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THE SYNTHESIS OF SOME (PENTAFLUOROPHENYL)GERMANIUM COMPOUNDS

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SUMMARY

Tris(pentafluorophenyl)germanium bromide reacts with lithium aluminium hydride or triethylgermane to give tris(pentafluorophenyl)germane. On reaction with HgR_2 [where R = Et, Et_3Ge and $(Me_3Si)_2N$] or reaction with diethylcadmium, the latter gave the hitherto unknown $[(C_6F_5)_3Ge]_2Hg$ or $[(C_6F_5)_3Ge]_2Cd$. The germylmercurial compound could also be prepared by reaction of metallic mercury with $[(C_6F_5)_3Ge]_2Cd$ and by exchange of bis(triethylgermyl)mercury with $(C_6F_5)_3GeBr$. Some reactions of bis[tris(pentafluorophenyl)germyl]mercury are discussed.

INTRODUCTION

It is well known that the properties of bimetallic organometallic compounds are essentially dependent on the type of organic radical attached to the metal–metal group. For example, the reaction of $(Me_3SiCH_2)_3SnH$ and diethylmercury at room temperature affords bis{tris[(trimethylsilyl)methyl]stannyl}mercury, $[(Me_3SiCH_2)_3Sn]_2Hg$[1], which is very stable relative to the related simple alkyl or phenyl derivatives $(R_3Sn)_2Hg$[2–4]. In contrast to the stable compound $Et_3GeHgCF(CF_3)_2$[5,6], (triethylgermyl)(perfluoro-tert-butyl)mercury, $Et_3GeHgC(CF_3)_3$, decomposes during synthesis at 20°[7].

To date, little of the chemistry of (pentafluorophenyl)germanium compounds has been studied[8]. Hence, using bis[tris(pentafluorophenyl)germyl]mercury, $[(C_6F_5)_3Ge]_2Hg$(I), and related compounds as examples, it would appear interesting to investigate the effect of C_6F_5 substituents upon the properties of compounds containing germanium–metal groups.

RESULTS AND DISCUSSION

The previously unknown tris(pentafluorophenyl)germane (II) may be produced in high yield by an exchange reaction between tris(pentafluorophenyl)germanium bromide (III) and triethylgermane in the absence of solvents or, alternatively, during the reduction of (III) with lithium aluminium hydride in an ether/toluene mixture. Unlike non-fluorinated organogermanes which may be readily brominated at the Ge–H bonds[9,10], compound (II) only reacts with bromine under conditions sufficiently drastic to give germanium bromide, (III), and hydrogen bromide.

On the other hand, tris(pentafluorophenyl)germane (II) reacts readily with diethylmercury or bis[bis(trimethylsilyl)amido]mercury in benzene or toluene to afford the germylmercury compound (I)*:

$$\underset{(II)}{2\,(C_6F_5)_3GeH} + R_2Hg \rightarrow 2\,RH + \underset{(I)}{[(C_6F_5)_3Ge]_2Hg} \tag{1}$$

$[R = Et \text{ or } (Me_3Si)_2N]$

Reaction with the amidomercury compound is completed within 10–15 min at room temperature. With diethylmercury, the reaction also proceeds at room temperature but completion requires heating on a water bath. In this respect compound (II) was found to be more reactive than $(C_6H_5)_3GeH$[12] and $(C_6F_5)_3SiH$[13]. In comparison, tris(pentafluorophenyl)silane does not react with diethylmercury even at a temperature of 140°, but with $[(Me_3Si)_2N]_2Hg$ the compound bis[tris(pentafluorophenyl)silyl]-mercury is produced but only after heating for many hours at 150–170°[13].

If reaction (1) involving diethylmercury and (II) is carried out using an equimolar ratio of the reactants, selective substitution of one ethyl group by a $(C_6F_5)_3Ge$ fragment occurs, the yield of asymmetric ethyl[tris(pentafluorophenyl)germyl]-mercury (IV) thus obtained being 38.4%.

We have shown that the germylmercury compound (I) can also be prepared in satisfactory yields via the following reactions:

$$2\,(C_6F_5)_3GeH + (Et_3Ge)_2Hg \xrightarrow[140°]{} 2\,Et_3GeH + [(C_6F_5)_3Ge]_2Hg \tag{2}$$

$$2\,(C_6F_5)_3GeBr + (Et_3Ge)_2Hg \xrightarrow[h\nu]{\text{Benzene}} 2\,Et_3GeBr + [(C_6F_5)_3Ge]_2Hg \tag{3}$$

$$\underset{(V)}{[(C_6F_5)_3Ge]_2Cd} + Hg \xrightarrow[100°]{\text{Toluene}} Cd + \underset{(I)}{[(C_6F_5)_3Ge]_2Hg} \tag{4}$$

It is conceivable that these exchange reactions are reversible. The yields of compound (I) in reactions (2), (3) and (4) were found to be 40, 75 and 34%, respectively.

The synthesis of bis[tris(pentafluorophenyl)germyl]cadmium (V) has been achieved by means of a reaction similar to that depicted in (1), *i.e.* by reaction of diethylcadmium with 2 moles of tris(pentafluorophenyl)germane in toluene at 80–100°.

The germylmercury compound (I) (m.p. 228–231°) is a colourless crystalline solid which does not oxidize upon short contact with air. Photochemical decomposition of this compound in toluene with UV irradiation filtered through Pyrex glass affords metallic mercury, hexakis(pentafluorophenyl)digermane (VI), tris-(pentafluorophenyl)germane (II) and benzyltris(pentafluorophenyl)germane (VII). Compound (VII) was also synthesized via a Grignard reaction. We believe that the germylmercurial compound (I) decomposes homolytically to produce $(C_6F_5)_3Ge\cdot$ radicals. The latter then dimerize, abstract hydrogen from the solvent molecules or recombine with $C_6H_5CH_2\cdot$ radicals to form the germane (VII). Photolysis of $(Ph_3Si)_2$-Hg in toluene proceeds in a similar manner. The main products are Hg, Ph_6Si_2, Ph_3SiH, Ph_3SiCH_2Ph and $(PhCH_2)_2$[11b].

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* For a preliminary communication of the preparation of this compound see ref. 11a.

If this view is correct, the mercury compound (I) differs from bis(triphenylgermyl)mercury[12] and $[(C_6F_5)_3Si]_2Hg$[13]. Photolytic decomposition of the former in toluene gives only the dimerization product of $Ph_3Ge\cdot$ radicals (*i.e.* hexaphenyldigermane). With the second compound, $(C_6F_5)_3SiH$ is obtained. Hexakis(pentafluorophenyl)disilane was not present in the reaction mixture.

Bis(triphenylgermyl)mercury is known to react readily with hydrogen chloride in toluene to give mercury, triphenylgermane and triphenylgermanium chloride in almost quantitative yield. A similar mixture of products is obtained from (I) and hydrogen chloride in benzene. However, this reaction is slow and even after heating for 20 h at 100° (sealed tube) only 49% of (I) had reacted.

In a similar manner to bis[tris(pentafluorophenyl)silyl]mercury[13], (I) reacts with mercury chloride in THF:

$$\underset{(I)}{[(C_6F_5)_3Ge]_2Hg} + HgCl_2 \xrightarrow[100^\circ]{THF} 2\,(C_6F_5)_3GeHgCl \rightarrow 2\,Hg + 2\,(C_6F_5)_3GeCl \qquad (5)$$

The yields of mercury and tris(pentafluorophenyl)germanium chloride obtained from this reaction are 84 and 61%, respectively. It should be noted that the reaction of bis(trimethylsilyl)mercury and bis(triethylgermyl)mercury with mercury halides are both exothermic[6].

It is interesting that unlike reaction (5) the exchange reaction of (I) with bis(triethylgermyl)mercury occurs at room temperature and is completed in several minutes to give a high yield of the asymmetric compound (triethylgermyl)[tris(pentafluorophenyl)germyl]mercury (VIII).

$$\underset{(I)}{[(C_6F_5)_3Ge]_2Hg} + (Et_3Ge)_2Hg \xrightarrow[20^\circ]{Benzene} 2\,\underset{(VIII)}{(C_6F_5)_3GeHgGeEt_3} \qquad (6)$$

The germylmercury compound (VIII) may also be prepared in 76% yield together with triethylbromogermane from the reaction of equimolar quantities of $(C_6F_5)_3GeBr$ and bis(triethylgermyl)mercury in benzene:

$$(C_6F_5)_3GeBr + (Et_3Ge)_2Hg \xrightarrow[20^\circ]{Benzene} \underset{(VIII)}{(C_6F_5)_3GeHgGeEt_3} + Et_3GeBr \qquad (7)$$

Hence it follows that compound (VIII) is an intermediate in the exchange reaction (3).

The IR spectra of (I) and (II) and of (IV)–(VIII) exhibit the following absorption bands: 1650, 1525, 1480, 1390, 1290, 1090, 1025, 820 and 625 cm^{-1} specific to the C_6F_5 group[14]. In addition, the spectra of (IV) and (VIII) contain bands at 700–1500 cm^{-1} characteristic of EtHg and Et_3Ge groupings[15]. It should be noted that the stretching modes of Ge–C (Ge–Et) in (VIII) (575 and 538 cm^{-1}) and those of Hg–C in (IV) (500 cm^{-1}) are considerably greater in value than those in $(Et_3Ge)_2Hg$ (559 and 526 cm^{-1}) and $Et_3GeHgEt$ (473 cm^{-1}). This may be taken as evidence of the large $-I$ effect of the $(C_6F_5)_3Ge$ group. This leads to the high value of the Ge–H stretching mode (2223 cm^{-1}) in compound (II) together with its low intensity.

EXPERIMENTAL

All reactions were carried out in evacuated sealed ampoules or under argon.

GLC analyses of volatile products were carried out on a Tsvet-I chromatograph with a catharometer detector using a 100 × 0.4 cm column packed with Apiezon L (20%) on Chromosorb W or a 50 × 0.4 cm column packed with Thermol 3 (15%) on Symalite B and with helium as the carrier gas. The IR spectra were recorded on a UR-20 spectrophotometer. Samples of the crystalline compounds were prepared in the form of a suspension in Vaseline oil. Melting points were determined using sealed evacuated capillaries. Analytical details are presented in Table 1.

TABLE 1

ANALYTICAL DATA RELATING TO THE COMPOUNDS OBTAINED

No.	*Compound*	*Formula*	*M.p.* (°C)	*Analysis found (calcd.)(%)*			
				C	*F*	*Ge*	*Hg(Cd)*
(I)	$[(C_6F_5)_3Ge]_2Hg$	$C_{36}F_{30}Ge_2Hg$	228–231	32.23 (32.05)	42.39 (42.28)	10.71 (10.76)	14.99 (14.88)
(II)	$(C_6F_5)_3GeH$	$C_{18}HF_{15}Ge$	130–132		49.56 (49.58)		
(IV)	$(C_6F_5)_3GeHgEt$	$C_{20}H_5F_{15}GeHg$	118–121[a]		34.84 (35.47)		
(V)	$[(C_6F_5)_3Ge]_2Cd$	$C_{36}CdF_{30}Ge_2$	218–220		45.04 (45.23)		8.62 (8.92)
(VI)	$(C_6F_5)_3GeGe(C_6F_5)_3$	$C_{36}F_{30}Ge_2$	312–318		49.50 (49.66)		
(VII)	$(C_6F_5)_3GeCH_2C_6H_5$	$C_{25}H_7F_{15}Ge$	97–99		42.50 (42.86)		
(VIII)	$(C_6F_5)_3GeHgGeEt_3$	$C_{24}H_{15}F_{15}Ge_2Hg$	Oil[b]				21.20 (21.47)

[a] B.p. 135–145°/1 mmHg. [b] n_D^{20} 1.5522.

Tris(pentafluorophenyl)germane (II)

(*a*). Triethylgermane (12.01 g, 75.0 mmoles) and tris(pentafluorophenyl)-germanium bromide (5.00 g, 7.6 mmoles) were refluxed for 1 h. After cooling, the product separated as a colourless solid. The product was filtered, washed well with petroleum ether (b.p. 40–60°) and dried *in vacuo* to give 3.85 g (89.5%) of (II), m.p. 127–129°. A further recrystallization from hexane gave an analytical sample, m.p. 130–132°.

(*b*). A solution of tris(pentafluorophenyl)germanium bromide (III) (4.00 g, 6.1 mmoles) in toluene (15 ml) and ether (20 ml) was added at room temperature to a suspension of lithium aluminium hydride (0.26 g, 6.9 mmoles) in toluene (10 ml). The mixture was stirred for 30 min at room temperature and decomposed with water and hydrochloric acid. Extraction with ether, followed by washing, drying ($CaCl_2$) and evaporation of the extract gave a solid which was sublimed (100°/1 mmHg) to give the desired product (2.76 g, 78.5%), m.p. (mixed m.p.) 129–132°.

Reaction of (II) with bromine

Bromine (0.48 g, 3.0 mmoles) and (II) (1.20 g, 2.1 mmoles) in 6 ml of benzene were sealed in an ampoule under vacuum. The mixture was heated at 100° for 6 h. The solvent was removed *in vacuo* and the crystalline solid recrystallized from hexane. The yield of crystalline (III) was 0.96 g (71.0%), m.p. (mixed m.p.) 108°.

Bis[tris(pentafluorophenyl)germyl]mercury (I)

(*a*). A mixture of (II) (2.46 g, 4.3 mmoles) and diethylmercury (0.58 g, 2.2 mmoles) in 5 ml of toluene was heated at 100° for 2.5 h; 62 ml (77%) of ethane was evolved. The precipitate was filtered off and recrystallized twice from toluene to give (I), m.p. 228–231°, yield 1.46 g (51.0%)*.

(*b*). To a previously degassed solution of (II) (1.62 g, 2.8 mmoles) in 4 ml of benzene in an evacuated ampoule, bis(triethylgermyl)mercury (0.72 g, 1.4 mmoles) was added. The mixture was heated for 20 min at 100° after which time the characteristic yellow colour of the germylmercurial had disappeared. The volatiles were recondensed *in vacuo* into a trap cooled with liquid nitrogen. The remaining colourless oil was heated at 140° for 1 h. Further volatiles were removed *in vacuo* and the white crystalline product obtained recrystallized from benzene when 0.75 g (40.2%) of (I) was obtained, m.p. 227–229°. A mixed melting point of (I) prepared by methods (a) and (b) showed no depression. GLC analysis of the volatiles revealed the presence of 0.22 g (1.37 mmoles, 48%) of triethylgermane. In addition, the reaction also afforded 0.08 g (0.4 mmoles, 29%) of metallic mercury.

(*c*) 1.1 g (2.11 mmoles) of $[Me_3Si)_2N]_2Hg$ was added to 2.46 g (4.28 mmoles) of (II) in 5 ml of benzene. A small rise in the temperature of the reaction mixture was observed. In order to complete the reaction, the mixture was heated at 50° for 15 min. Benzene and volatile products were then removed by recondensation *in vacuo* and the resulting crystals washed with hexane when 2.54 g (1.88 mmoles, 89.5%) of (I) was obtained, m.p. 209–224°. After recrystallization from benzene, the substance had m.p. 229–231°. Mixed melting with an authentic sample of (I) gave no depression in the melting point. Chugaev–Tserevitinov analysis on active hydrogen showed the presence of 0.6 g (3.72 mmoles, 88%) of $(Me_3Si)_2NH$ in the volatile products. This product was also identified by GLC analysis.

(*d*). A solution consisting of 4 g (6.13 mmoles) of tris(pentafluorophenyl)germanium bromide and 1.4 g (2.69 mmoles) of bis(triethylgermyl)mercury in 4 ml of benzene was heated at 100° for 20 min. The solution became colourless and a crystalline solid precipitated**. The mixture was left overnight at room temperature. The crystals which were precipitated were filtered off, washed with hexane and dried *in vacuo* when 1.4i g (1.05 mmoles, 75%) of (I) was obtained, m.p. 208–222°. After recrystallization from toluene, the substance had m.p. 228–230°. Mixed melting with an authentic sample of (I) gave no depression in the melting point. GLC analysis of the filtrate gave 1.12 g (4.69 mmoles, 87%) of triethylgermanium bromide.

(*e*). A mixture of 1.4 g (1.11 mmoles) of (V) and 3 g (1.5 mmoles) of mercury in 2 ml of toluene was heated for 30 h at 100° with periodic shaking. The organic products formed were separated from the amalgam of cadmium by decanting the hot toluene solution. Crystals of (I) which precipitated were recrystallized from toluene. The yield of (I) was 0.51 g (0.38 mmoles, 34%) m.p. 227–231°. Mixed melting with an authentic

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* The product yield is very dependent on the reaction conditions and the reagent purity.

** If no crystal formation occurred, the mixture was subjected to UV-irradiation for 2–3 min.

sample of (I) gave no depression in the melting point. Titration of the amalgam gave 0.12 g (1.07) mmoles, 98%) of cadmium.

The IR spectra of the samples of (I) obtained by the five above mentioned methods were found to be identical.

Bis[tris(pentafluorophenyl)germyl]cadmium (V)

A solution of 1.45 g (8.54 mmoles) of diethylcadmium and 10 g (17.4 mmoles) of (II) in 20 ml of toluene was gradually heated for 3 h from 20° to 100° and this temperature maintained for 30 min. During this procedure 370 ml (16.5 mmoles, 97%) of ethane was evolved. The solution was decanted from traces of precipitated cadmium and left overnight at −10°. The crystals of (V) which precipitated were separated by decanting and dried *in vacuo* when 7.7 g (6.1 mmoles, 69%) of (V) was obtained, m.p. 216–218°. Recrystallization from toluene gave a sample of m.p. 218–220°.

Ethyl[tris(pentafluorophenyl)germyl]mercury (IV)

1.19 g (4.61 mmoles) of Et_2Hg was added to an evacuated ampoule together with 2.64 g (4.6 mmoles) of (II) in 3 ml of toluene and linked to a gas burette. The evolution of ethane was observed for ~20 min at room temperature. The mixture was then heated at 100° for 1 h during which time 57 ml of ethane were evolved (2.55 mmoles, 59%). Vacuum distillation of the reaction mixture gave 1.42 g (1.77 mmoles, 38.4%) of $(C_6F_5)_3GeHgEt$ in the form of an extremely viscous liquid which gradually crystallized at room temperature. Two-fold recrystallization from hexane produced a product of m.p. 118–121°.

Photolysis of (I)

A suspension of 1.1 g (0.82 mmoles) of (I) in 3.5 ml of toluene was placed in a Pyrex glass ampoule and subjected to UV irradiation at 50–60° for 60 h. Mercury was precipitated (0.15 g, 0.75 mmoles, 94%) and was separated by decanting from the solution and finely dispersed solid. This solid was filtered off, washed with toluene and dried *in vacuo* when 0.49 g (0.43 mmoles, 52%) of hexakis (pentafluorophenyl)digermane was obtained, m.p. 330–336°. After sublimation (210–240°/1 mmHg) the substance had a melting point of 340–344° under normal pressure with slight sublimation. GLC analysis of the filtrate gave 0.15 g (0.26 mmoles, 32%) of (II) and 0.13 g (0.2 mmoles, 24%) of benzyltris(pentafluorophenyl)germane.

Reaction of (I) with HCl

An ampoule with 1.3 g (0.97 mmoles) of (I), 0.12 g (3.29 mmoles) of HCl and 4 ml of benzene was heated at 100° for 20 h when 0.08 g (0.4 mmoles, 42%) of mercury precipitated and was separted by decanting. On cooling the mixture, crystals of the initial compound (I) were obtained (0.66 g, 0.49 mmoles, 51%). After separating (I), GLC analysis of the mother liquor gave 0.34 g (0.56 mmoles, 61%) of $(C_6F_5)_3GeCl$ and 0.06 g (0.1 mmoles, 10.7%) of (II).

Reaction of (I) with $HgCl_2$

Addition of 1.84 g (1.37 mmoles) of (I) dissolved in 3 ml of tetrahydrofuran (THF) to a suspension of 0.37 g (1.37 mmoles) of $HgCl_2$ in 2 ml of THF afforded 0.1 g (0.5 mmoles) of Hg and a colourless solid. In order to enable the reaction to go to completion, the mixture was heated at 100° for 46 h yielding 0.46 g (2.3 mmoles, 83.5%) of Hg which was separated by decanting. After removal of THF the crystals were recrystallized from hexane when 1.4 g (2.3 mmoles, 84.4%) of $(C_6F_5)_3GeCl$ was obtained, m.p. 106–110°. Literature data[16] gave m.p. 106°. The substance was identified by means of GLC analysis.

Reaction of (I) with Br

0.28 g (1.75 mmoles) of bromine in 2 ml of benzene was added to 1.17 g (0.87 mmoles) of (I) in 3 ml of benzene. After 2–3 min, the mixture lost its colour and solid $HgBr_2$ precipitated (0.24 g, 0.67 mmoles, 77.5%). After separating $HgBr_2$ and removal of benzene *in vacuo*, 1.1 g (1.69 mmoles, 97%) of $(C_6F_5)_3GeBr$ (identified by GLC) was obtained.

(Triethylgermyl)[tris(pentafluorophenyl)germyl]mercury (VIII)

(*a*). A mixture of 0.4 g (0.77 mmoles) of bis(triethylgermyl)mercury, 1.04 g (0.77 mmoles) of (I) and 6 ml benzene was shaken for 15 min at room temperature. The mixture became colourless and (I) dissolved. The solution was filtered using a No. 4 glass filter and benzene removed by condensation *in vacuo* when 1.29 g (1.38 mmoles, 89.5%) of (triethylgermyl)[tris(pentafluorophenyl)germyl]mercury was obtained in the form of a colourless viscous liquid, n_D^{20} 1.5522.

(*b*). A mixture of 1.76 g (3.4 mmoles) of $(Et_3Ge)_2Hg$, 3.46 g (5.3 mmoles) of tris(pentafluorophenyl)germanium bromide and 4 ml of benzene was kept in the dark at room temperature for 3 days. The resulting colourless solution was filtered and benzene and volatile products removed by condensation *in vacuo* when 3.7 g (3.96 mmoles, 76%) of (triethylgermyl)[tris(pentafluorophenyl)germyl]mercury was obtained. A further 0.54 g (2.24 mmoles, 50%) of triethylgermanium bromide was found in the condensate by GLC analysis.

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