ways were recovered unchanged. No olefins or condensation products of the haloethanes could be found, either in the reaction mixture, or as gaseous products of the reaction.

1,2-Dichloro-1,1,2-trifluoro-2-iodoethane.—Chlorotrifluoroethylene (88 g.) was bubbled through a mixture of 162 g. of iodine monochloride and 500 ml. of 1,1,2-trichloro-1,2,2-trifluoroethane (Freon-113) during the course of 5 hours. As the reaction proceeded the iodine chloride passed into solution, and the temperature of the reaction mixture rose to 35°. After the absorption of the olefin had ceased, the reaction mixture was washed with sodium thiosulfate solution, and then with sodium carbonate solution until colorless, and was dried over sodium carbonate. Fractional distillation of the solution gave 119 g., 57% of theory, of 1,2-dichloro-1,1,2-trifluoro-2-iodoethane boiling 43–44° at 100 mm., $n^{25}{}_D$ 1.4474, $d^{25}{}_{20}$ 2.1959. *Anal.* Calcd. for $C_2Cl_2F_3I$: C, 9.29; mol. wt., 279; *MR*D 34.3. Found: C, 8.87; mol. wt., 271, determined in n-C_7F_{16}; *MR*D 34.0. This material was stored in the dark at room temperature for several months without excessive decomposition. The above structure was assigned by analogy to other addition reactions with this olefin.[4]

A much less stable compound, thought to be 1-chloro-1,2,2-trifluoro-1,2-diiodoethane, was obtained in 30–35% yield by the distillation of a mixture of equal weights of chlorotrifluoroethylene and iodine which had stood at room temperature for several days sealed in a Pyrex reactor. This material, boiling 54–55° at 20 mm., with slight decomposition, and having a density of greater than 2.5, decomposed readily upon standing to yield crystals of iodine, but no other residue. A reliable analysis or refractive index could not be obtained because of this instability.

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[4] W. E. Hanford and G. W. Rigby, U. S. Patent 2,409,274, issued October, 1946; C. W. Huskins and P. Tarrant, "Preparation and Reactions of Certain Chlorofluoroethylenes," Report of Office of Naval Research under Contract N8onr503, June 15, 1946; J. A. Young and P. Tarrant, THIS JOURNAL, **71**, 2432 (1949); K. E. Rapp, *et al.*, *ibid.*, **72**, 3642 (1950); R. L. Pruett, *et al.*, *ibid.*, **72**, 3646 (1950); J. T. Barr, *et al.*, *ibid.*, **72**, 4480 (1950).

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An Improved Synthesis of Mescaline

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In connection with certain studies of the synthesis of phenethylamines, we have had the occasion to prepare mescaline, β-(3,4,5-trimethoxyphenyl)-ethylamine, an alkaloid which occurs naturally in the cacti *Anhalonium*. The literature cites the synthesis of this alkaloid by two methods, neither of which gives a satisfactory yield.

Slotta and Heller[1] synthesized mescaline in four steps from 3,4,5-trimethoxybenzaldehyde in an over-all yield of 28%. A more direct method *via* the condensation of 3,4,5-trimethoxybenzaldehyde with nitromethane, followed by the reduction of the resulting nitrostyrene, was employed by Späth.[2] The nitrostyrene was first reduced to the corresponding aldoxime, which on further reduction gave mescaline. The two-stage reduction gave a 24.9% yield, and the over-all yield of mescaline from the aldehyde was only 20.8%.

An elegant method for reduction of β-nitrostyrenes to phenethylamines with lithium aluminum hydride has been reported.[3] Reduction of 3,4,5-trimethoxy-β-nitrostyrene by this method resulted in an 89% yield of mescaline hydrochloride. The over-all yield from 3,4,5-trimethoxybenzaldehyde has been increased to 65%.

Experimental[4]

3,4,5-Trimethoxybenzaldehyde.—Gallic acid was converted to trimethylgallic acid in 80% yield by methylation with methyl sulfate.[5] Trimethylgalloyl chloride was prepared in 81% yield by the action of phosphorus pentachloride on trimethylgallic acid.[1] The acid chloride was purified by distillation and redistillation under reduced pressure; b.p. 131–133° (2 mm.); m.p. 83–84°. Rosenmund reduction of 3,4,5-trimethoxybenzaldehyde was carried out as described[1] using 52 g. of 3% Pd–$BaSO_4$ catalyst per mole of acid chloride. Dry xylene was the solvent. The product was recovered by removal of the catalyst by filtration, stripping off the xylene and fractional distillation of the residue under reduced pressure. A forerun of pyrogallol trimethyl ether, b.p. 82–100° (0.1 mm.), was obtained, and 3,4,5-trimethoxybenzaldehyde was collected as the fraction boiling at 106–112° (0.1 mm.). After crystallization from alcohol the aldehyde melted at 73–74°; yield 59%.

3,4,5-Trimethoxy-β-nitrostyrene.—This compound was prepared by the procedure of Späth[2] in 82.7% yield; m.p. 121–122° after recrystallization from alcohol.

Mescaline Hydrochloride.—Reduction of 7.2 g. (0.03 mole) of 3,4,5-trimethoxy-β-nitrostyrene with 5.7 g. of lithium aluminum hydride was carried out as described[3a] for 4-hydroxy-3-methoxy-β-nitrostyrene. The yield of mescaline picrate, m.p. 210–212°, was 11.4 g. (86%). After recrystallization from alcohol, the picrate melted at 214–216°; reported[2] 216–218°. The mescaline picrate was converted to mescaline hydrochloride as described[3a] in 92% yield; m.p. 180–181° (after recrystallization from alcohol); reported[1] 181°.

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[1] K. H. Slotta and H. Heller, *Ber.*, **63**, 3029 (1930).

[2] E. Späth, *Monatsh.*, **40**, 144 (1919).

[3] (a) F. A. Ramirez and A. Burger, THIS JOURNAL, **72**, 2781 (1950); (b) R. F. Nystrom and W. G. Brown, *ibid.*, **70**, 3738 (1948); (c) K. E. Hamlin and A. W. Weston, *ibid.*, **71**, 2210 (1949).

[4] All melting and boiling points uncorrected.

[5] H. Gilman and A. H. Blatt, "Organic Syntheses," John Wiley and Sons, Inc., New York, N. Y., 1941, Coll. Vol. I, 2nd Ed., p. 537.

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Preparation of *o*-Bromobenzotrifluoride

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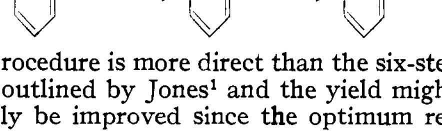
In view of the recent interest in ortho and para derivatives of benzotrifluoride,[1,2] we are hereby reporting a two-step synthesis of *o*-bromobenzotrifluoride.

Metallation of benzotrifluoride with *n*-butyllithium has been shown to yield largely the ortho isomer.[3] Treatment of the metallated product with bromine vapor forms *o*-bromobenzotrifluoride in a 28% over-all yield.



This procedure is more direct than the six-step synthesis outlined by Jones[1] and the yield might conceivably be improved since the optimum reaction conditions were not investigated. As a proof of structure, *o*-bromobenzotrifluoride was found to undergo halogen–metal interconversion with *n*-butyllithium. The product upon carbonation was *o*-trifluoromethylbenzoic acid. It is of interest that

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[1] R. Jones, THIS JOURNAL, **69**, 2346 (1947).

[2] M. Lilyquist, J. G. Wisler and P. Tarrant, Abstracts of 118th Meeting of the American Chemical Society, Chicago, Illinois, 1950.

[3] J. D. Roberts and D. Y. Curtin, THIS JOURNAL, **68**, 1658 (1946).