ethyl α-chloro-γ,γ,γ-trifluoroacetoacetate).[2] As would be expected, it fails to form a chelate compound with copper salts.

In an all-glass apparatus, protected from moisture by a calcium chloride tube, a solution of 45 g. (0.245 M) of ethyl γ,γ,γ-trifluoroacetoacetate in 100 ml. of dry chloroform was stirred and cooled to about −78° in a Dry Ice–acetone-bath. A solution of 45 g. (0.570 M) of dry pyridine and 78.5 g. (0.490 M) of dry bromine in 200 ml. of dry chloroform was added dropwise. When all of the bromine solution had been added, the reaction was allowed gradually to warm to room temperature, with gentle stirring for 12 hours, then stirred four hours at 55–60°. The bulk of the chloroform was removed *in vacuo*, and the orange residue warmed and stirred with 400 ml. of dry petroleum ether (b.p. 20–40°). The mixture was filtered, the crystals washed well with more petroleum ether, and the filtrate stripped of solvent *in vacuo*. Vacuum distillation of the residue gave 49.7 g. of orange liquid, b.p. 37–81° (9 mm.). This was redistilled through a small Widmer column from a trace of zinc dust, giving 28.3 g. (34%) of ethyl α,α-dibromo-γ,γ,γ-trifluoroacetoacetate, b.p. 74–5° (9 mm.), $n^{26}_{\rm D}$ 1.4420.

Anal. Calcd. for $C_6H_5O_3F_3Br_2$: C, 21.03; H, 1.45; F, 16.67; Br, 46.8. Found: C, 21.04; H, 1.46; F, 14.44; Br, 47.4.

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The Dipole Moment of Ethylgermanium Trichloride

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Ten years ago Smyth[2] determined the dipole moments of some trialkylgermanium halides, but the dipole moment of ethylgermanium trichloride was not included. Since ethyl- and diethylgermanium chlorides may now be prepared quite readily and conveniently by the direct synthesis,[3] the authors have determined the electric moment of ethyl germanium trichloride and have further characterized this material.

Experimental

Ethylgermanium Trichloride.—Ethylgermanium trichloride was prepared by the direct reaction of ethyl chloride with mixed copper and germanium powders at 317° in the manner that has previously been described.[4] The desired compound was isolated from the reaction mixture by fractional distillation. The fraction boiling at 140.0° and 763 mm. was collected as ethylgermanium trichloride.

The purity of the ethylgermanium trichloride was established by cryoscopic measurement of its molecular weight in anhydrous benzene and by the determination of the molar refraction of the compound. In a typical experiment, a 0.57% solution in benzene showed a depression of the freezing point corresponding to a molecular weight of 202 (calcd., 207.9). At 25.0° the refractive index of the pure ethylgermanium trichloride was found to be 1.4719. By employment of a dilatometric pycnometer the density of this liquid was found to be 1.5953 g./cm.³ at 25.0°. These physical constants lead to a value of the molar refraction, $R^{25}_{\rm D}$, of 36.50 cm.³. If the bond refraction of the Ge–C bond is assumed to be 4.13 cm.³ and the bond refraction of the Ge–Cl bond is taken as 7.89 cm.³, these values in combination with the bond refractivities of Denbigh[5] lead to a calculated molar refraction of 36.1 cm.³.

Benzene.—Merck and Co., Inc., Reagent Grade benzene was further purified by drying over phosphorus pentoxide for several weeks. The anhydrous benzene then was fractionated, and the portion boiling at 80.1° at 765 mm. was collected; $n^{25}_{\rm D}$ 1.49825 (previously published value, $n^{25}_{\rm D}$ 1.49821[6]).

Dipole Moment.—Dielectric constants of dilute solutions in benzene were measured with a modified heterodyne–beat apparatus which was similar to that which has been described by Stranathan.[7] By employment of the method of calculation which has been described by Smyth[8], the dipole moment of ethylgermanium trichloride was evaluated. In the calculation of the electric moment the dielectric constant of benzene was taken as 2.273[9] at 25.0°.

In Table I are presented the observed data which were used in the calculation of the dipole moment.

TABLE I

Mole fraction of solute c_2	Density d, g./cm.³	Dielectric constant E	Total molar orientation polarization P_2, cm.³
0.011649	0.8838_6	2.422	217.83
.007887	$.8795_9$	2.373	217.44
.004905	$.8761_5$	2.328	196.98
.002150	$.8733_2$	2.294	169.11
		$P_{2\infty}$ = 145.0	

In this table $P_{2\infty}$ represents the total molar orientation at infinite dilution (obtained by graphical extrapolation). If the sum of the electronic and atomic polarizations is taken as 1.05 $R^{25}_{\rm D}$, *i.e.*, 38.35 cm.³, the dipole moment of ethylgermanium trichloride is calculated to be 2.28 ± 0.09 D. This value is about 10% higher than those of most alkyl chlorides, and compares with 2.06 D for 1,1-dichloropropane and 2.028 for n-propyl chloride.[10] No value for 1,1,1-trichloropropane is available.[10]

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Preparation and Polymerization of Aryl Methacrylates and N-Arylmethacrylamides

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In contradistinction with aliphatic esters and amides of methacrylic acid and with aralkyl methacrylates,[1] only very few aryl methacrylates and N-arylmethacrylamides have been described.[2]

In the framework of a larger investigation, the preparation of the polymers of aryl methacrylates and of N-arylmethacrylamides was desired. The normal method of transesterification of methyl methacrylate failed, when applied to the preparation of the aryl methacrylates; good results were achieved, when methacrylyl chloride was treated with the appropriate sodium phenoxides. In the

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