tional assignment used was not correct. Since the same assignment is used in this paper the values of the vibrational entropy must also be in some error but the absolute value of this error must be small for the reason given above.

The corresponding potential of 2000 cal. for dimethyl sulfide[14] seems somewhat high in view of the fact that the potential hindering internal rotation of the methyl groups in the case of tetramethylmethane is 5000 cal.[15] while that in the case of silicon tetramethyl[16] is only 1300 cal.

Acknowledgment.—We wish to thank Dr. R. B. Greenburg for the preparation of the extremely pure sample of dimethyl ether, and also Dr. S. C. Schumann for making the liquid hydrogen and measurements with the adiabatic calorimeter. The Grants-in-Aid of the National Research Council with which much of the apparatus was purchased made this work possible.

Summary

1. Pure dimethyl ether has been prepared with impurity of the order of 1 part per million.

2. The normal boiling point of dimethyl ether is 248.34 ± 0.05°K. (− 24.82°C.). The vapor pressure of the liquid from 170 to 190°K. can be represented by the equation

$$\log P_{\text{mm.}} = (4245.203/T) + 120.4931 \log T - 0.1225960T - 272.249320$$

and from 190 to 249°K. by the equation

$$\log P_{\text{mm.}} = (-1691.806/T) - 6.045600 \log T + 0.001957540T + 23.6861850$$

3. The melting point of dimethyl ether is 131.66 ± 0.05°K. (− 141.50°C.).

4. The heat capacity of solid and liquid dimethyl ether has been measured from 13.2 to 248.3°K.

5. The heat of fusion of dimethyl ether has been found to be 1179.8 ± 0.9 cal. per mole.

6. The heat of vaporization determined calorimetrically is 5141 ± 5 cal. per mole.

7. The second virial coefficient of dimethyl ether as determined from the density is − 0.4461 ± 0.0015 liter per mole at 298°K.

8. The molal entropy of the ideal gas calculated from the experimental data at the normal boiling point and 1 atmosphere is 61.01 ± 0.15 cal./deg. mole. At 200°K. and 1 atmosphere it is 58.03 ± 0.10 cal./deg. mole.

9. To bring the values of the entropy calculated from molecular data into agreement with the values from the calorimetric data requires the assumption of three equal potential energy maxima of 3100 ± 150 cal. hindering the internal rotation of the methyl groups. These are shown probably to be due to repulsive forces.

10. The entropies of the ideal gas and the superheated liquid at 298.16°K. are 63.72 ± 0.20 and 44.98 ± 0.40 cal./deg. mole, respectively.

STATE COLLEGE, PENNA.

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[14] Osborne, Doescher and Yost, *J. Chem. Phys.*, **8**, 506 (1940).

[15] Aston, *Chem. Rev.*, **27**, 59 (1940).

[16] Aston and Kennedy, THIS JOURNAL, **62**, 2567 (1940).

NOTES

An Investigation of the Oil from *Amaranthus retroflexus* Seeds

BY BERT E. CHRISTENSEN AND LEWIS S. MILLER

The purpose of this investigation is to determine the nature of the oil present in the seeds of the *Amaranthus retroflexus* L. of the family *amaranthaceae* (red root pig weed plant). Although the plant is very common and yields a considerable crop of a small seed, little information is available regarding the nature of the fatty oils which might be obtained from this source.[1] The seeds in this study were collected in Oregon, in October, 1940. The hulls were removed and the ground seed was extracted in a soxhlet apparatus. The *Amaranthus retroflexus* seed was found to contain 7% of an ether-extractable oil, the physical and chemical characteristics of which are given in Table I. Due to the limited amount of oil available semimicro methods were used throughout the investigation.[2,3]

Experimental

To meet the demands of this problem it was necessary to

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[1] A. D. Furshaev and N. S. Nozarova, *Trans. Soratov Univ.*, **11**, No. 1, 25–45 (1934).

[2] E. Chargaff, *Z. physiol Chem.*, **199**, 221–224 (1931).

[3] Jamieson, "Vegetable Fats and Oils," American Chemical Society Monograph Series, The Chemical Catalog Co., Inc., New York, N. Y.

TABLE I

PHYSICAL AND CHEMICAL CONSTANTS OF AMARANTHUS RETROFLEXUS SEED OIL

Specific gravity (24°/4°)	0.9236
Refractive index (Abbe 25°)	1.4751
Iodine number (Hanus)	89.9
Saponification value	187.4
Unsaturated acids (basis of oil) (cor. %)	71.6
Saturated acids (basis of oil) (cor. %)	18.6
Acetyl value	14.4
Mean molecular weight saturated acids	263.0
Mean molecular weight unsaturated acids	302.0
Iodine number of unsaturated acids	122.0
Iodine number of saturated acids	22.5
Unsaponifiable matter, %	7.6
Acid value	3.9

develop a practical micro distillation unit for work at low pressure (1–4 mm.) which would have a low hold up and high efficiency.

Wyman and Barkenbus[4] have developed such a still based on the use of a spinning band in which the successful separation of the methyl esters of stearic and palmitic acids was reported. Since their column was not available in this Laboratory, another still based on a different principle was devised.

A small distilling unit constructed on the same principle as the Booth column[5] was designed. The still, one hundred centimeters over-all, was constructed from three concentric glass tubes and a glass rod. A cold finger served as the condensing unit.

Eighteen gage nichrome wire one turn per centimeter wound around a 6-mm. Pyrex rod and then inserted into a close fitting Pyrex tube served as the fractionating unit. This arrangement forced all the vapors up a spiral path between the two concentric walls.

A 30-mm. glass jacket, around which was wound nichrome wire, and an outer 50-mm. Pyrex glass tube provided the means of maintaining adiabatic conditions. The heating element was wound almost to the take off.

A thermometer was placed in the annular space between the fractionating unit and the outer jacket. The temperature of the column was controlled by means of a variable transformer (Adjustavolt) while the distillation flask was heated by means of a phosphoric acid bath.

Since no thermometer was provided at the take off, samples were removed at frequent intervals and compared by means of a refractometer.

Unsaturated Acids.—The unsaturated acids were separated by the lead salt-ether method.[3] Eight grams of methyl esters was prepared and about 3 g. then distilled at 2 mm. using the micro distillation apparatus described above. Various fractions were collected and characterized by their refractive indices.

The first and second fractions averaged 250 mg. and gave refractive indices at 25° of 1.4550 and 1.4574, respectively; next four fractions (3–6), averaging 430 mg. each, gave 1.4580 ± 0.0001, while the two remaining fractions (7–8) of 300 mg. each gave 1.4692 and 1.51; 150 mg. of material was unrecovered. The saponification equivalent of fraction 4 was 284, the iodine no. 147.1, and the percentage composition 77.1% C and 11.62% H. From the data it appears that fractions 3–6 are composed of a mixture of unsaturated acids.

The hydrogenation of fraction 5 gave a solid ester, with iodine no. of 11.7, percentage composition of 76.0 C and 12.5 H, and m. p. 37°. This agrees fairly well with corresponding data for pure methyl stearate (76.4% C, 12.8% H, and m. p. 38°) reported in literature. A further confirmation was the anilide m. p. 93° (reported 93.6°). From these data it is evident that the unsaturated fraction was composed largely of eighteen carbon atom acids, probably oleic and linoleic acids. Calculations from the iodine value (122) of the unsaturated acids indicate the presence of 46% oleic and 25% linoleic acids on the basis of the original oil.

Saturated Acids.—The mean molecular weight of the saturated fraction as obtained from the lead salt-ether separation was 263.0. This lies between the values for palmitic and stearic acids, indicating a mixture. Four and one-half grams of methyl esters was fractionally distilled at 3 mm. pressure. Various fractions were collected and characterized by their refractive indices.

The first five fractions collected, averaging about 400 mg. apiece, gave a refractive index at 45° of 1.4311 ± 0.0001 and a saponification equivalent of 271 ± 1, which agrees accurately with the properties of methyl palmitate. A further confirmation was the percentage composition, namely, 75.6 C and 12.5 H, of fraction 2 and its melting point, 29°, which agrees almost exactly with the corresponding data reported for methyl palmitate (75.6% C, 12.6% H, and 29°). The anilide of the fourth fraction melted at 87°, while the reported value for the anilide of palmitic acid is 87.5°. Three additional fractions were collected, giving refractive indices of 1.4353, 1.4376 and 1.4407, respectively, which agrees very closely with the properties and refractive index of methyl stearate. The saponification equivalents were, respectively, 278, 278, 292 and 296, the latter figure agreeing excellently with the saponification equivalent of pure methyl stearate. The percentage composition (76.3 C and 12.3 H) and the melting point, 34–36°, agreed closely with the values reported in the literature, namely, 76.4 C, 12.8 H and 38°. From these data it appears that the saturated fraction consists principally of palmitic acid with a small percentage of stearic acid. Calculations on the basis of the saponification equivalent gave a value of 16.9% palmitic and 1.7% stearic acids in the original oil.

DEPARTMENT OF CHEMISTRY
OREGON STATE COLLEGE
CORVALLIS, OREGON

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The Vapor Phase Catalytic Conversion of Tertiary Butylmethylcarbinol

BY E. A. KELSO, GEORGE WASH, J. T. HORECZY, BILLIE SHIVE WITH W. A. FELSING

Cramer and Glasebrook[1] reported that the vapor phase dehydration of *t*-butylmethylcarbinol over activated alumina yielded a mixture of ole-

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[4] F. W. Wyman and C. Barkenbus, *Ind. Eng. Chem., Anal. Ed.*, **12**, 658 (1940).

[5] H. S. Booth and A. R. Bozarth, *Ind. Eng. Chem.*, **29**, 470 (1937).

[1] Cramer and Glasebrook, THIS JOURNAL, **61**, 230 (1939).