COMMERCIAL VIBURNUM BARKS AND PREPARATIONS.*

BY ARNO VIEHOEVER, CLARE OLIN EWING AND JOSEPH F. CLEVENGER.

Among the domestic drugs which are collected rather extensively and used in medicinal preparations, mention must be made of two or three *Viburnum* species, namely *Viburnum opulus* L. (Cramp bark) and *Viburnum prunifolium* L. or *Viburnum lentago* L. (Black Haw bark). The demand for *Viburnum opulus* especially has been considerable during years past. The recent report in literature, referred to later, that *Viburnum opulus* was substituted by *Acer spicatum* L., to which the name cramp bark was given, therefore created considerable interest and suggested a collection of samples in order to verify the fact and the extent of the substitution. The survey was extended to the manufactured preparations and also to black haw bark and its preparations.

The substitution of *Acer spicatum* as "Cramp bark" for *Viburnum opulus* dates at least as far back as 1892. Here a bark obtained from a manufacturing house was submitted as "probably true Cramp bark, *Viburnum opulus*" to the Research Committee "C" of the Committee on Revision of the United States Pharmacopoeia (Sayre and Denniston 1898). The material, as we know to-day, however, consisted not of cramp bark, *Viburnum opulus*, but of maple bark, *Acer spicatum*.

The description which was given for cramp bark in the Pharmacopoeia VII and VIII, consequently was erroneous. In the latest (IX) revision of the U. S. Pharmacopoeia the description for this product has been omitted altogether, being given correctly now in the National Formulary (1916), with the omission, however, of the term "Cramp bark." Some dealers still list under the name "Cramp bark" or "So-called Cramp bark," the material obtained from *Acer spicatum*, and certain manufacturers appear to favor the application of the term to it.

In this connection it is interesting to note that leading pharmacognosists consider that the name "Cramp bark" applies to *Viburnum opulus* L. and not to *Acer spicatum* (Rusby, 1915, 1916; Kraemer, 1915; and Sayre, 1917). The following excerpts from an article on "Spurious Drugs" by John Uri Lloyd (1915), are of special interest:

"The use of *Viburnum opulus*, cramp bark, dates back to domestic medicine in the past century, its most conspicuous therapeutic introduction, perhaps, being in the Botanic Physician (1844), a work of 210 pages, by Dr. Elisha Smith, to which Dr. John King often referred in the first edition of his Dispensatory (1852). At that time, and for many years afterward, *Viburnum opulus* was employed in but small amounts outside the eclectic school of medicine. There was consequently no difficulty in obtaining, true to name, the moderate amount necessary to supply the demand for the drug. Came next a commercial demand for 'Viburnum,' instigated by proprietary preparations. *Viburnum opulus* was then the only drug recognized under the name 'cramp bark' (see the old American domestic remedy publications), and this demand for 'Viburnum' was abruptly stimulated. *Viburnum opulus* not being commercially very abundant, the drug practically disappeared from the market, the possible supply being insignificant in proportion to the amount required in commerce. It was then inexplicably replaced, under the name 'cramp bark' (which name originally applied to *Viburnum opulus* only), by the bark of *Acer spicatum*, or mountain maple, to which so far as I know, the name 'cramp bark' had not pre-

* Contribution from the Pharmacognosy Laboratory, Bureau of Chemistry, Department of Agriculture, Washington, D. C.

TABLE I.—EXAMINATION OF COMMERCIAL SAMPLES OF CRAMP BARK.

No.	Label.			Identity.	Part used.	Condition.		Quality.
	Popular name.	Scientific name.	Part employed			Physical state.	Wood present.	
1	Cramp bark	*Viburnum opulus*		*Acer spicatum*	Stem bark	Fine powder	Not excessive	Not U. S. P. spurious
2	" "	" "		" "	" "	Very coarse powder	" "	" "
	(so-called)							
3	Cramp bark	" "	Whole bark	" "	" "	Whole	Few small pieces	" "
4	" "	" "	"	" "	" "	"	Considerable	
	(so-called)							
5	Cramp bark	" "		" "	" "	"	Few pieces	" "
6	" "	" "		" "	" "	"	Several pieces	" "
7	" "	" "		" "	" "	Fine powder	Not excessive	" "
8	" "	" "		" "	" "	Chips	Considerable	" "
9	" "	" "		" "	" "	Very coarse powder	Not excessive	" "
10	" "	" "		" "	" "	Small chips	" "	" "
11	" "			" "	" "	Fine powder	" "	" "
12	" "	*Viburnum opulus*	Bark	" "	" "	Chips	" "	" "
13		" "	"	" "	" "	Coarse powder	" "	" "
14	Cramp bark	" "	Bark	" "	" "	Chips	" "	" "
15	" "	" "	"	" "	" "	Fine powder	" "	" "
16	" "			" "	" "	"	Not observed	" "
17	" "			" "	" "	Whole	" "	" "
18		*Viburnum opulus*		" "	" "	"	" "	" "
19	Cramp bark	" "		*Viburnum opulus*	" "	"	Not excessive	U. S. P
20		" "		*Acer spicatum*	" "	Coarse powder	" "	Not U. S. P. spurious
21	Cramp bark	" "		" "	" "	Whole	" "	" "
22	" "	" "		" "	" "	Small chips	" "	" "
23	" "	" "		" "	" "	Fine powder	" "	" "
24	" "	" "		" "	" "	Very coarse powder	Not observed	" "
25	" "	" "		" "	" "	Coarse powder	Not excessive	" "
26	" "	" "		" "	" "	Very coarse powder	" "	" "
27	" "	" "		" "	" "	Coarse powder	" "	" "
28	" "	" "	Bark	" "	" "	Chips	" "	" "
29	" "	" "	"	" "	" "	Very coarse powder	" "	" "
30	" "	" "		" "	" "	" " "	" "	" "
31	" "	" "		" "	" "	Whole	" "	" "
32	" "	" "		" "	" "	Very coarse powder	" "	" "
33	" "	" "		" "	" "	Fine powder	" "	" "
34	" "	" "		" "	" "	Small chips	" "	" "
35	" "	" "	Bark	" "	" "	Chips	" "	" "
36	" "	" "	"	" "	" "	"	" "	" "
37	" "	" "	"	" "	" "	"	Considerable	" "
38	" "	" "		" "	" "	"	Not excessive	" "
39	" "	" "		" "	" "	Very coarse powder	" "	" "
40	" "	" "		" "	" "	Chips	Considerable	" "
41	" "	" "		" "	" "	Fine powder	Not excessive	" "
42	" "			" "	" "	" "	" "	" "
43	" "	*Viburnum opulus*		" "	" "	Chips	Considerable	" "
44	" "	" "		" "	" "	"	"	" "
45	" "	" "		" "	" "	Coarse powder	Not excessive	" "
46	" "	" "	Bark	" "	" "	Chips	Considerable	" "
47	" "	" "	"	" "	" "	Fine powder	Not excessive	" "
48	" "	" "	Bark	" "	" "	Chips	" "	" "
49	Cramp bark (genuine)	" "		*Viburnum opulus*	" "	Coarse powder	" "	U. S. P.
50	Cramp bark or Mountain maple			*Acer spicatum*	" "	" "	" "	Not U. S. P. spurious

TABLE II.—EXAMINATION OF COMMERCIAL SAMPLES OF BLACK HAW.

No.	Label.		Identity.	Part used.	Condition.		Quality.
	Popular name.	Scientific name.			Physical state.	Wood present.	
1	Black haw	*Viburnum prunifolium*	*Viburnum prunifolium* about 82% *Viburnum lentago* " 18%	Root	Whole bark	About 17%	Not U. S. P. excessive wood
2	" "		*Viburnum lentago*	Root	" "	" 1.5%	U. S. P.
3		*Viburnum prunifolium*	*Viburnum prunifolium*	Stem	" "	" 1.5%	"
4	Black haw		*Viburnum lentago*	Bark	Powder	" 1.5%	
5	" "		" "	Root	Whole bark	" 18%	Not U. S. P. excessive wood
6	" "	*Viburnum prunifolium*	*Viburnum prunifolium*	"	Granulated	" 2%	U. S. P.
7	" "	" " "	*Viburnum* (*unofficial species*)	"	Whole bark	" 7%	Not U. S. P. spurious

TABLE III.—ON THE COMPOSITION OF SOME COMMERCIAL VIBURNUM PREPARATIONS.

No.	Label.		Tannin test.	Valerianic acid test.	Identity.
	Common name.	Scientific name.			
1	Fluidextract black haw	Fluidextractum *Viburni prunifolii*	Dark green	Positive	*Viburnum* species
2	" " "	*Viburnum prunifolium*	Medium green	"	" "
3	Black haw	Fluidextract *Viburnum prunifolium*	" "	"	" "
4	" "	" " "	Dark green	"	" "
5	" "	" " "	Light green	"	" "
6	" "	" " "	" "	"	" "
7	Fluidextract black haw	Fluidextractum *Viburni prunifolii*	Dark green	"	" "
8	Black Haw	*Viburnum prunifolium*	Medium green	"	" "
9	" "	Fluidextract *Viburnum prunifolium*	" "	"	" "
10	" "	" " "	" "	"	" "
11	Black haw fluidextract	" " "	" "	"	" "
12	Black haw	Fluidextractum *Viburni prunifolii*	" "	"	" "
13	Fluidextract black haw	*Viburnum prunifolium*	Light green	"	" "
14	Black haw	Fluidextract *Viburnum prunifolium*	" "	"	" "
15	Proprietary A		Very light green		*Viburnum* species
16	Cramp bark	Fluidextract *Viburnum opulus*	Medium green	Positive	*Viburnum* species
17	Viburnum	Viburnum	Light green	"	" "
18	Cramp bark	Fluidextract *Viburnum opulus*	Medium green	"	" "
19	Fluidextract cramp bark	Fluidextractum *Viburni opuli*	Deep blue	Negative	*Acer* species
20	" " "	Viburnum opulus	" "	"	" "
21	Cramp bark	Fluidextract *Viburnum opulus*	" "	"	" "
22	" "	" " "	" "	"	" "
23	" "	" " "	" "	"	" "
24	Fluidextract cramp bark	Fluidextractum *Viburni opuli*	" "	"	" "
25	Cramp bark	Fluidextract *Viburnum opulus*	" "	"	" "
26	" "	" " "	" "	"	" "
27	Fluidextract cramp bark	Fluidextractum *Viburni opuli*	" "	"	" "
28	" " "	Viburnum opulus	" "	"	" "
29	Cramp bark	Fluidextract *Viburnum opulus*	" "	"	" "
30	Proprietary B		Light blue		*Acer* species

viously been applied." * * * * In this connection we might emphasize the fact that true "cramp bark" is unquestionably *Viburnum opulus*, but undoubtedly the bark of *Acer spicatum* has long been about the only "cramp bark" on the general market. "As a historical record it may be added that the early eclectics used, under the names 'Cramp bark' and 'High cranberry' the bark of *Viburnum opulus* only, a shrub native to the East, and well known to them."

It was pointed out by Farwell (1913) that the bark of the mountain maple (*Acer spicatum*) was being substituted for cramp bark (*Viburnum opulus*). To verify this statement and to determine the extent of such substitution commercial samples of "cramp bark" were collected throughout the United States in 1915 and examined as to their identity.[1] The results of our observations are incorporated in Table I, from which it may be seen that of the fifty samples examined, forty-eight proved to consist of the bark of *Acer spicatum* L. The other two were derived from *Viburnum opulus* L. Zufall (1915) and Rusby (1916) also state that samples of "cramp bark" proved to be maple bark and F. Beringer, as well as H. Kraemer reported similar findings to meetings of local branches of the American Pharmaceutical Association.

A similar examination of commercial samples of bark of *Viburnum prunifolium* showed that six were derived from the official species, *Viburnum prunifolium* L., or *Viburnum lentago* L., while one was derived from an unofficial *Viburnum species* (Table II). Both tables, furthermore, show that samples bearing the same label were found in all degrees of physical state, from whole to finely powdered bark, some devoid of wood, others containing very considerable amounts.

In view of the fact that substitution was observed and especially that such a large percentage of the samples of cramp bark proved to be spurious, a survey was made of *Viburnum* preparations in general on the market, in order to ascertain if similar conditions prevailed. Samples were collected throughout the United States in 1916, and examined as to their identity. The results of this examination are incorporated in Table III. It will be noted that in every instance preparations of *Viburnum prunifolium* were apparently true to label. On the other hand, of the fifteen samples supposed to be made from *Viburnum opulus*, only three were found to give a positive test for *Viburnum*. The other samples gave the reaction for *Acer* species, and in view of our findings regarding commercial "cramp barks," were very probably prepared from the bark of *Acer spicatum*. In this connection it is interesting to note that recent preparations of a certain manufacturer gave positive tests for *Viburnum*, whereas earlier preparations of the same manufacturer gave a reaction for *Acer*, thus indicating that correction of the error had been made in one instance at least, since the attention of the trade was called to the substitution of *Acer spicatum* for *Viburnum opulus*.

On account of the great extent of the substitution of *Acer spicatum* for *Viburnum opulus*, it seems advisable to again call the attention of the trade to this fact and to point out easy means of differentiation between the two by simple chemical tests, as well as by macroscopic and microscopic characteristics. (See S. R. A. Chem. 20, Item 216, 1917.)

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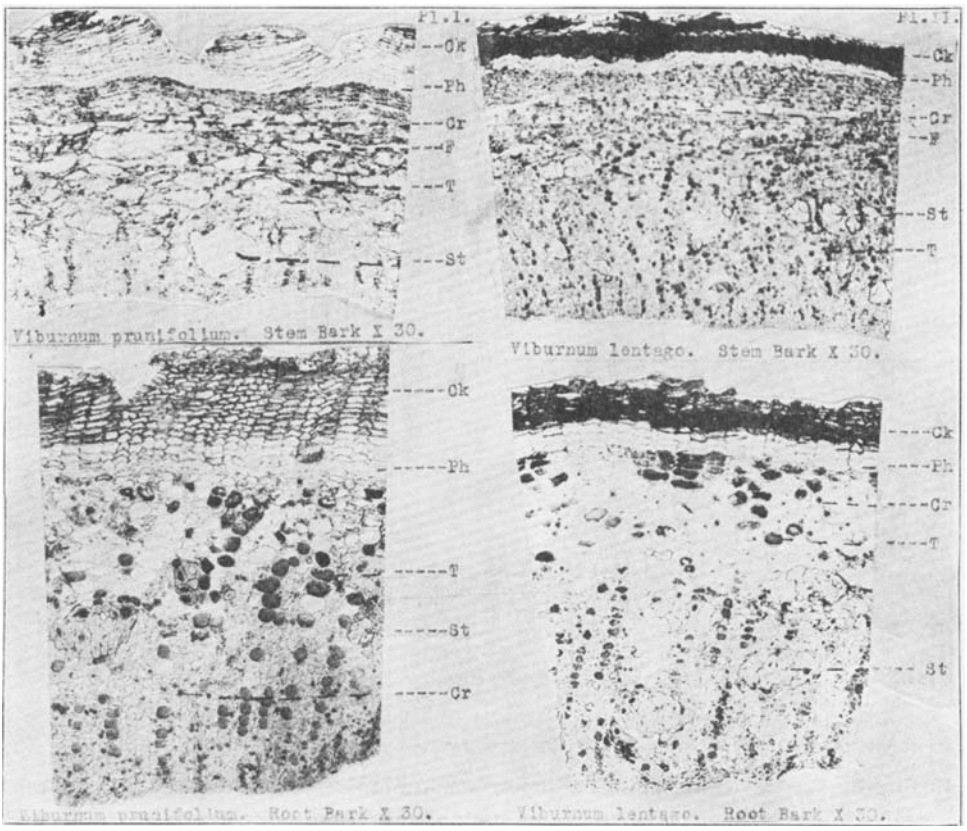
[1] In addition to authentic material collected by ourselves, authentic samples were obtained from C. J. Sargent, F. M. Crayton, and especially O. A. Farwell, to whom we wish to express our appreciation for their assistance.

The characteristics of the barks of *Acer spicatum* and of the official *Viburnum* species have been pointed out before in greater or less detail. Denniston (1898) has made a very thorough study of a number of American *Viburnums*, including *Viburnum opulus* L., *Viburnum prunifolium* L., and *Viburnum lentago* L., and has given accurate descriptions of the barks. Unfortunately, however, the material supplied to him as *Viburnum opulus* was in fact *Acer spicatum*, and his descriptions under the caption "*Viburnum opulus*" covers the main characteristics of mountain maple bark. Farwell (1913) described the morphology of the leaves as well as the histology of the bark of the American-grown *Viburnum opulus*, which he considers as *Viburnum opulus* L. var. *americanum Aiton*. We have as yet no evidence of any anatomical difference between the bark from *Viburnum opulus* and this variety *americanum*. Zufall (1915) suggested a revised official description of *Viburnum opulus*. His statement concerning the absence of oxalate crystals in maple and of fibers in cramp bark, as we understand from a personal communication, refers to the absence of oxalate rosettes in the one bark and the usual lack of fibers in the other. The author thus agrees with our findings.

The National Formulary IV (1916) gives a satisfactory description of *Viburnum opulus*, although omitting the name "cramp bark" altogether. The United States Pharmacopoeia VIII (1905) gives these names synonymously, but includes an erroneous description which applies to mountain maple instead of true cramp bark. Since these descriptions are easily accessible and generally satisfactory we consider it unnecessary to repeat them in detail. We desire only to point out the most striking characteristics useful for the identification and differentiation of the barks of the above-named species.

The bark of *Acer spicatum* is generally thicker and darker gray than that of *Viburnum opulus*, and usually has some woody tissue attached, while in *Viburnum opulus* the presence of attached wood is less frequent. The fracture of the bark of *Acer spicatum* is tough and very fibrous, due to the presence of large and numerous groups of long sclerenchyma fibers both in the stem and root bark. The fracture of *Viburnum opulus* is short and weak, since sclerenchyma fibers are usually absent in the secondary bark and are few and scattered in the primary cortex of the young bark. The bark of *Viburnum opulus* contains very numerous rosette crystals, scattered throughout the tissue of the inner bark, while *Acer spicatum* contains numerous prismatic crystals which occur in crystal fibers, generally accompanying groups of bast fibers. These are seen to best advantage in longitudinal radial sections. The root and stem barks of *Acer spicatum* are very similar in structure, while the root bark of *Viburnum opulus* differs from both of these and from its own bark as well, in showing almost an entire absence of stone cells or sclerenchyma fibers.

Viburnum prunifolium and *Viburnum lentago* both have short, weak fractures. The stem barks resemble each other very closely, but in the samples we examined the former had fewer and smaller groups of sclerenchyma fibers in the primary cortex and the groups of stone cells in the secondary bark were generally larger and more numerous. Their dimensions varied between 290–680 microns by 170–290 microns in cross section. The root barks also had a strong similarity to each other, but differed from the stem barks in that no sclerenchyma fibers were observed.

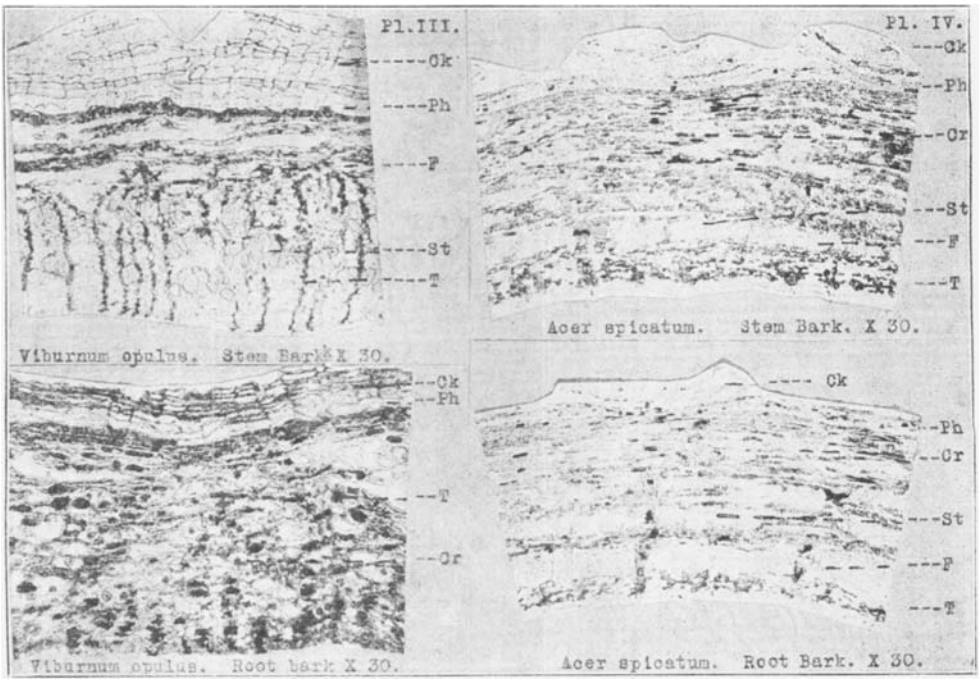


Viburnum prunifolium. Plate I.—Ck, cork; Ph, phellogen; Cr, calcium oxalate, crystals; St, stone cells; T, tannin cells; F, bast fibers. Plate II.—*Viburnum lentago*, abbreviations apply as in preceding.

The tannins are for the most part similarly located in all of the *Viburnum barks* described above. They occur chiefly in the medullary rays, but are also found scattered here and there in the parenchyma cells of the outer cortex. In the case of the *Acer* bark the latter type of distribution predominates. While tannins do occur also in the medullary rays they are possibly less numerous than in the case of the Viburnum barks, and are not quite so prominent. (See Plates I to IV.) The tannins of *Acer spicatum* can be distinguished from those of *Viburnum* species by their color reaction with freshly prepared one-tenth percent ferric chloride solution.

It was pointed out by one of us (Viehoever) at the October 1916, meeting of the American Pharmaceutical Association that both *Viburnum opulus* and *Viburnum prunifolium* contain tannins which give a green color or precipitate with iron salts, whereas *Acer spicatum* contains a tannin which gives a blue tannin or precipitate with iron salts. The reaction is given by both ferrous and ferric salts, but *freshly prepared* ferrous sulphate solution was selected because this solution possesses very little interfering color. This is advantageous, especially in the case of *Viburnum* preparations, where the color obtained is sometimes quite delicate.

In the case of the whole barks, the test was carried out by applying a drop



Viburnum opulus. Plate III.—Ck, cork; Ph, phellogen; Cr, Calcium oxalate, crystals; T, tannin cells; E, bast fibers; St, stone cells. Plate IV.—*Acer spicatum*, abbreviations apply as in preceding.

of dilute (1–1000) ferric chloride or ferrous sulphate solution directly to the inner surface of the bark. In the course of a few minutes a distinct green color appeared in the case of *Viburnum opulus* and *Viburnum prunifolium*, whereas a very deep blue to bluish black color developed in the case of *Acer spicatum*. The ground barks were tested by applying the reagent to the powder and examining in doubtful cases under the microscope.

Another simple and striking test is the red lignin reaction obtained in the case of maple bark, if a drop of phloroglucin-hydrochloric acid (phloroglucin 0.1 Gm., alcohol and concentrated hydrochloric acid 8 Cc., each) is applied to the inner side of the bark due to the numerous lignified sclerenchyma fibers in the bark. Some wood fragments are often attached to the barks; these have to be removed before making the test, which can, of course, also be applied to a section, preferably a longitudinal one. The phloroglucin-hydrochloric acid should be comparatively fresh and not too deeply colored.

The hydrochloric acid contained in the phloroglucin solution will furthermore develop, especially in the case of freshly collected *Viburnum* bark, the odor of valerianic acid, which is absent in maple bark.

In applying the tannin test to pharmaceutical preparations, the procedure was as follows:

Ten mils of the sample containing alcohol was diluted with about three volumes of water and shaken out with about 15 mils of ether. The ethereal layer was filtered and shaken gently in a test tube with an equal volume of water containing two drops of a *freshly prepared* saturated ferrous sulphate solution. A green color in the lower aqueous layer indicated a *Viburnum* species; a blue color indicated an *Acer* species.

In the case of preparations containing very small amounts of *Viburnum* accompanied by large amounts of interfering substances, it was necessary to evaporate a larger volume to dryness, take up with about 15 mils of 95 percent alcohol, dilute with water and proceed as usual.

St. John (1916) has pointed out that better results are obtained by shaking out with ether than with petroleum ether. We account for this by the fact that, although the tannins are insoluble in both petroleum ether and absolute ether, when a hydro-alcoholic extract is shaken out with ether, enough alcohol and water is taken up by the ether to carry along with it some of the dissolved tannins which are then tested for with the ferrous sulphate solution.

To confirm the results of the tannin test as applied to the pharmaceutical preparations, they were further tested in the following manner for the presence of valerianic acid, which is yielded by both *Viburnum opulus* and *Viburnum prunifolium* (Wehmer, 1911):

A small portion (several mils) was made alkaline with dilute sodium hydroxide, boiled to expel most of the alcohol, acidified with dilute sulphuric acid and warmed. In every instance where a *Viburnum* species had been indicated by the tannin test, the characteristic odor of valerianic acid developed.

In order to confirm the presence of valerianic acid, in one instance a larger amount of the preparation was neutralized with sodium bicarbonate, evaporated on a steam bath, acidified with dilute sulphuric acid and distilled with steam. The distillate was saturated with salt, shaken out with ether and the ethereal solution evaporated on the steam bath until the odor of ether was no longer perceptible. The remaining liquid was distilled and fractionated. It consisted largely of isovalerianic acid boiling at 170° and also probably contained a very small amount of normal butyric acid (b. 162–3). The isovalerianic acid was identified further by the microchemical characteristics of the copper, zinc and mercury salts (Behrens, *Mikrochemischen Analyse*, 1897). For this purpose the fraction containing mainly isovalerianic acid was treated with varying concentrations of solutions of copper acetate, mercuric nitrate, and zinc nitrate, the latter yielding the best results. When a small amount of alcohol was added and a too rapid evaporation was prevented by carrying out the reaction in a moist chamber, the crystallization was facilitated. Notwithstanding this, considerable difficulty was experienced in obtaining the copper salt, probably due to the presence of a small amount of butyric acid which is known to interfere with this reaction. While characteristic crystals were obtained with all three reagents, the least difficulty in obtaining satisfactory results was experienced with the zinc. The characteristics of the crystals obtained are shown in Fig. 1. The butyric acid was indicated by the development of an odor resembling pineapples,

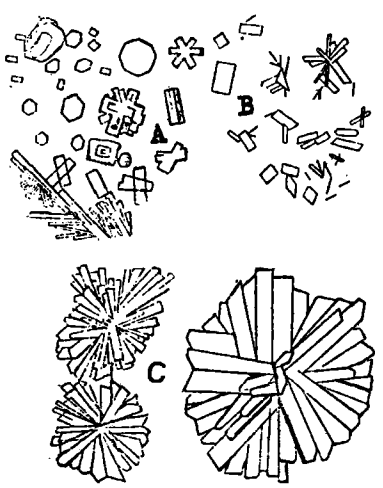


FIG. 1.

A. Copper valerianate. × 130.
B. Zinc valerianate. × 130.
C. Mercury valerianate. × 130.
After Behrens.

due to ethyl butyrate, when a drop of the first fraction was heated with several drops of ethyl alcohol and one drop of concentrated sulphuric acid. The material at hand was insufficient to prepare salts of the acids for further macroscopic confirmation.

SUMMARY.

(1) The bark of mountain maple (*Acer spicatum*) was found to be almost entirely substituted for true cramp bark (*Viburnum opulus*).

(2) All samples of black haw (*Viburnum prunifolium* or *Viburnum lentago*) proved to be genuine with the exception of one, obtained from a non-official *Viburnum* species.

(3) The preparations of black haw were made from *Viburnum* barks, while those of cramp bark were mostly manufactured from *Acer* species.

(4) The tannins in the barks give different color reactions with iron salts; blue in the case of *Acer* and green in that of the *Viburnum* species. These reactions can be used to distinguish the barks as well as their preparations.

(5) The tannins are distributed in the parenchymatic tissue, but can most easily be seen in the medullary rays.

(6) Maple bark can furthermore be readily distinguished from the Viburnum barks by the intense red coloration when the inner bark is treated with phloroglucin-hydrochloric acid solution; in the case of *Viburnum* barks, more than a faint reaction, if any, is rarely obtained.

(7) Among the differentiating tests of interest are those which were used to obtain and identify valerianic acid, yielded by the *Viburnum* barks but not by the *Acer* barks.

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S. R. A. Chem., 20, Item 216, p. 60.

WILD ANTHEMIS—A POSSIBLE MATRICARIA ADULTERANT.*

BY C. W. BALLARD.

Roman Chamomile or *Anthemis nobilis* L., was official in a previous edition of the U. S. Pharmacopoeia and this authority specified that the drug be obtained from cultivated plants. The product of wild growing plants contains more volatile oil and bitter principles. It is probably more active than the cultivated product. As the drug is used almost entirely as a carminative, the milder and more agreeable flavor of the cultivated flowers has resulted in their displacing wild anthemis. It is recorded that anthemis infusions will, in some instances, cause nausea and it is probable that if wild anthemis were used in their preparation this undesirable property will be more marked.

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* Prepared for Scientific Section, A. Ph. A., Chicago meeting, 1918.