obtained after dilution of the mother liquor with water. After two recrystallizations from glacial acetic acid the product melted at 231–232° (lit. m.p. 230–231°).[6]

Chlorination of 4-Methyl-*o*-phenylenedibenzenesulfonamide: 4-Methyl-3,5,6-trichloro-*o*-phenylenedibenzenesulfonamide.—Into a solution of 10 g. of 4-methyl-*o*-phenylenedibenzenesulfonamide in 40 ml. of dimethylformamide, chlorine was passed until the reaction mixture had gained 9.5 g. in weight, keeping the temperature below 50°. The mixture was worked up as described for the methyl-free product. The yield was 7.8 g. (62%). A sample was recrystallized for analysis from glacial acetic acid; white microcrystalline powder, m.p. 237–238°.

Anal. Calcd. for $C_{19}H_{15}Cl_3N_2O_4S_2$: C, 45.12; H, 2.95; N, 5.54. Found: C, 45.36; H, 3.20; N, 5.53.

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Preparation and Hydrolysis of Optically Active 2-Butyl Acetal

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O'Gorman and Lucas[3] have recently shown that hydrolysis with 5% aqueous phosphoric acid of D-(+)-2-octyl acetal leads to totally unracemized D-(+)-2-octanol. They concluded that the reaction does not proceed through a secondary octyl carbonium ion.

We have confirmed their conclusions using D-(+)-2-butyl acetal from D-(+)-2-butyl orthoformate.[4] The hydrolysis of this acetal to D-(+)-2-butanol gives alcohol of the same specific rotation as that used initially. It is, therefore, probable that none of the reactions employed in this cycle involves cleavage of the oxygen–butyl bond and that no secondary butyl carbonium intermediate is involved.

Experimental[5]

D-(+)-2-Butanol.—2-Butanol, Eastman Kodak Company White Label, was resolved by the method of Pickard and Kenyon[6] according to the modification of Sprung and Wallis[7] and had an observed rotation of +7.98 ± 0.02° in a one-decimeter tube at 25°.

D-(+)-2-Butyl Orthoformate.—The preparation of D-(+)-2-butyl orthoformate, $[\alpha]^{25}_D$ +27.07 ± 0.02° (*l* 1, no solvent), was carried out as described by Alexander and Busch.[4]

D-(+)-2-Butyl Acetal.—To 24.0 g. (0.103 mole) of D-(+)-2-butyl orthoformate, 2.0 g. (0.025 mole) of granulated ammonium nitrate and 6.0 ml. of D-(+)-2-butanol was added 15.0 g. (0.34 mole) of freshly prepared acetaldehyde. After the mixture was refluxed for 90 minutes, 150 ml. of ether was added to the cooled solution. It was then washed with aqueous ammonium hydroxide (1:1) and distilled water. The ether solution was dried over anhydrous potassium carbonate. The ether was removed and the solution was distilled under vacuum through a five-inch column packed with glass helices. Active *s*-butyl acetal (11.5 g., 64.5%) was obtained as a colorless liquid, b.p. 66–68° (16 mm.); n^{20}_D 1.4050; d^{20}_4 0.8279, $[\alpha]^{25}_D$ +25.40 ± 0.02° (*l* 1, no solvent).

Anal. Calcd. for $C_{10}H_{22}O_2$: C, 68.91; H, 12.73; *MR*, 51.68. Found: C, 68.62; H, 12.95; *MR*, 51.59.

Hydrolysis of Acetal.—A mixture of 11.5 g. (0.066 mole) of D-(+)-2-butyl acetal and 100 ml. of 5% phosphoric acid was refluxed for 1 hour. The cooled solution was saturated with potassium carbonate and extracted with ether. After drying over anhydrous potassium carbonate, the ether solution was distilled. Alcohol, (6.7 g., 71%) b.p. 97–98° with $[\alpha]^{25}_D$ +8.00 ± 0.02° (*l* 1), was obtained.

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[1] Deceased.

[2] University of Illinois, College of Dentistry, Chicago, Illinois.

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[7] M. Sprung and E. Wallis, THIS JOURNAL, **56**, 1717 (1934).

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The Addition of Fluorene to, and the Fine Structure of, Benzylidenefluorene

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Like all fulvenes,[1] benzylidenefluorene has a polar semicyclic double bond; the moment of the latter is directed toward the five-membered ring. It is, therefore, correct to regard the addition of fluorene to benzylidenefluorene, which has been observed by Pinck and Hilbert,[2] as a special case of the Michael condensation; the latter occurs only with polar unsaturated systems.[3]

The direction of the dipole moment in benzylidenefluorene demands that in the addition of fluorene the proton derived from the 9-hydrogen atom of fluorene combines with the 9-carbon atom of the benzylidene compound and the fluorenyl anion with the phenylated carbon atom, thus giving di-(9-fluorenyl)-phenylmethane (I), in analogy to the addition of lithium aluminum hydride in which the negative hydrogen ion[4] combines with the phenylated carbon atom, the $(LiAlH_3)^+$ ion with the central carbon atom of benzylidenefluorene.[5]

Pinck and Hilbert[2] have not decided whether their condensation product of m.p. 240° was (I) or the isomeric 9-benzyl-9,9′-difluorenyl (II), which would have resulted from the inverse addition of fluorene to benzylidenefluorene. (II)[6] has, moreover, the same melting point as Pinck and Hilbert's hydrocarbon.

It has now been shown that this hydrocarbon is not identical with (II); a mixture of the two compounds gives a strong melting point depression.[7] It is, therefore, concluded that formula (I) is correct. The Michael condensation of fluorene and benzylidenefluorene (I), is, thus, an additional proof for the direction of the moment in the latter which has been predicted by the theory.

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[6] R. C. Fuson and H. D. Porter, THIS JOURNAL, **70**, 895 (1948).

[7] The referee kindly informed the authors that he has made the same observation with the two original samples obtained by Pinck and Hilbert and by Fuson and Porter, respectively.