tracted with ether. No attempt was made to isolate the free aldehyde but it was at once converted to the *semicarbazone*, white crystals from ether; m.p. 155–156°. The yield was poor.

*Anal.* Calcd. for $C_{15}H_{26}N_6O$ (mol. wt. 306.41); C, 58.79; H, 8.55. Found: C, 58.98; H, 8.32.

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## A Convenient Method for the Preparation of Di-*n*-butylfluoroborane

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Hennion, McCusker, and Rutkowski[1] have recently reported the preparation of diisobutylfluoroborane by treatment of the corresponding chloroborane with antimony trifluoride. Antimony trifluoride is known to replace the other halides for fluoride, but it is unusual for this reagent to replace an alkyl group for fluoride. Such a reaction was used to prepare di-*n*-butylfluoroborane in a very convenient one step process. Tri-*n*-butylborane was heated with antimony trifluoride. At about 135°, the di-*n*-butylfluoride began to distill, and an antimony mirror formed on the flask walls. The reaction was completed by heating to 180°. The lower boiling butyldifluoroborane might be expected to form, but none was detected.

Buls, Davis, and Thomas[2] have reported the di-*n*-butylfluoroborane to boil at $-5°/100$ mm. The material reported here had a much higher boiling point, 48°/20 mm. ($d_4^{24}$ 0.783). Hennion *et al.* reported their isobutyl isomer to boil at 49°/52 mm. ($d_4^{25}$ 0.7662). The higher boiling point (48°/20 mm.) would be expected since it lies between the monoalkyldifluoroborane (36.3°/742 mm.)[3] and the tri-*n*-butylborane (90°/9 mm.).[3]

### EXPERIMENTAL

*Di-n-butylfluoroborane.* The reaction system consisted of all glass connections, and all operations were carried out under nitrogen. A flask was charged with 32 g. of antimony trifluoride and 50 g. of tri-*n*-butylborane. On heating to 135°, a volatile material began to distill and was collected in a cooled receiver. The temperature was gradually increased to 180°, and when distillation ceased, the heat was removed. The water-white product weighed 41.3 g. This material was distilled twice through a 1-ft. × $^1/_4$-in spiral wire packed column. The water-white mobile liquid weighed 25 g. and was flammable in air. The product distilled at 47°/20 mm. ($d_4^{24}$ 0.783).

*Anal.* Calcd. for $C_8H_{18}BF$: F, 13.18. Found: F, 12.91, 13.09.

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## Synthesis of Cyanotriphenylmethane from Hexaphenylethane

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The postulation of the involvement of the cyanyl radical in the reaction of mercuric cyanide with disilicon hexachloride[1] has prompted a search for further examples in which a carbon-nitrogen pair might react as an uncharged unit. By the reaction of hexaphenylethane and mercuric cyanide, cyanotriphenylmethane has been formed in 15% yield.

Whether this reaction has involved the cyanyl radical or the cyanide ion is not certain. It is of interest to note earlier reactions between organic radical precursors and mercury salts other than mercuric cyanide[2] and to note reactions between hexaphenylethane and several inorganic salts.[3]

### EXPERIMENTAL

A solution of 0.09 mole of hexaphenylethane in 250 ml. of benzene was prepared.[4] Silver chloride and excess silver were removed by filtration under nitrogen. A suspension composed of the benzene solution and 0.1 mole of mercuric cyanide was stirred magnetically for 10 days at room temperature beneath an atmosphere of nitrogen. The suspension was filtered. The filtrate was extracted with water. The washed benzene filtrate containing the crude product was evaporated to dryness. This residue was extracted with boiling ethanol. From the ethanol-soluble portion was isolated by fractional sublimation and fractional crystallization (ethanol solvent) 7.55 g. (0.028 mole) of cyanotriphenyl methane. This product was shown to be identical with an authentic sample prepared by the method of Fischer[5] by the identity of their infrared spectra and on the basis of a mixture melting point.

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