TABLE III.—COTTONSEED OIL.
Composition of the Saturated Acids.

	G.	%.	Original oil. %.	Glycerides in original oil. %.
Myristic acid	1.47	1.40	0.32	0.34
Palmitic acid	87.10	83.06	19.10	20.04
Stearic acid	8.64	8.24	1.90	1.98
Arachidic acid	2.56	2.44	0.56	0.58
Oleic acid	5.10	4.86	0.51	0.53
Linolic acid			0.61	0.64
Total	104.87	100.00	23.00	24.11

Table III gives, in Col. 2, the percentage composition of the saturated acids, in Col. 3, the percentages present in the original oil, and in Col. 4, the percentages of glycerides in the original oil.

Summary.

A study has been made of the composition of a sample of cottonseed oil, the results of which are given below:

COMPOSITION OF COTTONSEED OIL.

		%.
Glycerides of	Myristic acid	0.3
	Palmitic acid	20.0
	Stearic acid	2.0
	Arachidic acid	0.6
	Oleic acid	35.2
	Linolic acid	41.7
	Total	99.8

WASHINGTON, D. C.

[CONTRIBUTION FROM THE ESSENTIAL OILS LABORATORY, DRUG DIVISION, BUREAU OF CHEMISTRY.]

THE COMPOSITION OF OIL OF CHENOPODIUM FROM VARIOUS SOURCES.

BY E. K. NELSON.

Received February 28, 1920.

Since the oil of chenopodium, or American wormseed, has become the standard remedy for hookworm disease, the demand for it has increased considerably. In the producing region, Carroll county, Maryland, there are at least 7 distilling plants in operation during the season.

During October, 1919, authentic samples were collected by the author from 5 of these distilling plants, for the purpose of studying the variations in the product.

As pointed out by Schimmel and Co.,[1] the distillation of chenopodium

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[1] Report of Schimmel and Co., April, 1908.

oil requires special precautions, for the reason that ascaridole, the valuable and chief ingredient, is unstable, and is decomposed gradually on boiling with water. Consequently, the distillation must be carried on rapidly with steam at a good pressure, the condenser kept warm, and the warm distillation water separating from the oil in the receiver, discarded.

The examination of these samples comprised a determination of their physical constants and an estimation of the amount of ascaridole present in them. No difficulty was encountered in obtaining ascaridole of constant boiling point by fractionating oil of chenopodium under diminished pressure. This is contrary to the findings of Hamilton and Hall.[1] Evidently these investigators either did not have a true chenopodium oil, or, as seems more probable, allowed the oil to become overheated in the bath, thus causing decomposition and loss of part of the ascaridole.

As pointed out by Schimmel and Co. and by the writer, ascaridole suffers a molecular rearrangement when heated to 150°. In carrying out a fractional distillation of the oil, it is, therefore, necessary to maintain the bath at as low a temperature as possible, never allowing it to reach 150°. Most of the distillations involved in the work herein described were carried out at 3 to 6 mm. pressure. During the distillation of the ascaridole fractions, a bath temperature of 115°, and during that of the terpene fraction, one of 80°, was found sufficient to maintain an even distillation.

The first distillation gave a very good separation of the terpenes from the ascaridole, and only 2 fractionations were required to yield ascaridole of constant boiling point. The estimation of ascaridole in this way gives results somewhat low, as it is difficult to recover all of the ascaridole from the lower fraction.

When chenopodium oil is properly made the residue on fractionation will be small, but if it has been manufactured with low pressure steam and without the precautions above mentioned, the percentage of ascaridole and the specific gravity will be low and an excessive distillation residue, which is evidently a product of the decomposition of ascaridole, will be found.

In the samples collected by the writer in the producing district, the highest distillation residue was only 2.3%, and in 8 samples experimentally distilled with very low pressure steam the distillation residues amounted to from 7 to 25%, and the ascaridole content was very low. The amount of distillation residue, therefore, gives an idea of the care with which the oil has been made.

The distillation residue appears to consist in part of water-soluble, glycol-like substances, resulting from the hydration and rearrangement of the ascaridole molecule. This indicates that during a protracted

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[1] *J. Pharmacol. Exper. Therapeutics*, 11, 231 (1918).

distillation with low-pressure steam a part of the ascaridole must be decomposed and the soluble products in part pass away in the condensation water.

A very simple test of the purity of the ascaridole fractions was found in the readiness with which a few drops will ignite when poured on an electric hot plate heated to about 250°.

In addition to the authentic samples collected by the writer, 7 in number, the following specimens were also examined.

An oil from chenopodium grown and distilled in Buitenzorg, Java, received through the Bureau of Plant Industry.

Two samples distilled from wild chenopodium growing around Orlando, Florida, one from dried and the other from green plants. These were also obtained from the Bureau of Plant Industry, having been produced under the supervision of Mr. H. L. Funk of that Bureau.

One sample distilled from wild plants in the Eastern United States, transmitted by the International Health Board.

ANALYTICAL DATA ON OIL OF CHENOPODIUM.

Sample.	$d\frac{25°}{25°}$.	$[\alpha]_D$ (100 mm. tube).	Solubility in 8 volumes of 70% alcohol.	Ascaridole (% by vol.).		Opt. rotation of ascaridole.	Dist'n residue. %.
				By dist'n %.	Calc from d. %.		
Samples Collected in Carroll Co., Md., Oct., 1919.							
K_1	0.9758	—4.25°	Complete	74	81.5	—2.1°	2.3
K_2	0.9792	—3.9°	Complete	77	83.7	—2.15°	1.1
D	0.9713	—5.5°	Complete	75	78.7	—3.05°	1.1
E_1[a]	0.9538	—6.2°	Complete	60	70.0	—1.65°	1.7
E_2	0.9592	—6.2°	Complete	65	71.0	—2.5°	1.1
Bo	0.9783	—3.6°	Complete	76	83.1	—2.0°	0.9
Br	0.9564	—6.5°	Complete	64	69.3	—2.45°	1.4
Oil from Chenopodium Grown and Distilled in Java.							
	0.9762	+0.25°	Complete	70	81.8		1.3
Oil from Wild Chenopodium, Orlando, Florida.							
From dry plants	0.9148	—7.75°	Not completely soluble	41	43		2.2
From green plants	0.8940	—10.25°	Not completely soluble	26	30		2.4
Oil from Wild Plants, Eastern U. S.							
	0.8922	+1.65°	Not completely soluble	20	28.8	—4.0° (α_D of terpenes + 4.15°).	
Samples Returned from Brazil, Over a Year Old.							
A	0.9628	—5.0°	Complete	68	73.4		2.7
B	0.9645	—7.5°	Complete	61	74.4		7.8
C	0.9470	—6.45°	Slightly turbid	56	63.4		2.8
D	0.9589	—6.6°	Slightly turbid	62	70.9		3.1
6	0.9730	—5.33°	Complete	72	79.8		9.1

[a] Sample of first distillation of the season.

Five oils of American origin, which had been used by the International Health Board in treating hookworm disease in Brazil. These oils were at least a year old and the results are interesting as showing the keeping qualities of the oil under varying conditions. It will be noted that the distillation residues, while higher than those found in fresh oils, are not excessive.

The United States Pharmacopeia (IX) specifies that oil of chenopodium should be soluble in 8 volumes of 70% alcohol, should have a specific gravity of 0.955 to 0.980 at 25°, and an optical rotation varying between —4° and —10° in a 100 mm. tube at 25°.

An approximate check upon the ascaridole estimation was obtained by calculating the ascaridole content of each oil from its density, assuming that the density of the terpenes is 0.8466 and that of the ascaridole 1.005, and that no other constituents are present. The resulting figure is influenced, of course, by any variations in the density of the terpene mixture, and by the amount of distillation residue, which consists, of course, neither of terpenes nor of ascaridole. The figures thus obtained will be seen to run roughly parallel with those of the direct estimation of ascaridole by distillation.

Terpenes of Chenopodium Oil.

Schimmel & Co.[1] have reported the presence of *p*-cymene in chenopodium oil, but the optical activity of the terpenes and their sensitiveness toward oxidizing agents indicate the presence of other hydrocarbons.

The terpenes were separated from the authentic samples collected in Maryland, and were found to boil at 176–178° under atmospheric pressure, after rectifying over sodium. The optical activity was $[\alpha]_D =$ —15.9° (100 mm. tube).

A small yield of a nitrosite, melting at 155°, was obtained. This is probably the nitrosite of α-terpinene, though oxidation experiments designed to show the presence of that terpene, as well as the presence or absence of γ-terpinene, gave no satisfactory results.

l-Limonene was shown to be present by the formation of its tetrabromide, melting at 104–105°.

A residue left after oxidation of the terpenes by shaking with cold permanganate solution was almost optically inactive. Boiled with permanganate solution, it yielded *p*-hydroxy-isopropyl benzoic acid, melting at 155°, thus proving the presence of *p*-cymene.

The terpenes of chenopodium oil therefore contain *p*-cymene, *l*-limonene and probably α-terpinene.

An examination of the terpenes separated from the oil distilled from the wild plants collected in Florida showed them to be of the same character.

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[1] Semi-Annual Report of Schimmel and Co., Apr., 1908, 113.

Summary.

Emphasis is placed on the necessity of carefully observing certain precautions in the distillation of chenopodium oil from the plant, in order to avoid the decomposition of the ascaridole by prolonged contact with steam or boiling water.

An examination of 7 authentic samples of chenopodium oil collected during the season of 1919 shows them to comply with the requirements of the United States Pharmacopeia and to contain from 60 to 77% of ascaridole.

The examination of a sample of chenopodium oil distilled in Java shows it to be very similar to the oils of American origin, and the analysis of 5 American oils returned from Brazil, and over a year old, shows them to be normal in character.

Chenopodium oil distilled from wild plants collected in Florida was found to contain less ascaridole than the oil distilled from cultivated plants in Maryland. The same constituents were found in it that were found in the Maryland oil.

The terpenes of chenopodium oil were found to contain *p*-cymene, *l*-limonene and probably α-terpinene.

WASHINGTON, D. C.

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF JOHNS HOPKINS UNIVERSITY.]

REACTIONS AND DERIVATIVES OF β,β'-DICHLORO-ETHYL SULFIDE.

BY OREGON B. HELFRICH[1] AND E. EMMET REID.

Received March 5, 1920.

Introduction.

In view of the prominence into which β,β'-dichloro-ethyl sulfide has been recently brought by reason of its use in the war as "mustard gas," it was thought that an investigation of the chemistry of this substance would be of particular interest. While mustard gas on the battle field was the most permanent of the gases used in large quantity and was apparently inert, it has been found that it can take part in quite a variety of reactions. The purpose of this work[2] has been to make a study of certain of these reactions. The study has been limited to the reactions which involve the sulfur atom and the 2 chlorine atoms. Since the reactions involving the chlorine atoms always removed them, generally as sodium

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[1] From dissertation of Oregon B. Helfrich.

[2] The mustard gas used in this investigation consisted of crude material obtained from both the American University Experiment Station and the Edgewood Arsenal, and of some of the redistilled product from the first mentioned place. The crude material was used as a source of the dichloro-ethyl sulfoxide and sulfone while the redistilled product was used in the condensations.