 at the Indian Mission near Black River Falls. Two determinations, using 10 g. in each case as usual, yielded 0.6 Cc. and 0.6 Cc., respectively, of water, corresponding to 6 p. c. and 6 p. c., respectively, of moisture in the drug.

The oil content of this particular leaf was determined by Leonard.²

Another lot of gaultheria leaves from the same source was assayed by Hammersley³ for moisture content according to the xylene method, using 10 grammes for each assay. Five determinations yielded 0.45 Cc., 0.5 Cc., 0.45 Cc., 0.48 Cc. and 0.46 Cc., respectively, of water, corresponding to a moisture content of 4.5 p. c., 5.0 p. c., 4.5 p. c., 4.8 p. c. and 4.6 p. c., respectively.

¹ H. H. Holmes, "Determination of the Moisture Content of Vegetable Drugs," U. W. Thesis, 1911.

² H. W. Leonard, "On the distillation of oil of wintergreen in the United States," Thesis University of Wisconsin, 1911.

³ W. S. Hammersley, "The Determination of the Water Content of Some U. S. P. Drugs by Distillation with Xylene," University of Wisconsin, Thesis, 1911.