the flask; III appeared to be only slightly soluble in the ammonia. Stirring was begun, and a small piece of sodium added; the blue color faded very slowly. On the addition of 50 ml. of dry ether, a homogeneous solution resulted. Subsequent addition of sodium was followed by rapid disappearance of the blue color. Sodium was added until the blue color persisted for 15 minutes; 6.9 g. (0.3 mole) was required. Sixteen grams (0.3 mole) of ammonium chloride was added portionwise, followed by 100 ml. of dry ether. The flask was removed from the cooling bath and the ammonia allowed to evaporate. Water was added until two clear layers resulted. The aqueous layer was withdrawn and extracted twice with ether. These ether extracts, together with the original ether layer, were shaken with saturated sodium chloride solution and filtered through anhydrous sodium sulfate. Distillation gave 11.1 g. of I, b.p. 94–98° at 8 mm., n^{25}D 1.4640 (87% yield). The hydrochloride melted at 208.0–209.5°, alone and when mixed with an authentic sample of the hydrochloride of I.

RESEARCH DIVISION
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NEW COMPOUNDS

6-*p*-Toluenesulfonyldiisopropylidene-D-galactose

6-*p*-Toluenesulfonyldiisopropylidene-D-galactose Diethylmercaptal.—One and one-half grams (0.0034 mole) of 6-*p*-toluenesulfonyl-D-galactose diethylmercaptal was suspended in 25 ml. of dry, redistilled acetone containing 2% by weight of sulfuric acid. Five and one-half grams of anhydrous copper sulfate was added and the mixture shaken for fifty hours. The suspension was filtered, an excess of finely divided anhydrous sodium carbonate introduced, and the mixture shaken for nine hours. After filtration, the solution was treated with norite for two hours at room temperature, centrifuged clear, and the solvent removed under reduced pressure. A brown sirup weighing 0.7 g. (39%) was obtained which crystallized spontaneously on standing. By dissolving in petroleum ether and storing for several days in the ice box, a small quantity of long white needles were obtained. These were thought to be a monoacetonated product and were removed by centrifugation. On further standing, 6-*p*-toluenesulfonyldiisopropylidene-D-galactose diethylmercaptal crystallized as flat plates. It was recrystallized in a similar manner to constant properties. The rotation of this material, m.p. 53°, was $[\alpha]^{28}$D −36.4° in chloroform (*c* 3.06).

Anal. Calcd. for $C_{23}H_{36}O_7S_3$: C, 53.1; H, 6.92. Found: C, 52.8; H, 7.02.

1-Bromo-2,3-isopropylideneglycerol.—Fifty-seven grams (0.66 mole) of anhydrous lithium bromide was dissolved in 150 ml. of dry acetone. To the viscous liquid which resulted was added 66.8 g. (0.22 mole) of 1-*p*-toluenesulfonyl-2,3-isopropylideneglycerol. The clear solution was heated to refluxing and after a few minutes a crystalline precipitate began to appear, which soon filled the entire flask. Fifty milliliters of dry acetone was added and the mixture refluxed for two hours. The following day, 100 ml. of dry acetone was introduced and the solid filtered off. The weight of white flaky lithium *p*-toluenesulfonate obtained was 31 g. The acetone was removed from the filtrate under reduced pressure and the resulting sirup extracted with portions (100 ml. each) of ether under reflux. After drying over sodium sulfate, the ether was removed from the combined extracts under reduced pressure and the remaining liquid distilled through a small column. The total yield of 1-bromo-2,3-isopropylideneglycerol of b.p. 45° (4 mm.), n^{25}D 1.4601, and m.p. −23°, was 6.9 g. (14%).

Anal. Calcd. for $C_6H_{11}O_2Br$: C, 37.0; H, 5.68. Found: C, 37.3; H, 5.62.

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Studies in Silico-organic Compounds. XX. Preparation of a Silahydrocarbon and a Dichloride

The preparation of two new organic compounds of silicon, 2,2,5,5-tetramethyl-2,5-disilahexane and di-*n*-butyldichlorosilane, is herein described.

Sodium metal (9.2 g., 0.4 mole) was cut into small pieces and placed in 100 cc. of anhydrous amyl ether. Bromomethyltrimethylsilane (66.8 g., 0.4 mole) was added dropwise with stirring. As no apparent reaction was taking place after about one fourth of the bromomethyltrimethylsilane had been added, the reaction mixture was heated. The mixture soon began to turn blue and maintained a temperature of 50° without heating during the addition of the remainder of the bromide. The reaction mixture was then heated to the melting point of sodium and a sharp rise to 130° was noted. A cooling bath was applied and the temperature maintained at 105° for four hours. This temperature was chosen as the bromomethyltrimethylsilane boils at 115° and its loss was to be prevented. A Dry Ice–acetone trap had been placed at the condenser exit to catch any escaping low boiling material. Examination of the contents of the trap showed it contained 5 cc. of a low boiling liquid. This liquid was identified as tetramethylsilane by its refractive index (found) n^{23}D 1.3580 (literature),[1] n^{20}D 1.3591.

2,2,5,5-Tetramethyl-2,5-disilahexane was isolated, b.p. 150.5–151.5° (750 mm.), n^{25}D 1.4170, d^{25}_4 0.7457, 14.3 g., yield 40%.

Anal. Calcd. for $C_8H_{22}Si_2$: Si, 32.18; mol. wt., 174.37; *MR*, 58.43. Found: Si, 31.84; mol. wt., (cryoscopic in benzene) 174.0; *MR*, 58.81.

From the interaction of 170 g. (1.0 mole) of tetrachlorosilane and 3 moles of *n*-butylmagnesium bromide in absolute ether, there were obtained tri-*n*-butylchlorosilane and di-*n*-butyldichlorosilane. The flask was cooled during the reaction with an ice-bath and the Grignard reagent added dropwise with stirring. After addition, the system was heated and stirred for eight hours, then filtered with suction and distilled. Tri-*n*-butylchlorosilane, b.p. (found and literature)[2] 142–144° (29 mm.), n^{25}D 1.4460, 27.0 g., yield 11.5%; di-*n*-butyldichlorosilane, b.p. 109.0–109.5° (28 mm.), n^{25}D 1.4430, d^{25}_4 0.9850, 75.5 g., yield 35%.

Anal. Calcd. for $C_8H_{18}Cl_2Si$: Si, 13.16; Cl, 33.26; *MR*, 57.32. Found: Si, 12.77; Cl, 32.83; *MR*, 57.34.

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(1) H. H. Anderson, THIS JOURNAL, **69**, 3049 (1947).

(2) H. Gilman and H. J. Marshall, *ibid.*, **71**, 2066 (1949).

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The Coördination Compound of Boron Trifluoride with Tri-*n*-butylamine

$(C_4H_9)_3N:BF_3$ was prepared in a manner similar to that described by Kraus and Brown[1] in the case of the preparation of $(C_2H_5)_3N:BF_3$.

When 5 ml. of freshly distilled boron trifluoride etherate was added to a solution of 8.5 ml. of anhydrous tri-*n*-butylamine (dried over anhydrous sodium sulfate) in dry diethyl ether at −78°, a white precipitate was formed. However, this solid completely dissolved in the ether when the solution was warmed to room temperature. Addition of petroleum ether (b.p. 30–60°) yielded a precipitate of white crystals (7.1 g.) which were filtered off and washed several times with petroleum ether. These crystals may be crystallized from pure petroleum ether and melt at 59–61°. *Anal.* Calcd. for $C_{12}H_{27}N:BF_3$: C, 56.93; H, 10.75; N, 5.53. Found: C, 55.60; H, 10.64; N, 5.35.

The infrared spectrum of $(C_4H_9)_3N:BF_3$ in chloroform was obtained with a Baird spectrophotometer. The principal bands in the range of 2–16 μ are: 3.54, 6.80, 7.24, 8.78, 9.00, 10.75, 11.10 and 11.84 μ.

The coördination compound of tri-*n*-butylamine with boron trifluoride is very soluble in benzene, chloroform, carbon tetrachloride and ether at room temperature, but is sparingly soluble in petroleum ether. The colorless

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(1) C. A. Kraus and E. H. Brown, THIS JOURNAL, **51**, 2690 (1929).