point in the case of Benzene-Alcohol. Sodium carbonate, it was shown, is appreciably titrated in the Alcohol methods to an extent determined by the solubility of sodium carbonate at the titrating temperature. The Barium Chloride Method gave results independent of the carbonate concentration, which checked the corrected results of other methods.

A comparison was made between the four methods, averaging the results on 12 samples of soap chips containing 12% moisture. It was shown that by making appropriate correction for carbonate solubility in each solvent and the temperature effect of the end-point, all methods are in substantial agreement. The following table shows the comparison between results by the proposed Benzene-Alcohol Method and the Standard A. O. C. S. Method.

	% Na_2O	
	Benzene-Alcohol	Standard A.O.C.S.
	.024	.046
Correction for Na_2CO_3 solubility	—.003	—.013
Correction for Titration at 71° C.	.015	...
Corrected Value Free Caustic Alkali	.036	.033

Hence, if the Benzene-Alcohol Method is used, the results of the Standard A. O. C. S. Method are obtained by adding .012% Na_2O to the % Na_2O calculated from the titration. This additive factor will vary slightly with variations in the moisture of the soap and water added through titration, the quantitative effect of which was given.

A study was made of the effect of indicator, water, temperature, and amount of sample on the titrated free caustic alkali. The addition of water increases the free caustic alkali titrated in a commercial chip about .001% Na_2O per cc., using 12.4 g. of soap and 175 cc. of solvent. An increase of 10° increases the free caustic alkali titrated by .0031% Na_2O in a carbonate-free soap and .0055% Na_2O in a commercial soap.

Preliminary study of the titration of a pure neutral soap indicates that soap undergoes some hydrolysis or solvation in the presence of alcohol and water and that this effect is greater the higher the temperature. The absolute value for free alkali is, according to our present concepts, arbitrary. It is suggested for practical purposes that until the absolute value of free alkali in soap is established experimentally, that a soap be called neutral when 12.4 g. carbonate-free, dissolved in 150 cc. of 95% alcohol at 70° C. containing one cc. of phenolphthalein indicator, is colorless, or assumes a slight pink upon addition of one drop of .1N NaOH.

ELDERBERRY SEED OIL (*Sambucus canadensis* L.): PRELIMINARY COMMUNICATION*

By H. A. SCHUETTE and JOHN W. BROOKS

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FOR centuries man has found in the genus Sambucus, of which there are approximately twenty different species, one source or another of satisfying a medicinal, a pharmaceutical or a domestic need. An infusion of the leaves has been an old-fashioned household remedy (Fliedertee) among German families since the Middle Ages. The flowers, although perhaps almost devoid of active medicinal virtue and possessing no real curative properties, besides having been esteemed for their diaphoretic and diuretic properties, in infusion, ointment or poultice have been applied to old sores, blisters and hemorrhoides. The young shoots and leaves of one species not only share the diuretic, and in larger doses, the purgative properties of the inner bark but are also effective, it is said, as a repellant for flies. The American Indians sought the ripe berries for their pigment, later chemists discovered that the juice had possibilities as an indicator, and man from time immemorial seems to have appreciated the fact that a very palatable spicy wine can be made from them.

The fatty oil, however, remains alone of those products of the elder shrub for which man—except in times of national emergency as when Germans and Austrians cast contemplative eyes upon this plant as a war-time source of fat—has not at one time or another found an alimentary or a medicinal use. Although it was hinted some seventy-five years ago[1] that this oil bears a similarity to linseed oil, an observation which was eventually verified experimentally by Zellner[2] with respect to the seed oil of *S. racemosa* L., and by Thoms[3], in so far as that he demonstrated the presence herein of acids representative of three types of unsaturation, yet the possibilities of adapting it to some useful technical ends appear still to be latent.

The literature on the characteristics and the component acids of the fatty oil is not large nor is the treatment of the subject comprehensive. It covers two types of oil somewhat unlike in properties because of differences in their respective origins: fruit flesh and seed. Relevant only to this communication are the reports by Zellner[2] and Thoms[3] on *S. racemosa* L.

In view of the wide-spread distribution in this country of another member of this genus, the habitat of which was so charmingly described by Peter Kalm, the Swedish botanist and traveler, *ca* 1749 ("Travels"), who said of it

" The American elder, *S. canadensis* Linn., and the wild vines, only appear in places where the ground has been cultivated, as if they were desirous of being the companions of man,"

it is indeed strange that the oil of this species to date has not been investigated.

This communication marks the beginning of an attempt to fill this gap in the literature. It also records data which make possible comparisons with those pertinent to other species from the standpoint of genetic relationships of the plant source and biochemical similarities of the oil. Presented herein are (1) data showing yield of fatty oil with respect to menstruum employed in their recovery, (2) the simpler physical and a few chemical constants of the oils and their fatty acids, and (3) a comparative tabulation of the characteristics of the fatty oils of related species of *Sambucus*.

*A paper presented at the Fall Meeting, A.O.C.S., Chicago, October 8-9, 1936.

The oils which were used in this study were extracted with chemically pure solvents in the fall of 1935 from seeds recovered from the elderberry crop of the previous year. Large, all-glass Soxhlet type intermittent extractors[4] were used for this purpose. Because of the oily nature of the seeds, it was found necessary to make the extraction a two-step operation for maximum and speedy recovery of the oil: a preliminary extraction of the whole seed, then comminution and re-extraction. The solvent was removed from the combined extracts at reduced pressure and in the presence of carbon dioxide. Filtration through asbestos completed the operation. Yields of fatty oil varied from 28 per cent with petroleum ether to 31.9 per cent with ethylene dichloride. Extremes in pigmentation were also represented by these solvents, or citron yellow and reddish brown for their respective products. Oil yields follow (Table 1):

TABLE I

Oil Content of Elderberry Seeds (*S. canadensis* L.) as Affected by Choice of Menstruum

Menstruum	Yield per cent	Color* (Lovibond system) yellow	red
Petroleum ether	28.08	2.5	0.4
Ethyl ether	28.81	11.1	1.2
Benzene	29.51	9.7	1.4
Carbon disulfide	29.68	9.8	1.3
Chloroform	29.74	6.2	0.9
Acetone	30.56	12.9	2.1
Carbon tetrachloride	30.75	7.9	0.9
Ethylene dichloride	31.91	29.8	3.9

*Dilution of oil: fifty per cent solution in petroleum ether.

Comparison of these yields with those reported from foreign sources for other species of Sambucus shows that the oil content of the seeds of this genus is approximately of the same order of magnitude. Specifically, the following data have been recorded:

Species	Solvent	Oil Content of seeds per cent	Authority
S. racemosa L.	benzol	27.36	Zellner[2]
	ethyl ether	26.55	Muth[5]
S. nigra L.	ethyl ether	29.51	Muth[5]
S. nigra var. *laciniata hortensis*	ethyl ether	28.73	Muth[5]
S. ebulus L.	ethyl ether	25.56	Muth[5]

On determining the physical constants of the several oils the following minimum and maximum values were found for each: specific gravity at 20°, 0.9320 and 0.9523; refractive index at 20°, 1.4781 and 1.4795; relative viscosity, 26.4 and 40.8 poises; and surface tension, 33.8 and 36.6 dynes per sq. cm. Complete data are recorded in Table II.

TABLE II

Physical Characteristics of Elderberry Seed Oil

Menstruum employed in recovery of oil	Specific gravity 20°/20°	Refractive index 20°	Viscosity absolute poises 25°	Viscosity relative 25°	Surface tension dynes cm² 25°
Petroleum ether	0.9351	1.4781	0.299	33.5	35.4
Ethyl ether	0.9352	1.4783	0.325	36.4	35.4
Benzene	0.9494	1.4795	0.236	26.4	34.0
Carbon disulfide	0.9329	1.4791	0.302	35.4	33.8
Chloroform	0.9523	1.4781	0.285	31.9	36.1
Acetone	0.9413	1.4785	0.318	35.7	33.9
Carbon tetrachloride	0.9336	1.4789	0.285	38.2	36.6
Ethylene chloride	0.9320	1.4794	0.364	40.8	35.6

The only available comparable data on other species are those reported by Zellner[2] for *S. racemosa* with respect to a benzene-extracted oil. A close similarity is apparent, viz:

Characteristics	*S. canadensis* L.	*S. racemosa* L.
Specific gravity	0.9494 20°	0.9344 15°
Refractive index 20°	1.4795	1.4850
Iodine number	162 (Wijs)	162 (Hübl)
Saponification number	186.5	190.8
Unsaponifiable matter	1.68	0.61

The most striking differences in the observed chemical properties of these oils is the 2.5-point variation in the amount of unsaponifiable matter which they contain (min. 1.48, max. 4.00 per cent.). Saponification values offer nothing unusual except in the case of the chloroform-extracted oil. Inasmuch as this value is so far out of line with the others, it is to be regarded as tentative until the oil of a new crop, obtained by similar means, can be secured for verification of this figure. A minimum iodine number of 161.9, and a maximum of 171.7 was noted. The order of magnitude of this number definitely assigns this oil to the drying oil class. Complete data for the oils are recorded in Table III.

Confirmation[6] of the drying qualities of this oil was obtained by spreading thin films of it on glass plates approximately three by eight inches in size supported at a 30-degree angle. Using linseed oil as a control, it was found that the time necessary for the product of the petroleum ether extraction to dry was shorter than that required by

TABLE III

Constants of the Fatty Oil and Fatty Acids of Elderberry Seed Oil.

Menstruum employed in recovery of oil	Iodine number (Wijs)	Fatty Oil Saponification number	Unsaponifiable matter, per cent	Refractive index 20°	Fatty Acids Iodine number (Wijs)	Thiocyanogen number
Petroleum ether	171.15	188.1	1.48	1.4712	175.0	98.90
Ethyl ether	171.71	191.2	1.96	1.4700	175.2	109.95
Benzene	161.98	186.5	1.68	1.4701	177.3	99.10
Carbon disulfide	171.28	195.9	1.83	1.4701	179.4	104.83
Chloroform	163.91	257.0	4.00	1.4707	173.0	107.20
Acetone	169.98	192.2	2.01	1.4701	176.4	108.70
Carbon tetrachloride	170.04	191.1	1.93	1.4696	175.3	105.50
Ethylene dichloride	167.76	195.7	2.20	1.4702	177.6	106.95

the former, or about three days against four for the other. It was also observed that the nature of the menstruum employed in recovery of the oil apparently exerts some influence on the drying qualities of the latter. However, in each case a hard, nearly colorless, transparent film, which was not sticky to touch, was formed.

SUMMARY

The seed oil of the American elder (*S. canadensis* L.), like that of other members of this genus on which data are extant, is a drying oil. In so far as present information reveals, it appears that genetic relationships are here qualitatively reflected by biochemical similarities in the fatty oils. Yields of the latter depend upon the nature of the menstruum employed.

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4. F. C. Oppen, *Ind. Eng. Chem. Anal. Ed.*, **8**, 110 (1936).

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6. H. C. Loeffler, B. S. thesis 1934, University of Wisconsin.

Report Of Color Committee A. O. C. S. For 1935-1936*

THE work of the Color Committee for 1935-1936 was not quite completed in time for a report at the spring meeting, and it was agreed at that time, that the same members would complete the work and make a report, since considerable expense would be involved by including new members.

Your committee has examined the "Stevenson Colorimeter." This instrument was described in a paper presented before the American Oil Chemists Society's fall meeting of 1935, and published in the January, 1936, issue of "Oil and Soap."

The colorimeter follows the previously adopted specification for tintometers, except that provision is made for holding the color glasses in a dust-proof magazine.

Each member of the committee used the colorimeter in his laboratory and compared its operation with his present instrument.

With the exception of one or two details, the colorimeter was found to be satisfactory. These exceptions are as follows:

1. Provision should be made for removing the oil tube more easily.
2. A magazine oil tube holder may be desired by some operators.

Both of these changes could be worked out by individual makers without changing the color reading characteristics of the instrument.

The committee recommends the approval of the Stevenson Colorimeter for official use. In order to provide for such approval in the methods, it is recommended that the paragraph titled "Tintometer," on page 16d of the methods of analysis, be changed to read as follows:

> Colorimeter—An enclosed lightproof box containing an approved light bulb and magnesia block, and equipped with a device for holding the color tube and color glasses in such a manner that light passing up through the oil and also light passing through the color glasses can be observed simultaneously through an eyepiece. The details of the various parts and their arrangement must conform to the approved design for the manual type or for the magazine type, which can be obtained from the secretary of the American Oil Chemists Society.

A copy of the specifications and tracings for blue prints of the colorimeter will be filed with the secretary as a part of this report.

The committee wishes to thank Mr. H. B. Stevenson, The Procter & Gamble Company and the W. H. Simmons Mfg. Co. of Cincinnati, Ohio, who have spent a large amount of time and money on perfecting the design and manufacture of this colorimeter.

COLOR COMMITTEE

A. O. C. S.

L. A. Spielman
H. C. Dormitzer
G. Worthen Agee
H. B. Stevenson
H. P. Trevithick
J. Lappen
G. G. Grant, Chairman

Editor's Note: No official action taken by the society.

*As presented at the Fall Meeting, A.O.C.S., Chicago, October 8-9, 1936.

ERRATA

"The Recovery of Crude Glycerin," by Oscar H. Wurster. OIL & SOAP, **13**, 246-53.

Page 246, column 1, under "Treatment of Spent Soap Lyes." After line 5, insert the line "chloride and hydrochloric acid. The ferric." In line 14 of the same paragraph, the formula should appear as $FeCl_3.6H_2O$). In column 3 of the same page, next to the last paragraph, the phrase "and it may be less" should be omitted (lines 7 and 8).