COMMERCIAL HYDRASTIS (GOLDENSEAL).*

BY ARNO VIEHOEVER.

Hydrastis is defined by the United States Pharmacopoeia as "the dried rhizome and roots of *Hydrastis canadensis* L.) (Fam. *Ranunculaceae*) without the presence or admixture of more than 2 percent of the stems, leaves or other foreign matter and yielding not less than 2.5 percent of the ether soluble alkaloids of Hydrastis." From this definition and standard one should expect to find on the market a drug which is not only up to the alkaloid standard but also quite free from foreign matter.

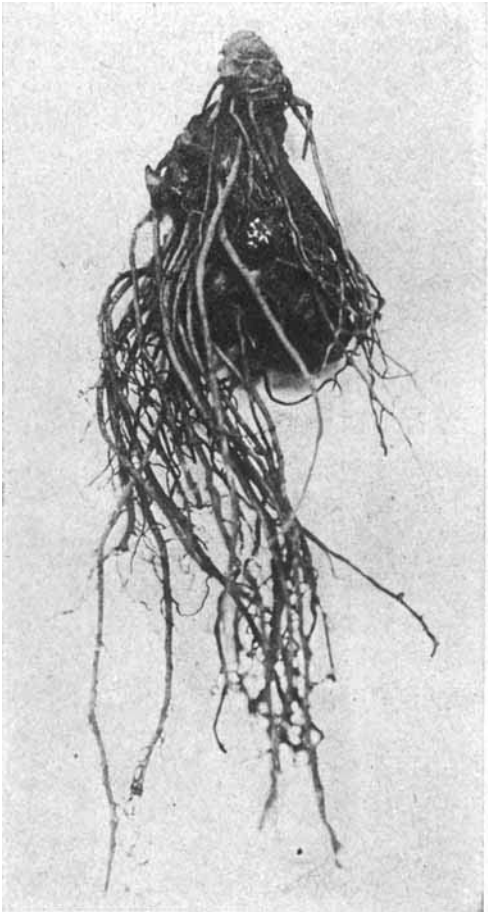

FIG. 1.—Mature rhizome of *Hydrastis* with roots, showing habit of growth. × 1. (After Van Fleet.)

In continuation of the Bureau's policy to extend the drug inspection from the imported drugs also to domestic drugs,[1] samples of Hydrastis were collected in the different states of the Union in the years 1917 and 1918. Hydrastis is certainly one of the most important domestic drugs, and, as is well known, is exported to a very considerable extent. Since it is a very expensive drug, its price per pound now being $5.70–$5.80 for the whole drug, and $6.50–$6.75 for the powdered drug,[2] it is especially important that it should come fully up to the standard. The samples were examined with regard to moisture, alkaloid content, total ash, and acid-insoluble ash. With the exception of one very small sample, in which the alkaloids could not be determined, the determinations indicated were carried out on all samples.

A few samples of known origin were also obtained, to ascertain, aside from the amount of alkaloid present, the amount of total and acid-insoluble ash in samples which had been carefully collected and specially washed after collecting. In one instance the material was dried at room temperature, in the other at temperatures rising up to 110° C.

The material was separated into rhizomes and roots and the determinations made on the separate parts. The results are tabulated below:

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* Read before Scientific Section, A. Ph. A., City of Washington meeting, 1920.

[1] A. Viehoever, C. O. Ewing and J. F. Clevenger, "Commercial Viburnum Barks and Preparations," THIS JOURNAL, 7, No. 11, 944–952, 1918.

[2] *Drug and Chemical Market*, 7, 658, April 7, 1920.

TABLE I.—EXAMINATION OF COMMERCIAL SAMPLES OF HYDRASTIS (GOLDENSEAL).

No.	Condition. Whole Rhizome.	Foreign Matter: Vegetable. Stems. %	Foreign Matter: Vegetable. Stems and Leaves. %	Foreign Matter: Mineral. %	Moisture. %	Ether soluble Alkaloids. %	Ash. Total. %	Ash. Acid insoluble. %	Analysts.[2]	Quality.	Remarks.
1	Whole root (Rhizome).....	...	...	.....	9.57	3.70	4.87	1.27	Woodruff	Good	.....
2	Abundant roots.....	...	...	.....	9.11	3.88	5.25	1.30	Zufall and Millner	Good	.....
3	Thick rhizome; has small amount of roots.....	...	...	.....	7.92	(2.24) 2.42	4.49	1.34	Glycart	Good	Deficient in alkaloids
4	Partially broken; pieces of rhizome 61.7%; rootlets and short pieces 35.5%..	...	2.8	Sand and clay[1]	4.89	3.76	6.16	1.68	Wichmann	Fair	Excessive foreign vegetable matter
5	Abundant roots; small rhizomes.....	...	...	.....	8.55	2.84	6.72	1.80	Glycart	Fair	.....
6	Rhizomes and roots, whole.	...	...	.....	...	(2.70) (2.69)	5.4	1.80	Clevenger Viehoever	Fair	Fairly clean
7	Rhizomes and roots, whole.	...	...	.....	...	..	6.4	1.90	Clevenger	Fair	Fairly clean
8	Rhizomes, roots.....	...	...	.....	7.72	(3.54) 3.74	6.46	2.09	Glycart	Fair	.....
9	Abundant roots, less rhizomes.....	2.0	...	.....	8.45	(4.24) 4.31	6.72	2.10	Glycart	Fair	.....
10	Probably whole root.....	...	...	.....	10.27	4.72	6.25	2.45	Zufall and Millner	...	.....
11	Rhizomes and roots.....	...	About 0.5	.....	...	2.90	7.14	2.64	Harrison	...	Considerable soil present
12	Probably whole root.....	...	...	.....	8.15	Sample too small	7.11	2.90	Kub	...	.....
13	Probably whole root.....	...	...	.....	7.90	4.64	8.35	3.45	Zufall and Millner	...	Excessive mineral matter
14	Probably whole root.....	...	...	.....	9.16	3.86	8.00	3.65	Zufall and Millner	...	Excessive mineral matter
15	Rhizomes and roots; coarsely broken.....	0.5	...	.....	6.95	3.58	10.16	5.00	Glycart	...	Excessive mineral matter
16	Probably whole root.....	...	...	.....	9.72	4.16	9.70	5.10	Zufall and Millner	...	Excessive mineral matter
17	Rhizomes, broken.....	...	Trace	.....	8.95	(2.28) 2.30	10.65	5.95	Eaton	...	Deficient in alkaloids; excessive mineral matter
18	Probably whole root.....	...	...	.....	...	2.43	(13.35)	(9.1)	Zufall and Grant	...	Shipment held for export

[1] Dried *in vacuo* at 70° C. [2] All from U. S. Department of Agriculture, Bureau of Chemistry.

Powdered Rhizomes						14.8	10.5		
1 Powdered	...	...	.....	9.60	3.23	8.67	3.67	Grant	... Excessive mineral matter
2 Powdered	...	...	Sand	9.86	2.90	11.03	4.83	Woodruff	... Excessive mineral matter
3 Powdered	...	...	Sand	...	3.0	10.28	5.08	Calloway	... Excessive mineral matter
4 Powdered	...	...	.....	5.88	2.69	11.49	5.63	Glycart	... Excessive mineral matter
5 Powdered	...	...	Sand and clay	4.30[1]	2.70	11.30	6.61	Wichmann	... Excessive mineral matter
6 Powdered	...	...	Sand and clay	5.12[1]	2.32	12.07	6.67	Wichmann	... Deficient in alkaloids; excessive mineral matter, and so forth
7 Powdered	...	...	Sand	9.00	2.85	12.26	6.67	Woodruff	... Excessive mineral matter
8 Powdered	...	Trace	.....	8.05	2.64	12.15	7.05	Eaton	... Excessive mineral matter
9 Powdered	...	Trace	.....	8.85	2.44	12.54	7.20	Eaton	... Excessive mineral matter
10 Powdered	...	...	.....	6.07	2.51	14.71	8.25	Glycart	... Excessive mineral matter
11 Powdered	...	...	.....	...	2.62	13.01	8.31	Grant	... Excessive soil
12 Powdered	...	...	.....	...	1.15	15.50	10.00 (sand)	Murray	... Consists largely of roots
13 Powdered	...	...	Sand	8.72	2.64	17.81	11.22	Woodruff	... Excessive mineral matter
14 Powdered	...	...	.....	...	(1.74) 1.75	11.72	...	Grant	... Deficient in ether soluble alkaloids. Many hairs, which indicate much leaf

TABLE II.—EXAMINATION OF AUTHENTIC SAMPLES OF HYDRASTIS (GOLDENSEAL).

Laboratory Number	Sample. Part used.	Sample. Amount present. %	Ether Soluble Alkaloid. %	Ash. Total. %	Ash. Acid-insoluble. %	Analysts.	Remarks.
2089	Rhizome	82.5	3.38	4.7	1.4	J. F. Clevenger	.....
	Roots	17.5	2.36	9.4	3.4	J. F. Clevenger	.....
2886	Rhizome	51.6	3.90	5.6	2.6	J. F. Clevenger	6.9% moisture
	Roots	48.4	2.55	8.3	3.3	J. F. Clevenger	6.8% moisture
2913	Rhizome	59.4	3.88	6.2	1.5	A. Viehoever and J. F. Clevenger	.....
	Roots	40.6	2.15	9.5	2.9	A. Viehoever and J. F. Clevenger	.....

DISCUSSION OF RESULTS.

Alkaloid Content.—The data in Tables I and II show an amount of alkaloids present, which, with the exception of a few samples, exceeds the minimum required by the U. S. P. limit of tolerance. Even where the amount found is less than 2.50 percent, the differences are not great.

This finding agrees with the report in the literature[1] that authentic samples of hydrastis usually contain materially more alkaloid than is required by the U. S. P. limit of tolerance. Of particular interest is the finding of this difference in alkaloid content of root and rhizome. LaWall[2] had already pointed out that he found 2.48 percent in a rhizome, while the roots contained only 1.38 percent, and concludes on the basis of the examination of this one sample: "Hydrastis rhizomes are between 1.5 and 2 times as rich in alkaloids as the rootlets." Our data show that there is indeed a difference in the alkaloid content, though it may be less than that found by LaWall.

That the amount of alkaloid may vary greatly between rhizomes and roots of the same plant material has been observed in other drugs. Bredemann, for instance,[3] reports that roots of White Hellebore (*Veratrum album* L.) in some cases were found richer, in other instances, poorer in alkaloids than the rhizomes. He points to the possible influence of time of collection and manner of drying upon the alkaloid content. At any rate, the following statement in the literature:[4] "This 'fiber' as it (mass of roots) is commercially termed, has equal medicinal value with the rootstock," is evidently too general. The further statement that the fiber realizes only about half the price when separated from the rootstock deserves special attention.

While no analytical data are included, the following statement by Lloyd[5] is of considerable interest:

"When the dried rhizome is kept from season to season, it gradually changes internally to brown, or greenish brown. This alteration commences at the surface and creeps inward, until after some years, by this form of decay, the yellow principles will have nearly perished, and the drug will have become proportionally of less value."

Since analytical data are lacking, it appears still undecided whether the drug harvested in the spring or in the fall contains more alkaloid. According to Dohme[6] "the spring root is better than the fall drug," and according to Henkel and Klugh[7]

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[1] Reports of Committee on Quality of Medicinal Products; Reports of Committee on Drug Market; Report of Penna. Ph. Association Committee on Drug Market, in THIS JOURNAL, "Digests of Comments on the Pharmacopoeia of the United States," etc.

[2] Charles H. LaWall, "Comparative Alkaloidal Strength of Hydrastis Rootlets and Rhizome," THIS JOURNAL, 1, 799, 1912.

[3] G. Bredemann, "Über die Alkaloide der Rhizome von *Veratrum album* und über die quantitative Bestimmung derselben," *Apotheker-Zeitung*, Nos. 5 and 6, 1906.

[4] Walter Van Fleet, "Goldenseal Under Cultivation," U. S. Department of Agriculture, *Farmer's Bulletin*, No. 613, 2.

[5] *Bulletin of the Lloyd Library*, No. 10, 76–184, 1884; *Hydrastis canadensis*, Goldenseal.

[6] A. R. L. Dohme, "How Drugs Vary in Strength and Quality," in *Apothecary*, 2, 942, 1905.

[7] Alice Henkel and G. Fred Klugh, "The Cultivation and Handling of Goldenseal," U. S. Department of Agriculture, Bureau of Plant Industry, *Circular* No. 6, 10, 1908.

"the root should be collected in the autumn after the plants have matured seed. Spring-dug root shrinks more in drying and always commands a lower price than the fall-dug root."

ASH CONTENT.

The cleanliness was only in a few instances that which could be considered desirable or of falling within the limit of 2 percent for foreign matter. While little foreign vegetable matter was observed, high amounts of total ash, and especially also of acid-insoluble ash, were found in all powdered samples, and such of the unground samples as were obviously dirty. The low amount of ash in samples which were clean, or fairly so, rather demonstrated that the natural content in mineral substances is by no means high. It is of interest in this connection that the Austrian (VIII, 1906), Swiss (IV, 1907), Italian (III, 1909), and Netherlands (IV, 1915) pharmacopoeias all have a standard limiting the amount of total ash to 6 percent. The British Pharmacopoeia alone has a higher standard, namely, 11 percent.

Among others, Riedel[1] reports samples of hydrastis having only 4.7–6.3 percent total, and 1.9 percent acid-insoluble ash. The data which we obtained with the separated parts of samples that had been washed and largely or wholly freed from adhering dirt, show conclusively, we think, the low natural content of mineral substance in the rhizomes as well as the roots. The roots appear to have a somewhat higher total and acid-insoluble ash than the rhizomes, though the samples examined are too few to make a general statement. In contrast to this low limit, the British Pharmacopoeia has a standard of 11 percent. The few samples which were clean gave an amount of ash which was either somewhat below 6 percent or occasionally slightly above the limit. When rhizomes and roots were examined separately it was evident that the rhizomes contained appreciably less than 6 percent, the roots appreciably more than 6 percent. The proportions of roots to rhizomes varied; in one sample we found roughly one-fifth roots, in another about equal parts. As the proportion of roots may reach 50 percent and possibly more, the ash standard should take the highest amount of ash in the roots into consideration. The limit of 6 percent, while possibly satisfactory for the rhizomes, appears to be too rigid for the roots. Since the roots contain considerable amounts of alkaloid, though probably at times less than the limit requirement, and since, furthermore, the root is admitted in the pharmacopoeias which have an ash limit for the drug, it is believed that a maximum value of 8 percent total ash would be more satisfactory than one of 6 percent. The standard of 11 percent, which the British Pharmacopoeia adopted, is believed to be too high, as our data show.

While it is conceded, from the habit of growth (see Fig. 1), that soil and sand are apt to adhere to the rhizome and roots and may not be easily removed when the drug is in the dried condition, there are useful devices which should prove of value in the cleaning[2]; furthermore, there seems to be no objection to the washing

[1] *Riedel's Berichte*, 1912, p. 50; from *Digest of Comments*, 1914, p. 308.

[2] C. H. Rogers and E. L. Newcomb, "A Method for Cleaning Digitalis, with a Study of the Inorganic Constituents," *Am. J. Pharm.*, 90, p. 239–252, 1918.

of the drug shortly after collection in order to remove the soil. In fact, Van Fleet,[1] speaking of digging and curing the cultivated roots, suggests this procedure:

"The rootstocks and attached rootlets are washed clean of all soil and freed from sticks, pebbles, or other foreign matter lodged in the fibrous masses."

CONCLUSIONS.

It appears clear that a maximum limit of tolerance of 8 percent for total ash would be quite liberal, and the adoption of such a tentative limit by this country, producing the drug for domestic use and for export, as stated, has therefore been proposed.[2] A tentative limit of 3.0 percent of acid-insoluble ash is also suggested as a fair maximum limit of tolerance.

The findings with regard to the alkaloid content also indicate that the alkaloid requirement could well be raised to 2.75 percent, if the drug were in a properly cleaned condition.

SUMMARY.

It has been shown that:

(1) The proportion of rhizome to roots varied considerably in the samples examined.

(2) The alkaloid content was found to be higher in the rhizomes than in the roots. A previous report in literature is thus confirmed.

(3) The raising of the required minimum alkaloid content to 2.75 percent of ether-soluble alkaloids is suggested.

(4) Samples collected in interstate trade, while containing sufficient alkaloid, as required by the United States Pharmacopoeia, contained rather generally amounts of mineral matter greatly in excess of that naturally present.

(5) The total and acid-insoluble ash in the roots was found to be higher than in the rhizomes.

(6) A maximum limit of 8 percent for total ash, and 3 percent for acid-insoluble ash is proposed.

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PHARMACEUTICAL PROBLEMS PRESENTED BY THE DAKIN PRODUCTS.*

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Early in the World War it became evident that the economics of warfare demanded investigation of the problems of sepsis and antisepsis. Quick repair of wounds was a consideration of prime value as the return to the field of injured experts meant more for ultimate success and speedy victory than the training of recruit substitutes.

Attention was early drawn to the advantages presented by the hypochlorites and an investigating committee appointed under Col. E. F. Martin as chairman.

We shall pass the therapeutics of hypochlorites to emphasize the essential importance of using a calcium hypochlorite of known and definite free chlorine strength, as brought out by the fact that few if any products on the market could

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[1] Walter Van Fleet, "Goldenseal Under Cultivation," U. S. Department of Agriculture, *Farmer's Bulletin* No. 613, 12, 1914.

[2] C. O. Ewing and A. Viehoever, "Acid-Insoluble Ash Standards for Crude Drugs," THIS JOURNAL, 8, No. 9, p. 725–730, September 1919.

* Read before Scientific Section, A. Ph. A., City of Washington meeting, 1920.