623. *Organometallic Fluorine Compounds. Part I. The Synthesis of Trifluoromethyl and Pentafluoroethyl Mercurials.*

By H. J. Emeléus and R. N. Haszeldine.

The reaction between iodine pentafluoride and di-iodotetrafluoroethane yields iodopentafluoroethane quantitatively. Iodotrifluoromethane and mercury, when irradiated and heated, form trifluoromethylmercuric iodide, from which *trifluoromethylmercuric hydroxide, chloride, bromide,* and *nitrate* have been prepared. These compounds are soluble in water, and preliminary observations on the conductivities of the solutions are recorded. Iodopentafluoroethane undergoes a similar reaction with mercury and forms pentafluoroethylmercuric iodide, which is also soluble in water. Unsuccessful attempts to prepare fluorine-containing organometallic derivatives of magnesium, zinc, cadmium, lead, arsenic, lithium, and gallium are described.

The synthesis of iodotrifluoromethane and iodopentafluoroethane (Banks, Emeléus, Haszeldine, and Kerrigan, *J.*, 1948, 2188) leads naturally to the study of how far these compounds resemble their hydrocarbon analogues in forming organometallic derivatives. Iodotrifluoromethane can now be prepared from tetraiodomethane and iodine pentafluoride in yields of over 90%. The original synthesis of iodopentafluoroethane was from tetraiodoethylene and iodine pentafluoride. Yields were, however, low, and a greatly improved method has been found in which tetrafluoroethylene is converted into *di-iodotetrafluoroethane* which, on treatment with iodine pentafluoride, yields iodopentafluoroethane quantitatively.

The reactions of iodotrimethane and iodopentafluoroethane with metals differed greatly from those of methyl and ethyl iodides.

Iodotrifluoromethane, for example, was recovered unchanged after being in contact with magnesium for several weeks. The normal initiators for the formation of Grignard reagents also failed to induce reaction. Heating of the reactants either alone or with solvents led ultimately to decomposition. Addition of water at various stages of the reaction gave no fluoroform, which would have been expected had the compound $CF_3{\cdot}MgI$ been present. At higher temperatures, however, small amounts of fluoroform were produced in absence of water by attack of the fluorocarbon radical on the solvent. Since no hexafluoroethane was isolated, neither the Wurtz-type reaction nor the decomposition of any Grignard reagent by excess of trifluoroiodomethane took place. Parallel experiments with zinc and a few preliminary experiments with cadmium, gallium, lithium, and alloys of sodium and lead or arsenic were also negative. The slow decomposition of iodotrifluoromethane by lithium in a hydrocarbon solvent was similar to that observed with chlorotrifluoromethane in trimethylamine as solvent (Simons, Bond, and McArthur, *J. Amer. Chem. Soc.*, 1940, **62**, 3477).

The reaction of iodopentafluoroethane with magnesium and zinc was more complex than that of iodotrifluoromethane. The reactants in benzene solution or in the absence of a solvent did not react at room temperature, and when the temperature was raised decomposition to metal iodide and fluoride eventually occurred; small amounts of pentafluoroethane were isolated when a solvent was used. The reaction of the fluoro-iodide in ether with magnesium or zinc was, however, rapid and complete at room temperature. The usual indications of Grignard formation were given initially, but the solution rapidly became brown, heat was evolved, and the final reaction mixture was similar to that from iodotrifluoromethane and magnesium or zinc at higher temperatures. The products have not been fully identified, but they included the metal iodide and fluoride, viscous polymeric substances containing carbon and fluorine only, and comparatively large amounts of pentafluoroethane. Pentafluoroethane was not liberated when water was added to the reaction mixture but it is possible that in these two cases unstable Grignard compounds were formed.

The stabilising influence of the di- and tri-fluoromethyl groups on adjacent hydrogen and other halogen atoms in a molecule is now well known, and the ease of formation of Grignard reagents from aliphatic halides containing these groups clearly depends on the distance of the halogen from the fluoro-group. The influence of a trifluoromethyl group extends to a chlorine atom on position 2, since 2-chloro-1 : 1 : 1-trifluoroethane will not yield a Grignard reagent. The influence on a chlorine atom in position 3 is much less, however, and McBee and Truchan (*J. Amer. Chem. Soc.*, 1948, **70**, 2910) observed that 3-chloro-1 : 1 : 1-trifluoropropane reacts normally with magnesium. The only recorded case of Grignard formation involving a halogen α to a fluorinated group is that of 1-bromoheptafluoropropane, C_3F_7Br (Brice, Pearlson, and Simons, *ibid.*, 1946, **68**, 968), a reaction of particular interest in view of the inability of 2-bromo-1 : 1 : 1-trifluoroethane to form a Grignard reagent. The iodofluoroethanes react readily with magnesium in the presence of ether at room temperature but the products are olefins and not organometallic compounds. Thus 2-iodo-1 : 1 : 1-trifluoroethane yields 1 : 1-difluoroethylene (Gilman, *ibid.*, 1943, **65**, 2037) and 2-iodo-1 : 1-difluoroethane yields fluoroethylene (Henne, *ibid.*, 1938, **60**, 2275). The corresponding bromo-compound, 2-bromo-1 : 1-difluoroethane was stable to magnesium, but gave the olefin when treated with sodium or potassium. In view of these facts it may seem surprising that iodopentafluoroethane did not yield tetrafluoroethylene when treated with magnesium under Grignard conditions. It is probable, however, that any tetrafluoroethylene would undergo polymerisation with excess of iodopentafluoroethane (Haszeldine, in the press).

The inability of iodotrifluoromethane to yield a Grignard reagent under normal conditions is attributed to the increased stability of the carbon–iodine bond and its failure to undergo heterolytic fission. At higher temperatures the main reaction is decomposition, but in addition homolytic fission of the carbon–iodine bond occurs to a certain extent and the trifluoromethyl radical is formed. Like other free radicals the fluorocarbon radical reacts with the solvent in preference to magnesium or zinc, and fluoroform is liberated. Thus it is known that free phenyl radicals will not react with magnesium in solution, nor will free alkyl radicals combine with magnesium in the gas phase. Elements such as mercury, arsenic, tellurium, and antimony, on the other hand, readily combine with such free radicals and form stable organometallic derivatives. The experiments described below do in fact show that iodotrifluoromethane and iodopentafluoroethane yield stable mercurials of the type $R{\cdot}HgI$, where R is the fluorocarbon radical, and the investigation is being extended to the other elements mentioned. The requisite experimental conditions are outlined later; in general, however, it is necessary to initiate reaction with mercury by heat or irradiation, or both. Hydrogen-containing solvents are attacked by the radicals, but perfluoromethyl*cyclo*hexane was used successfully. During the reaction with mercury the Wurtz type of reaction, leading to hexafluoroethane or decafluorobutane, does not occur to any great extent, but there is a reaction of the fluorocarbon radicals with the glass reaction vessel to form silicon tetrafluoride and carbon dioxide.

Trifluoromethyl- and pentafluoroethyl-mercuric iodide show a general resemblance to their hydrocarbon analogues, but both are soluble in water and may be recrystallised from it, although a slow decomposition occurs. They react when heated with excess of iodine to yield the original fluoro-iodide and mercuric iodide. The free base, *trifluoromethylmercuric hydroxide*, obtained from the iodide by interaction with moist silver oxide, is alkaline to phenolphthalein and forms salts with acids. *Trifluoromethylmercuric chloride, bromide*, and *nitrate* were thus prepared. The nitrate was also prepared by the action of silver nitrate on the iodide. Trifluoromethylmercuric hydroxide is a weaker electolyte than methylmercuric hydroxide, conductivity data for which are given by Slotta and Jacobi (*J. pr. Chem.*, 1929, **120**, 249), Hein and Meininger (*Z. anorg. Chem.*, 1925, **145**, 95), and Maynard and Howard (*J.*, 1923, **123**, 960), but a stronger electrolyte than phenylmercuric hydroxide (Schramm, *J. Amer. Chem. Soc.*, 1947, **69**, 1831). The conductivity of trifluoromethylmercuric nitrate, on the other hand, approaches that of a neutral strong electrolyte and is greater than that of either methyl- or phenyl-mercuric nitrate. This high conductivity is due to the hydrolysis of the nitrate which is strongly acidic in aqueous solution.

EXPERIMENTAL.

Reaction of Iodine Pentafluoride with Di-iodotetrafluoroethane.—Tetrafluoroethylene (1·56 g.) was condensed into a Pyrex tube containing excess of iodine, and the tube was sealed *in vacuo*. After being heated at 175° for 14 hours, 70% of the tetrafluoroethylene had reacted. The product (3·85 g.) was *di-iodotetrafluoroethane*, b. p. 113°, n_D^{23} 1·482 (Found : I, 71·0%; *M*, 348. $C_2I_2F_4$ requires I, 71·7%; *M*, 354). The yield was quantitative in terms of the tetrafluoroethylene used. When heated at 200° for 24 hours, 91% of the tetrafluoroethylene reacted. In a typical experiment di-iodotetrafluoroethane (6·7 g.) was poured on excess of iodine pentafluoride (5 ml.) which had been frozen in liquid air in a small Pyrex flask fitted with a vertical reflux condenser leading to a trap cooled in liquid air. The reactants were allowed to reach room temperature, and the exothermic reaction was initiated by warming. After the initial reaction had subsided the product was heated under reflux for 15 minutes. The residue in the flask was iodine mixed with excess of the pentafluoride and was refluorinated for further preparations. The volatile products were silicon tetrafluoride and iodopentafluoroethane (4·65 g.), b. p. 13° (Found : I, 51·3%; *M*, 246. Calc. for C_2IF_5 : I, 51·6%; *M*, 246. The yield of the fluoro-iodide was quantitative. The following vapour pressures were recorded : −24·6°, 215 mm.; 0°, 538 mm.; 4·0°, 616 mm.; 7·0°, 681 mm.; 9·8°, 717 mm.

Reaction of Iodotrifluoromethane with Mercury.—The experiments were made in sealed tubes containing 10 c.c. of mercury together with the fluoro-iodide. The reactants were either heated in a rocking furnace or shaken vigorously in a vibro-shaker while exposed to ultra-violet radiation. In some experiments the tubes were fitted with coils of heating wire wound on an open frame so that they could be irradiated at elevated temperatures. With heat alone to initiate reaction, the optimum conditions with 4·5 g. of iodotrifluoromethane were 260—290° for 12 hours. Below 200° reaction was very slow and 90% of the fluoro-iodide was recovered unchanged after 10 hours. Above 300°, or with longer reaction times, considerable decomposition of the iodotrifluoromethane and the mercurial occurred. Under the optimum conditions about 50% of the fluoro-iodide reacted to yield 1 g. (22%) of trifluoromethylmercuric iodide (Found : C, 3·2; I, 32·1; Hg, 50·4. Calc. for CIF_3Hg : C, 3·0; I, 32·0; Hg, 50·6%). Other products identified were mercury iodides, silicon tetrafluoride, carbon dioxide, and hexafluoroethane. No mercuric or mercurous fluoride was detected. The unchanged fluoro-iodide was purified by treatment with aqueous alkali and fractionation. The solid contents of the reaction vessel were extracted with ether, and the crude trifluoromethylmercuric iodide so obtained was purified by sublimation.

In irradiating reaction mixtures the full radiation of a Hanovia lamp 12″ from the vessel was used. Reaction was slow at room temperatures with Pyrex vessels. In silica tubes the rate was greater but the reaction product itself was extensively decomposed by light of short wave-length. It was possible to shield a portion of the reaction tube from irradiation and to adjust the shaker so that much of the solid product collected in this zone. In a typical experiment iodotrifluoromethane (9·0 g.), irradiated in Pyrex for 165 hours while being shaken with excess of mercury, yielded 3·04 g. of trifluoromethylmercuric iodide, a 50% yield on the fluoroiodide used (35%). The effects of pressure, temperature, and time of irradiation were examined. Increase in pressure appeared to increase the yield of mercurial. It was not possible, however, to seal more than about 10 g. of the fluoro-iodide in the 50-c.c. reaction tubes used without risk of explosion on heating them. The optimum temperature with this amount and excess of mercury was 150°; above this temperature excessive decomposition occurred. Irradiation for 7—8 days, during which time the temperature was gradually raised to 150°, was found to be suitable. Under these conditions in a typical experiment in Pyrex, 10·5 g. of trifluoroiodomethane yielded 11·8 g. of trifluoromethylmercuric iodide (80% yield on the iodotrifluoromethane used). Only small amounts of mercuric iodide and volatilile by-products were formed.

Reaction in Presence of a Solvent.—Acetone, ether, alcohol, benzene, hexane, and carbon tetrachloride were unsuitable as solvents for the reaction between iodotrifluoromethane and mercury because of the preferential attack of the trifluoromethyl radical on the solvent. Iodotrifluoromethane was, however, soluble in perfluoromethyl*cyclo*hexane, which was not attacked by the fluorocarbon radical. Good yields of the mercurial were obtained, although reaction was slower than in absence of a solvent. In a typical experiment iodotrifluoromethane (7·5 g.) dissolved in perfluoromethyl*cyclo*hexane (4 c.c.) was heated in a Pyrex tube to 110° for 36 hours, and shaken with mercury while exposed to the mercury arc. Crystals of the mercuri-iodide were deposited on cooling. Removal of the solvent and extraction with ether gave 7·5 g. of pure trifluoromethylmercuric iodide, an 80% yield based on the iodotrifluoromethane used (4·7 g.).

Reactions of Trifluoromethylmercuric Iodide.—The compound is a white crystalline solid which sublimes at 80°. It has a characteristic odour quite distinct from that of methylmercuric iodide and bistrifluoromethylmercury. The mercuri-iodide is decomposed by light of short wave-length. When heated at 120° for 2 hours with excess of iodine, trifluoromethylmercuric iodide (1·0 g.) yielded 92% of the theoretical amount of iodotrifluoromethane. The mercurial is readily soluble in organic solvents such as ether, acetone, alcohol, and *cyclo*hexane, but sublimation has been found more suitable for purification than crystallisation. The mercurial is also soluble in water, from which it may be extracted with ether. Mercurous and mercuric iodide are slowly deposited from the aqueous solution, and during the decomposition a gas is evolved which is probably a mixture of hexafluoroethane and fluoroform; no trifluoromethylmercuric hydroxide (see below) is formed and the reaction is not therefore a simple hydrolysis. The specific conductivity at 25° of an M/8-solution of the mercurial in water is $5{\cdot}5 \times 10^{-5}$ ohm^{-1} cm.$^{-1}$, but this value increases as the decomposition reaction proceeds. An aqueous solution of trifluoromethylmercuric iodide (0·005 mol.), when treated with a solution of potassium iodide (0·01 mol.), slowly gave fluoroform, the yield of which was 60% after 3 days and 72% after 10 days. The solution became alkaline and yellow, and the complex K_2HgI_4 was formed $(CF_3{\cdot}HgI + 3I^- + H_2O \longrightarrow CHF_3 + OH^- + HgI_4^{--})$.

Trifluoromethylmercuric Hydroxide.—This was prepared by shaking trifluoromethylmercuric iodide (4·0 g.) for 2 hours with a suspension of excess of silver oxide in conductivity water, air being excluded to avoid formation of carbonate. The silver compounds were filtered off and the crude base was isolated by evaporation at room temperature *in vacuo*. It was purified without detectable decomposition by sublimation at 130°/10^{-5} mm. The purified *hydroxide* (2·6 g., 90%) gave a negative test for iodide [Found: C, 4·2; Hg, 69·8%; *M* (ebullioscopic), 280. $CHOF_3Hg$ requires C, 4·2; Hg, 70·0%; *M*, 287].

Trifluoromethylmercuric Chloride.—The *chloride* was prepared by dissolving 0·5 g. of the hydroxide in 5 c.c. of water and acidifying the solution with dilute hydrochloric acid to pH 1. The aqueous solution was extracted with ether, and the ethereal extract allowed to evaporate at room temperature. The residual solid was purified by sublimation at 70°/1 atm. (0·48 g., 91%). The compound, m. p. 76° (sealed tube), resembled the mercuri-iodide in smell and crystal form [Found: C, 3·8; Cl, 11·2%; *M* (ebullioscopic), 306. $CClF_3Hg$ requires C, 3·9; Cl, 11·6%; *M*, 305].

Trifluoromethylmercuric Bromide.—The *bromide* was similarly prepared in 80% yield (Found: C, 3·6; Br, 22·6%; *M*, 340. $CBrF_3Hg$ requires C, 3·4; Br, 22·9%; *M*, 350).

Trifluoromethylmercuric Nitrate.—A solution of the hydroxide was acidified with dilute nitric acid and evaporated to dryness *in vacuo* at room temperature, the residual solid *nitrate* being purified by repeated sublimation at 100°/10^{-5} mm. Alternatively trifluoromethylmercuric iodide (7·0 g.) was dissolved in water and treated with a slight excess of silver nitrate; the precipitated silver iodide was filtered off, dilute nitric acid added to prevent the formation of the possible double salt $CF_3{\cdot}HgNO_3,CF_3{\cdot}HgOH$ (cf. $C_6H_5{\cdot}HgNO_3,C_6H_5{\cdot}HgOH$), and the solution evaporated as above (Found: C, 3·6; N, 3·8; F, 16·9; Hg, 60·3. CO_3NF_3Hg requires C, 3·6; N, 4·2; F, 17·2; Hg, 60·5%).

Conductivity of Trifluoromethyl Mercurials.—Conductivity measurements on aqueous solutions of the trifluoromethyl mercurials were made at 25° ± 0·02° with a standard bridge apparatus incorporating a cathode-ray oscilloscope as the null-point detector, and Pyrex cells with platinum electrodes. Solutions were made up initially by weight in conductivity water (specific conductivity $1{\cdot}4$—$1{\cdot}5 \times 10^{-6}$ ohm^{-1} cm.$^{-1}$) and diluted by volume. Results for trifluoromethylmercuric hydroxide are given in Table I.

TABLE I.

Molarity	M/4.	M/8.	M/16.	M/32.	M/64.	M/128.	M/256.	M/512.
Sp. conductivity × 10^6 (ohm^{-1} cm.$^{-1}$)	32	18	11	7	4	3	2·5	2
Molar conductivity × 10^2	13	15	18	22	26·5	40	64	106

Cf. M/16-methylmercuric hydroxide, specific conductivity 134×10^{-6} ohm^{-1} cm.$^{-1}$ (Hein and Meininger, *loc. cit.*).

Results for trifluoromethylmercuric nitrate are similarly summarised in Table II.

TABLE II.

Molarity.	M/2.	M/4.	M/8.	M/16.	M/32.	M/64.	M/128.	M/256.	M/512.	M/1024.	M/2048.
Sp. conductivity × 10^4 (ohm^{-1} cm.$^{-1}$)	496	277	160	90	50	28	16	9	5	3	2
Molar conductivity	100	111	128	144	161	176	205	240	278	318	355

Cf. specific conductivity of M/16-methylmercuric nitrate 66×10^{-4} ohm^{-1} cm.$^{-1}$, M/1024-phenylmercuric nitrate 1×10^{-4} ohm^{-1} cm.$^{-1}$, M/100-KCl 14×10^{-4} ohm^{-1} cm.$^{-1}$.

Reaction of Iodotrifluoromethane and Iodopentafluoroethane with Magnesium.—Iodotrifluoromethane was condensed into a tube containing dry ether and magnesium, the tube then being sealed either when evacuated or when filled with pure nitrogen. No change occurred on warming the mixture to room temperature, and after 6 weeks 91% of the fluoro-iodide was unchanged. Further experiments with added Grignard catalysts (iodine and methylmagnesium iodide) or using a copper–magnesium alloy failed to yield a fluoro-organomagnesium compound. The effect of temperature on the uncatalysed reaction was investigated: after 30 hours at 80°, 15 hours at 120°, 140°, and 180° the proportions of unchanged fluoro-iodide recovered were 89, 80, 64, and 5%, respectively. The ethereal solution was coloured brown and a flocculent solid consisting mainly of magnesium fluoride was present. No magnesium carbide was present since the addition of water did not liberate acetylene. The reaction products from experiments at 180° contained fluoroform in an amount corresponding to 5% of the fluoro-iodide taken. Addition of water to the reaction products under various conditions did not liberate fluoroform. Similar results were obtained when benzene and tetrahydropyran were used as solvent instead of ether. In the absence of a solvent there was negligible reaction after several weeks at room temperature. Above 160° magnesium iodide and fluoride were formed in increasing amounts, and silicon tetrafluoride, carbon dioxide, and small amounts of tetrafluoroethylene were identified. Even after heating to 260° for 12 hours, 85% of the fluoro-iodide was unchanged. Heating and irradiating iodotrifluoromethane and magnesium in the absence of a solvent brought about a slow decomposition at temperatures above 150°. When iodotrifluoromethane and magnesium were exposed to ultra-violet light in the presence of ether fluoroform was produced slowly at room temperature.

Magnesium and iodopentafluoroethane did not react at temperatures up to 150° in the absence of a solvent. In presence of ether, however, a ready reaction ensued at room temperature, and the magnesium began to dissolve. Within a few seconds, however, the colour deepened to a dark brown and a syrupy carbonaceous deposit formed. The iodopentafluoroethane was completely destroyed. It is possible that a true organometallic compound is formed initially in this case, but attempts to control its decomposition or reaction with the solvent have so far been unsuccessful.

Reaction of Iodotrifluoromethane and Iodopentafluoroethane with Zinc.—Zinc–copper couples which were active to ethyl iodide were prepared by the methods of Noller (*J. Amer. Chem. Soc.*, 1929, **51**, 594) and Gladstone (*J.*, 1873, **26**, 961), and from an alloy of zinc 90% and copper 10%. Iodotrifluoromethane was treated with the zinc–copper couples in toluene solution, with ethyl acetate as catalyst in some experiments. The fluoro-iodide was slowly decomposed when the mixture was kept and a small amount of fluoroform was isolated. No organozinc compound was, however, isolated. There was a reaction in absence of solvent only at elevated temperatures and it resembled that for magnesium under similar conditions. Preliminary experiments with iodopentafluoroethane showed that its reaction with zinc resembled that with magnesium. No reaction occurred in a hydrocarbon solvent, or in absence of solvent, at temperatures up to 80°. In ether there was an immediate and vigorous reaction in which the fluoro-iodide was completely destroyed. No organometallic compound was isolated, but a 40% yield of pentafluoroethane was obtained.

Reaction of Iodotrifluoromethane with Cadmium, Lead, Arsenic, Lithium, and Gallium.—Cadmium, arsenic, and gallium did not react with iodotrifluoromethane at room temperature in the presence or absence of a solvent, and increasing the temperature brought about the slow decomposition of the fluoro-iodide. A lead–sodium alloy with benzene as solvent decomposed 17% of the fluoro-iodide after 14 hours at 65°. Fluoroform was isolated in 6% yield, together with a small amount of an oil containing carbon, hydrogen, and fluorine, but no lead. Similarly when an arsenic–sodium alloy was allowed to react with iodotrifluoromethane in benzene solution for 56 hours at 150°, practically all of the fluoro-iodide was decomposed and a 33% yield of fluoroform was obtained. Iodotrifluoromethane in benzene was slowly decomposed by lithium ribbon at room temperature. Reaction was more rapid at 70° but the only products isolated were lithium fluoride and iodide. These preliminary experiments indicate therefore that iodotrifluoromethane does not yield organometallic compounds by the standard methods and techniques applicable to methyl iodide.

Reaction of Iodopentafluoroethane with Mercury.—When iodopentafluoroethane (3·0 g.) was irradiated and shaken with mercury a slow reaction occurred, 40% of the fluoro-iodide reacting in 17 days to form 0·06 g. of the mercurial (see below). Increase in temperature raised the yield, and the optimum condition, starting from 8·0 g. of iodopentafluoroethane, was irradiation for 5—6 days at 120°, a 50—55% yield of pentafluoroethylmercuric iodide being then formed, based on the fluoro-iodide used (50—60%). This optimum temperature was lower than that for iodotrifluoromethane. The mercurial was more readily decomposed by light, and, unless the shaking conditions were adjusted so that the compound was deposited in a section of the tube shielded from direct irradiation, the yields were low. The volatile by-products consisted mainly of silicon tetrafluoride, carbon dioxide, and small amounts of perfluorobutane. Mercury iodides, but no mercury fluorides, were also formed. The pentafluoroethylmercuric iodide was extracted from the reaction products with ether and purified by sublimation (Found: I, 28·0; Hg, 44·7. Calc. for C_2IF_5Hg: I, 28·3; Hg, 44·9%). A more convenient method of preparation was by the purely thermal reaction of the fluoro-iodide with mercury. The optimum temperature range was 200—240°, and 8·0 g. of pentafluoroiodoethane in 36 hours gave a 60—65% yield of the mercurial based on the fluoro-iodide used (50—60%). Pentafluoroethylmercuric iodide (2·0 g.), when allowed to react with excess of iodine at 120° for 4 hours, gave a 96% yield of iodopentafluoroethane. The mercurial is soluble in water and in a variety of organic solvents.

UNIVERSITY CHEMICAL LABORATORY, CAMBRIDGE. [*Received, July* 29*th*, 1949.]