June 1920 AMERICAN PHARMACEUTICAL ASSOCIATION

RELATIVE CONTENT OF VOLATILE OIL AND ASH IN SAGE LEAVES AND STEMS.*

BY ARNO VIEHOEVER AND JOSEPH F. CLEVENGER.

One of the spice products seriously affected by the war is sage. Before the war the material imported came almost exclusively from Austria, representing usually normal sage from *Salvia officinalis* L. With the increasing difficulty of obtaining Austrian sage, due to the blockade of Austrian ports, substitutes were imported, obtained from *Salvia triloba* L., collected in Greece, and *Salvia lavandulaefolia* Vahl., collected in Spain. These products were so different in appearance and flavor that the Department deemed it advisable to require the labeling "Greek" or "Spanish Sage," respectively.¹ Especially in the Greek product the amount of stems was frequently far in excess of that found in the previously imported Austrian sage. This excess was due in part to the different manner of growth of the plant, but especially to the lack of care in collecting the leaves.

With the raising of the blockade of Austrian ports new shipments of Dalmatain sage, obtained from Salvia officinalis L., were offered for entry. These, however, contained very considerable amounts of stems, more or less in excess of the 12 percent limit adopted by the Government.² Since so much difficulty has been and is still being experienced in obtaining material from European markets, properly collected, without excessive stems, the suggestion was made by representatives of the trade that such products, although they contained considerable amounts of stems, might be permitted entry. The limit of stems in the whole product, as mentioned above, is 12 percent, while a subsequent ruling of the Department³ limits the crude fiber in the ground sage to 25 percent. Definite data, however, were lacking to show the justification for the exclusion of products containing considerable amounts of stems.

It should be noted here that American sage, which was grown in increasing amounts during the war, to our knowledge did not usually contain such an excess of stems as noted in the imported material. We are informed by growers of American sage that the domestic product generally contained excessive amounts of ash and acid-insoluble ash, exceeding those set by the United States regulations. To verify these findings, and, if possible, to find the explanation, consideration was given to this question.

VOLATILE OIL.

While the organoleptic tests of different parts of the sage plants—leaf, petioles, and the stem—suggested distinct difference in oil content and consequently spice value, no data could be found in literature or the records of the Bureau showing the quantitative difference in the oil content of these different parts.

Botanical Data.—The volatile oil, as is generally conceded, is located in glands and glandular hairs.

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

¹ Service and Regulatory Announcements. Chemistry, 23, Item 277, 1918.

² Service and Regulatory Announcements. Chem., 19, Item 195, 1917.

³ Department of Agriculture Circular, 136, "Standard of Purity for Food Products," p. 15,

The glands are disc-like, forming an 8-celled head, containing the oil in a brownish yellow secretion. They are either sessile or supported by a very short one-celled stalk. Glandular hairs occur in two types: (a) having a 2- to 4-celled

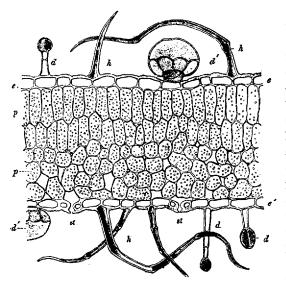


Fig. 1.—Leaf of Salvia officinalis L. (Cross section.) e, upper epidermis; e', lower epidermis; p, mesophyll; h, non-glandular hairs; d, glandular hair; d', disc-like gland; st, stomata. Approx. \times 150. After Vogl.

stalk and a small one-celled more or less spherical head; (b) having a one-celled stalk and a 2-celled more or less oval head. In either case the heads contain the glandular secretion (see Fig. 1). The glands and glandular hairs are found on the epidermis of the leaves. Here they occur on either side, though Wall⁴ reports them only on the lower side. We also found them on the petiole and even on such parts of the stem where the epidermis had not been replaced by cork or woody tissue. They evidently occur most abundantly on the leaves, and the younger leaves or those of the flowering stem likely contain more unimpaired glands and glandular hairs than the old leaves.

The suggestion is made in

books, dealing with the cultivation of sage, to collect the leaves during the flowering period in June and July, and is based, quite likely, on the experience that the leaves contain more oil at that stage of growth. At any rate, an analysis of domestic sage leaves collected in late summer and fall proved them below standard for volatile oil, as shown later on.

Fig. 2 shows the bushy habit of growth of sage (*Salvia officinalis* L.) collected in this country. It demonstrates how, by careless collection, many stems might be included in the drug. Even the harvesting of the leaves by cutting the leaves with a mower will likely include many woody stems. This method of harvesting is referred to in *Farmers' Bulletin* 663⁵ as a means of getting a cheap grade.

Chemical Data.—The material imported as "Dalmatian Sage," as well as the American sage, was carefully separated into leaves and stems. The separations were ground to powder and immediately extracted. The amount of volatile oil was determined by the indirect method for volatile ether extract described in "Methods of Analysis"⁶ and also under "General Tests."⁷ The amounts found by this method are given in Table I.

As seen from the table, the amount of volatile ether extract found in the

⁴ Wall, Otto A. "Handbook of Pharmacognosy," p. 359, 1917.

⁵ U. S. Department of Agriculture, *Farmers' Bulletin*, No. 663, p. 33, 1915. "Drug Plants under Cultivation."

⁶ Jour. of Official Agricultural Chemists, p. 318, 1916. "Spices and Condiments."

⁷ United States Pharmacopoeia, Ninth Decennial Revision, p. 591, 1916.

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 ⁴ Determined by J. F. Clevenger, ⁵ Collected in fall of 1919, in Virginia. ⁶ Collected in late summer, 1919, in Maryland, ⁴ Collected in late summer in Wisconsin, 	1 D		2922		2921			2920				3038			3037			3036			2990			2943		No.	Lab.			
	etermined		Rejections		Leaves			Lcaves				Leaves			Leaves			L,eaves		L,eaves				Leaves	endua	employed	Part			
	Stems by J. F. C	Stems	6	Stems	2		Stems			Stems Stems		ļ		Stems				Stems		Stems				Stems		yed.				
	Woody, from bases	some leaves	Herbaceous, many woody stems and	Herbaceous and woody, without leaves.	Without stems		Herbaceous and woody, without leaves.	Without stems		Somewhat woody	Herbaceous, without leaves	Without stems	out leaves	Herbaceous and somewhat woody, with-	Without stems	out leaves	Herbaceous and somewhat woody, with-	Without stems	out leaves	Herbaceous and somewhat woody, with-	Without stems	out leaves	Herbaceous and somewhat woody, with-	Without stems		Condition.				
		0.59	5		1.63			1.18	H			1.06			1.06			1.26			0.92	:		0.92	A		(%).	extract	ether ¹	Volatile
	0.20	0.94		0.60 Rej		Rele	0.48		almati	0.22	0.47		0.29			0.49			0.24						umerica		Ÿ	act.		1.
	4.0	4.7 5.3		0 5.6 Rejected Portion.	6.2	Released Portion.	:	7.2	Dalmatian Sage.	5.0	7.4	17.9	7.3		15.4	8.0		13.3	5.5		12.6	6.1		8.0	American Sage.	Total.		(%).	Ash. ¹	
	0.2	0.2 0.5		0.5	0.6		:	0.9		1.6	2.9	11.8	2.9		8.0	2.4		5.3	0.9		:	1.1		1.8		insoluble.	Acid			
	Commercial sample	Commercial sample Commercial sample		Commercial sample	Commercial sample		Commercial sample	Commercial sample			Non-commercial sample	Non-commercial sample ⁴	Non-commercial sample		Non-commercial sample ⁴	Non-commercial sample			Non-commercial sample			Non-commercial sample		Non-commercial sample ²		Source.				
	Separated in laboratory	Commercially separated Separated in laboratory		Separated in laboratory	Separated in laboratory	•	Separated in laboratory	Separated in laboratory		Separated in laboratory, 1919 crop	Separated in laboratory, 1919 crop	Separated in laboratory, 1919 crop	Separated in laboratory, 1918 crop		Separated in laboratory, 1918 crop	Separated in laboratory, 1917 crop	:	Separated in laboratory, 1917 crop	Separated in laboratory		Separated in laboratory	Separated in laboratory		Separated in laboratory		Remarks.				

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leaves of samples collected in Maryland and Viriginia, was below the limit of tolerance of 1 percent. This is quite likely explained by the fact that the material was collected in late summer or fall. Material collected in Wisconsin yielded higher amounts. Since these leaves also, as far as we could learn, were collected

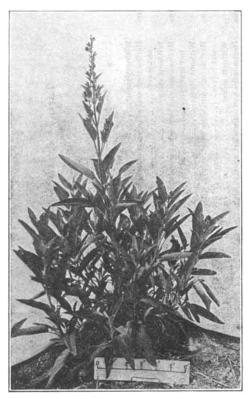


Fig. 2.—Salvia officinalis L. Cultivated plant, showing bushy habit of growth.

rather late in the year, the data given are likely not expressive of the highest amounts of volatile ether extract occurring in good American sage.

The amount of volatile ether extract found in the leaves of Dalmatian sage was higher than 1 percent. In both the American and the Dalmatian sages the stems yielded decidedly less volatile ether extract than the leaves. Of especial interest are the results obtained with the stems, separated into herbaceous tops and woody bases. From the data given, it is evident that appreciable amounts, though lower than the limit of tolerance, were found in the tops containing no woody portions. The more or less woody part, representing the main portion of the stems, however, contains very little volatile oil. The limitation of the stems in sage, therefore, is justified.

The data recorded are the highest found in a number of determinations. Material apparently somewhat heated during grinding, and other material in a ground condition kept in an open container for a number of

days, yielded results which were appreciably lower, though indicating the relative difference in oil content of the different parts. These observations may have a bearing on commercial practices of grinding sage and marketing it in the ground state. When testing for the amount of volatile ether extract it is considered advisable to extract the material immediately after grinding.

It is of interest in this connection to note that the separation of stems from the leaves is entirely feasible on a commercial scale. Such separations, to our knowledge, have been carried out quite successfully by passing the material between rollers.

ASH.

Both stems and leaves of American, as well as Dalmatian sage, were examined for the amount of total and acid-insoluble ash. The data are given in Table I. It will be seen that the amounts found in imported Dalmatian sage are well within the limit of tolerance. In only one sample of American sage was the total ash with-

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in the limit of tolerance; in all other samples both the total and acid-insoluble ash exceeded the limits of tolerance, namely 10 percent for total ash, and 1 percent for acid-insoluble ash. As was expected from other general experience, the amount of both total and acid-insoluble ash was found to be higher in the leaves than in the stems. While the explanation for the high acid-insoluble ash may be found in the fact that the samples examined were grown on sandy soil, the samples were too few to justify general conclusions.

CONCLUSIONS.

The amount of volatile oil (volatile ether extract) found in sage leaves was considerably higher—about three times that found in the stems—being in the ratio of 1.63-0.60, 1.26-0.49, 1.18-0.48, 1.06-0.29, 0.92-0.24, respectively.

The herbaceous parts of the axis located close to or representing the tops of the plant, as might be expected, yielded more volatile ether extract than the woody basal portion of the axis, the amounts being about 0.9 percent and 0.2 percent, respectively.

The chemical findings are supported by the following botanical data: The glands and glandular hairs containing the volatile oil are to be found only in the epidermis of leaves, petioles and herbaceous stems. They are most abundant on the leaves, either upper or under side, and completely absent on woody stems, where the epidermis has been replaced by cork and woody tissue.

Since the stems are low in volatile oil, and hence, in spice value, a limitation of their amount is justified.

The leaves generally contain more ash than the stems. With regard to the total and acid-insoluble ash, determinations made on 5 samples indicate a tendency in domestic sages of high total and especially acid-insoluble ash, a tendency observed by growers of domestic sage.

PHARMACOGNOSY LABORATORY, BUREAU OF CHEMISTRY.

HEMLOCK BARK (Tsuga canadensis) FOR PHARMACEUTICAL PURPOSES.*

BY H. D. GARR AND GEORGE E. ÉWE.

Hemlock bark finds its greatest use in the tanning industry, but considerable is employed in Pharmacy. Its use in the tanning industry is dependent upon its tannin content. In pharmacy the tannin content is also the most important consideration but, in addition, the oleoresin and volatile oil contents are frequently of importance.

For the purposes of tanning, the whole bark is usually employed, and it is not considered necessary to remove the outer cork layers. But in pharmacy the rossed bark is to be preferred since the outer cork layers are deficient in tannin, practically devoid of volatile oil, and carry excessive color and extractive matters which are non-essential to the purposes of pharmacy.

Kraemer states in "Scientific and Applied Pharmacognosy" that only the

^{*} Read before Philadelphia Branch, A. Ph. A., May meeting, 1920.