NOTES ON THE ASH YIELD OF GENTIAN.

BY C. E. SMYITHE AND E. L. NEWCOMB.

Gentian is a drug which is deserving of a much more extended study than has as yet been accorded it. The low normal ash yield is particularly interesting. Ordinarily, one would expect to find in a drug consisting chiefly of parenchyma tissue rich in protoplasmic constituents, a relatively high ash. On the other hand, woody tissue in abundance and with little calcium oxalate is usually indicative of a low ash. In the latter case, Licorice seems to stand out as a prominent exception.

Gentian contains no calcium oxalate and is composed chiefly of parenchyma tissue. The cells are rich in organic constituents. Qualitative and quantitative determinations of the ash yielded by our different drugs should be made. Such studies would no doubt throw much light on the nature of many of these complex substances.

We report herewith the results of a series of examinations to determine the total amount of ash yielded by various commercial samples and the amount of the ash which is soluble in 5% hydrochloric acid. The results also include figures obtained when the samples were specially treated in order to remove practically all foreign matter, such as sand, clay, etc. A subsequent report will present a method for quickly removing such foreign material by machinery and include ash percentages of the cleaned drug and of the dirt containing portion separated.

Our present results are tabulated herewith as follows:

Sample No.	Source of sample and remarks.	Percent total ash.	Percent insoluble in 5% HCL.
1.	Commercial powdered, in carton, purchased in 1920	3.33	0.64
		3.26	0.61
2.	Commercial powdered, in carton, purchased in 1918	3.13	0.66
		3.17	0.83
3.	Ordinary commercial whole drug, garbled to remove coarse foreign matter but not specially cleaned to remove sand or clay. Bought in 1920. Powdered in this laboratory	4.10	....
		4.12	....
		4.01	1.53
		3.79	1.36
4.	Commercial powdered, from stock, no date, probably purchased from local source	4.74	2.46
		4.79	2.59
5.	Commercial powdered, from stock, no date, probably purchased locally	2.95	0.77
		3.00	0.89
6.	Commercial powdered, from stock, no date, probably purchased locally	3.12	....
		3.13	0.35
		3.081	0.41
		3.103	0.485
7.	Commercial powdered, in tin, purchased in 1920	3.84	1.30
		3.51	1.37
8.	Commercial powdered, in carton, purchased in 1920	7.22	3.30
		7.31	3.52

9.	Select whole drug, bought 1920, dried, brushed with hand brush, powdered in this laboratory	2.89	0.76
		2.94	0.98
10.	Select whole drug, bought 1920, dried, brushed with hand brush, powdered in this laboratory	3.03	0.87
		3.05	0.64
11.	Select whole drug, bought 1920, dried, brushed with hand brush, powdered in this laboratory	2.34	0.61
		2.43	0.79
12.	Brushings from No. 9, above	24.03	20.67
13.	Brushings from No. 10, above	16.53	13.05
14.	Brushings from No. 11, above	19.52	16.15
15.	Leaf and stem remains and young leaves separated by grabling, powdered in this laboratory	5.656	1.264
		5.802	1.538

These results show that the normal ash, *i. e.*, the ash of the thoroughly cleaned drug, is between 2 and 2.5 percent. Commercial samples usually contain between one-half and three percent of foreign inorganic matter, insoluble in 5% hydrochloric acid.

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DETERMINATION OF SODIUM BENZOATE IN OLEOMARGARINE.

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After trying the methods recommended for the determination of Benzoate in fatty products without concordant results, the writer devised the following method by which one may check results within one one-hundredth percent. The method is also applicable to other fatty substances than oleomargarine.

Take 100 grammes of sample, treat with 50 mils of saturated salt solution containing approximately 0.5% of sodium hydroxide. Add phenolphthalein indicator and heat to boiling, adding a solution of sodium hydroxide if necessary to maintain alkalinity. Stir vigorously and transfer to a separatory funnel while hot and separate the aqueous layer. Repeat this operation five times. (Test has shown this to be sufficient.)

Filter the combined liquids with suction and to the filtrate add 4 grammes of sodium hydroxide and an excess of calcium chloride, an excess over that necessary to combine with the fatty acids. Calcium hydroxide is also precipitated which forms a very efficient filter bed. Again filter by suction.

Acidify the filtrate with hydrochloric acid and shake out five times with ether, using 15 mils each time. Wash the combined ether extractions with water and evaporate spontaneously. Dissolve the benzoic acid in neutral 50% alcohol and titrate with $\frac{N}{10}$ alkali, phenolphthalein indicator. Some albuminous matter is extracted by the ether but this in no way interferes with the titration.

The number of mils required for titration multiplied by 0.0072 gives the weight of Sodium Benzoate in 100 grammes of sample.