Experimental

Silicochloroform.—This was prepared by the action of hydrogen chloride gas on ferrosilicon containing 95–97% silicon.[4,5] The volatile products were condensed by alcohol and solid carbon dioxide and a fraction (75%) boiling between 28 and 36° was refractionated and a fraction boiling close to 31.8° collected (44.6% of original condensate).

Anal. Silicon and chlorine were determined after reaction with water as silica and hydrogen chloride. Calcd.: Si, 20.72; Cl, 78.53. Found: Si, 20.2, 20.6, 20.6; Cl, 79.6, 78.7, 78.7.

Trimethyl Silane.—To 500 cc. of an ether solution containing 175 g. of methylmagnesium bromide was added slowly with strong cooling and stirring 60 g. of silicochloroform in 150 cc. of ether. The reflux condenser was cooled by circulating cold brine. After completion of the reaction, the reflux condenser was allowed to rise to room temperature and connection made to a cooled receiver through another condenser cooled by alcohol and solid carbon dioxide. Cooled dilute sulfuric acid was added slowly and trimethyl silane together with some ether distilled over. Concentrated sulfuric acid was added to the receiver and allowed to stand cold overnight. Trimethyl silane was then distilled off; b. p. uncor. 9 to 11°. A small amount of dissolved gas assumed to be methane was lost below 0°. The yield was poor. In each of several experiments 15 cc. of trimethyl silane was obtained from 40 cc. of silicochloroform.

Trimethyl Silicon Chloride.—Trimethyl silane was treated at −20° with chlorine and then allowed to stand at room temperature until most of the hydrogen chloride had escaped. A fraction boiling between 58 and 63° was collected and redistilled and collected between 57 and 59.4° at 747 mm. This represented 75% of the original total. There was a small residue, non-volatile at 100°, left from the first distillation.

Anal. Silicon content by adding concd. sulfuric acid, warming, then adding concd. nitric acid and finally weighing silica. Cl content by hydrolyzing and titration. Calcd.: Si, 25.85; Cl, 32.67. Found: Si, 25.4, 25.9, 25.8, 25.6; Cl, 32.1, 32.2, 33.0. Taking CH_3 by difference, the mean percentages correspond to $(CH_3)_{2.99}SiCl_{1.00}$.

Vapor Pressure of Trimethyl Silicon Chloride.—This was determined by the method of Smith and Menzies.[6] Temperatures were read on a thermometer corrected by reference to the b. p.'s of purified ether, carbon tetrachloride, and water. Interpolation by least squares gives

t, °C.	28.9	35.3	40.0	45.2	49.8	55.0	56.1
p, mm.	308	366	427	481	575	676	725

$\log_{10}p = 6.926 - 1344/T$. The b. p. at 760 mm. is calculated to be 59°; density, d^{25}_{4}, 0.846; freezing point, approx. −40°.

---

[4] Buff and Wöhler, *Ann.*, **104**, 94 (1857).

[5] Booth and Stillwell, This Journal, **56**, 1529 (1934). Other references are given in this paper.

[6] Smith and Menzies, *ibid.*, **32**, 897 (1910).

RESEARCH LABORATORY
ALBRIGHT & WILSON, LTD.
OLDBURY, BIRMINGHAM
ENGLAND

RECEIVED SEPTEMBER 3, 1943

The Viscosity of the Methyl Ester of Dilinoleic Acid

BY DAVID W. YOUNG AND RUTH E. BIERTUEMPFEL

The purpose of this note is to present viscosity measurements on a pure sample of methyl dilinoleate over a range of temperature from −40 to 212°F. (−40–100°C.).

Experimental Procedure

The ester used for this work had been purified by double distillation in a molecular still by Dr. John C. Cowan in the Northern Regional Research Laboratory. The ester had an index of refraction of n^{30}_{D} 1.4766. Recently the method used to formulate and purify the methyl dilinoleate from soybean oil has been reported.[1]

The kinematic viscosity method used in the study is intended for determining the viscosity of any product which is a true viscous liquid at the temperature of the test. The viscometers available were the Ubbelohde type with one capillary tube. Care was taken to calibrate each viscometer against standard oils of known viscosity as determined in the National Bureau of Standards. The viscosity method is given under American Society for Testing Materials, Designation D 445-42 T.

For the viscosity determination at −40° the procedure given by Baldeschwieler and Wilcox[2] was used.

Results

Experimental results are listed in Table I and Fig. 1. A straight line relationship exists when kinematic viscosity is plotted as a function of temperature on A. S. T. M. tentative viscosity–temperature chart (D 341–37 T). The temperature coördinate has been extended 10°, that is, to −40°, in order to present complete data.

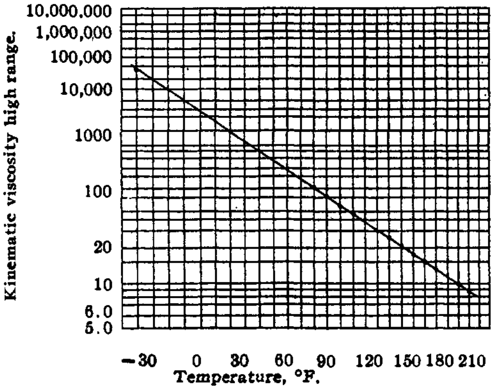

Fig. 1.

TABLE I

KINEMATIC VISCOSITY OF METHYL DILINOLEATE

Temp., °F.	Viscosity in centistokes
−40	42,464
80	102.5
90	78.3
100	60.0
110	48.0
140	25.4
150	21.1
160	17.8
170	15.2
180	13.1
200	10.0
210	8.70

It was held that these results would be of interest as the pure material is rather difficult to

---

[1] Cowan and Wheeler, This Journal, **66**, 84 (1944).

[2] Baldeschwieler and Wilcox, *Ind. Eng. Chem., Anal. Ed.*, **11**, 221 (1939).

obtain and the dimer had a viscosity index (Dean and Davis system) of 123.0.

STANDARD OIL COMPANY OF NEW JERSEY
ELIZABETH, NEW JERSEY
RECEIVED MARCH 3, 1944

NEW COMPOUNDS

New Phenolic Mercurials[1]

In a study of the preparation of new phenols of possible pharmaceutical interest the following new derivatives, which include mercurials of *p*-*t*-octylmono-,[2a] cyclohexylidenedi-,[2b] and hexyl-tetraphenols,[2c] have been prepared and characterized.

TABLE I

NEW PHENOLIC MERCURIALS

Name	Formula	M. p., °C. (uncor.)	Yield, %	Percentage Hg[3] Calcd.	Percentage Hg[3] Found
I. 4-$\alpha,\alpha,\gamma,\gamma$-Tetramethylbutylphenols					
(a) 2-Acetoxymercuri-	$C_{16}H_{24}O_3Hg$	158	80	43.16	43.10
(b) 2-Chloromercuri-	$C_{14}H_{21}OHgCl$	161	90	45.46	45.30
(c) 2,6-Diacetoxymercuri-	$C_{16}H_{26}O_5Hg$	181	90	55.46	54.60
(d) 2,6-Dichloromercuri-	$C_{14}H_{20}OHg_2Cl_2$	238D[a]	80	59.18	58.60
(e) 2,6-Diacetoxymercuri-3-hydroxy-	$C_{18}H_{26}O_4Hg_2$[b]	183D[a]	75	54.26	54.68
(f) 2-Acetoxymercuri-6-methyl-	$C_{17}H_{26}O_3Hg$	149	80	41.87	42.05
II. 1,1-Bis-(4′-hydroxyphenyl)-cyclohexane					
(a) Bis-(2′-acetoxymercuri-6′-methyl)-	$C_{24}H_{28}O_6Hg_2$	200D	90	49.33	49.60
(b) Bis-(2′-chloromercuri-6′-methyl)-	$C_{20}H_{22}O_2Hg_2Cl_2$	225D	80	53.05	52.10
(c) Bis-(2′,6′-diacetoxymercuri)-	$C_{26}H_{28}O_{10}Hg_4$	210D	82	61.60	61.50
(d) Bis-(2′,6′-dichloromercuri)-	$C_{18}H_{16}O_2Hg_4Cl_4$	222D	75	66.45	66.10
III. 2,2,5,5-Tetrakis-(4′-hydroxyphenyl)-hexanes					
(a) Tetrakis-(2′,6′-diacetoxymercuri)-	$C_{46}H_{46}O_{20}Hg_8$	308D	91	63.60	63.00
(b) Tetrakis-(2′,6′-dichloromercuri)-	$C_{30}H_{22}O_4Hg_8Cl_8$	247D	80	68.74	67.70

[a] Decomposes. [b] The chloromercurial is an oil.

Polyacetoxymercurials.—A molar equivalent of mercuric acetate was allowed for each free position ortho-to-the-hydroxyl group per mole of phenol. Knowing the ratio of the reactants to be used, the quantities of phenol and mercuric acetate could be calculated for any size batch desired or found to be convenient. A 2–6% solution of the phenol with the calculated amount of mercuric acetate was refluxed in 95% ethyl alcohol containing 5% glacial acetic acid in an appropriate flask, fitted with a reflux condenser, for one to two hours. The flask was then chilled in the refrigerator overnight, the polymercurial filtered off and recrystallized from ethanol or glacial acetic acid. The yields were in the neighborhood of 70–90%.

Monoacetoxymercurials.—For these one mole of phenol was allowed to react with only one-half the quantity of mercuric acetate as calculated above, in a 50% aqueous ethyl alcohol solution containing 5% glacial acetic acid, at room temperature, over a period of one to two weeks. When the solution no longer contained free mercuric ions, as shown by the absence of a positive reaction when tested for such ions with potassium iodide solution (red precipitate of mercuric iodide soluble in excess of reagent), the solution was then chilled in the refrigerator and the crude mono-mercurial filtered off and dried. The monomercurial was recrystallized from 50% aqueous alcohol containing 5% glacial acetic acid. The yield was about 80–85%.

Chloromercurials.—These were obtained by pouring an alcoholic or a glacial acetic acid solution of the acetoxymercuri derivative into an equal volume of a 20% aqueous solution of reagent grade sodium chloride. The chloromercurial was filtered off and recrystallized from ethanol or glacial acetic acid. The yields varied from 70–90%.

THE CHEMICAL LABORATORIES OF
NEW YORK UNIVERSITY
WASHINGTON SQUARE COLLEGE
NEW YORK, N. Y.
JOSEPH B. NIEDERL
ANTHONY J. SHUKIS
RECEIVED FEBRUARY 8, 1944

---

[1] Abstracted from the thesis of Anthony J. Shukis presented to the Graduate School of New York University, New York, N. Y., in partial fulfillment for the degree of Master of Science, June, 1940.

[2] J. B. Niederl and co-workers, (a) THIS JOURNAL, **55**, 2571 (1933); (b) *ibid.*, **61**, 345 (1939); (c) *ibid.*, **63**, 1235 (1941).

[3] A. J. Shukis and R. C. Tallman, *Ind. Eng. Chem., Anal. Ed.*, **12**, 123 (1940).

Double Invert Soaps: Symmetrical Di-piperidinium Salts[1]

Several long chain substituted dipiperidinium salts have been prepared in the course of investigation of various types of symmetrical double invert soaps. Methylene- and benzal-di-piperidine, prepared by condensing formaldehyde or benzaldehyde respectively with two moles of piperidine, were treated with double molar quantities of various *n*-alkyl bromides, such as *n*-heptyl, *n*-octyl, *n*-tetradecyl and *n*-hexadecyl bromides. Reaction took place rather readily in all cases and evidently proceeded as follows

$$(C_5H_{10}N)_2CH_2 + 2RBr \longrightarrow [(C_5H_{10}NR)_2CH_2]^{++}\ 2Br^{--}$$

$$(C_5H_{10}N)_2CHC_6H_5 + 2RBr \longrightarrow [(C_5H_{10}NR)_2CHC_6H_5]^{++}\ 2Br^{--}$$

$$R = -C_7H_{15}\text{-}n,\ -C_8H_{17}\text{-}n,\ -C_{14}H_{29}\text{-}n,\ -C_{16}H_{33}\text{-}n$$

Procedure.—To 0.02 mole of methylene- or benzal-dipiperidine was added 0.04 mole of the respective alkyl bromide and 20 ml. of 95% ethyl alcohol. The mixture was then refluxed gently for four hours. The alcohol was then removed by evaporation under reduced pressure. The remaining residue was repeatedly crystallized from ethyl acetate; yields, 50–70%.

---

[1] Abstracted from the thesis of Anthony E. Lanzilotti presented to the Graduate School of St. Peter's College, Jersey City, N. J., in partial fulfillment for the degree of Master of Science, May, 1944.