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ORGANOMERCURY COMPOUNDS

XIX*. THE PREPARATION OF PENTAFLUOROPHENYLMERCURIALS BY MERCURATION UNDER BASIC CONDITIONS

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Summary

Mercuration of pentafluorobenzene under basic conditions in aqueous tert-butanol by tetrabromomercurate(II) ions, phenylmercuric chloride, and *p*-tolylmercuric chloride yields bis(pentafluorophenyl)mercury, (pentafluorophenyl)-phenylmercury, and (pentafluorophenyl)-*p*-tolylmercury respectively, in a single simple preparative step. Cleavage of bis(pentafluorophenyl)mercury with iodide or bromide ions in alcohols or aqueous alcohols gives pentafluorobenzene, tetrahalogenomercurate(II) ions, and base. Under alkaline conditions, tetrahalogenomercurate(II) ions are reduced to mercury by ethanol or methanol. Decomposition of (pentafluorophenyl)phenylmercury by iodide ions has also been studied.

Introduction

Some acidic hydrocarbons can be mercurated under basic conditions [2], and the method is of particular importance for the synthesis of alkynylmercury [2,3] and trihalogenomethylmercury [4—7] compounds. In general, arenes are not sufficiently acidic to be mercurated in alkaline media [2], but the method is suitable for some polyfluoroarylmercurials [8—10], e.g. bis(2,3,5,6-tetrafluorophenyl)mercury [8, 9] and bis(4-methoxytetrafluorophenyl)mercury [10]. A preliminary account has been given of applications to pentafluorophenylmercurials [8], and we report full details of these simple and convenient preparations. Cleavage of the mercurials with halide ions, essentially reversal of the syntheses, is also described. (For a preliminary report, see ref. 11.)

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*For part XVIII see ref. 1.

Results and discussion

(a). Preparations of pentafluorophenylmercurials

Bis(pentafluorophenyl)mercury has been prepared by reaction of mercuric chloride, an excess of lithium bromide, pentafluorobenzene, and sodium hydroxide.

$$2\,C_6F_5H + HgBr_4^{2-} + 2\,OH^- \rightarrow (C_6F_5)_2Hg + 4\,Br^- + 2\,H_2O \qquad (1)$$

Aqueous tert-butanol was the most satisfactory solvent, but low yields of mercurial were still obtained with water alone (see Experimental). Ethanol or methanol cannot be used, as they are oxidised by alkaline tetrabromomercurate(II) ions (see below). Maximum yields were obtained using either an excess of hydroxide ions or of pentafluorobenzene, the former being preferable in view of the cost of the polyfluoroarene*, and the minimum excess of lithium bromide (mol. ratio $LiBr/HgCl_2 \approx 15/1$, giving [12] predominantly $HgBr_4^{2-}$) necessary to prevent significant precipitation of mercuric oxide. Although both pentafluorobenzene [13] and bis(pentafluorophenyl)mercury [14] undergo nucleophilic substitution with hydroxide ions, the high yield of mercurial obtained when using an excess of base shows that fluoride elimination is unimportant under the present conditions.

The simplicity and convenience of the single preparative step, coupled with the good yield and easy purification, should make mercuration under basic conditions the preferred route to bis(pentafluorophenyl)mercury. Of the many reported syntheses [15] (for preparations subsequent to the review [15], see refs. 16—18), the Grignard method [19], the reaction of pentafluorophenylhydrazine with mercury and mercuric oxide (requiring prior preparation of finely divided mercury [20]), preparation and symmetrization of pentafluorophenylmercuric trifluoroacetate [21], and reaction of pentafluoroiodobenzene with mercury [22], appear the most suitable for medium-to-large scale quantities. The first three have the disadvantage of two preparative steps [19—21], whilst the last has a more difficult procedure (sealed tube at elevated temperatures) and a longer reaction time [22] than the present method, which has the further advantage of using the cheapest source (cost/mol basis) of the pentafluorophenyl group.

(Pentafluorophenyl)phenylmercury and (pentafluorophenyl)-*p*-tolymercury have been obtained by reaction of the corresponding arylmercuric chlorides with pentafluorobenzene and sodium hydroxide in aqueous tert-butanol.

$$RHgCl + C_6F_5H + OH^- \rightarrow RHgC_6F_5 + H_2O + Cl^- \; (R = Ph \text{ or } p\text{-}MeC_6H_4) \qquad (2)$$

In previous preparations, Grignard (R = Rh) [19] or decarboxylation [23] (R = Ph or *p*-MeC_6H_4) methods were used. (Pentafluorophenyl)phenylmercury required more purification (several recrystallizations) than reported [19] for the compound from the Grignard synthesis (vacuum sublimation), but we have found that both the Grignard and the decarboxylation [23] products require several recrystallizations. In any case, the simple procedure and short reaction time for

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*Currently £14/100 g from Bristol Organics.

TABLE 1

YIELDS (%) OF PRODUCTS FROM CLEAVAGE OF BIS(PENTAFLUOROPHENYL)MERCURY[a]

Reaction time (min)	HgI_4^{2-}	Base	HgI_4^{2-}	Base	Base	Base
	(in EtOH)		(in EtOH/H_2O 3/2)		(in MeOH)	(in t-BuOH/H_2O 3/2)
1	32	29	35	35 (98)[b]	23	19 (97)[b]
5	72	69	80	80		52
10	52[c]	49[c]	88[d]	86[d]	92	72[e]
15			84	85		83
30	0	2	55	52	82	92
60			42	35		
120					61	99[f]

[a] Yields calculated on the basis of reaction (3). [b] % acid consumed, when HCl, mol. ratio $(C_6F_5)_2Hg/HCl = 1/2$, added. Corresponding yields of pentafluorobenzene; [c] 100%, [d] 100%, [e] 77%. [f] Yield of HgI_4^{2-} 96%.

mercuration under basic conditions should make it the preferred route to these aryl(pentafluorophenyl)mercury compounds.

(b). Cleavage of bis(pentafluorophenyl)mercury with halide ions

Reaction of bis(pentafluorophenyl)mercury with an excess of iodide ions in alcohols or aqueous alcohols gives tetraiodomercurate(II) ions, pentafluorobenzene, and base [reaction (3); X = I]*.

$$(C_6F_5)_2Hg + 4\,X^- + 2\,H_2O\ (\text{or } 2\,ROH) \rightarrow HgX_4^{2-} + 2\,C_6F_5H + 2\,OH^-\ (\text{or } 2\,OR^-) \tag{3}$$

With increasing reaction time, yields of tetraiodomercurate(II) ions and base (Table 1) from cleavage in ethanol or aqueous ethanol increase to a maximum ($<$ 100%, even though corresponding yields of C_6F_5H show that cleavage is quantitative), and then decrease owing to further reaction giving mercury metal and acetaldehyde [reaction (4); X = I].

$$HgX_4^{2-} + 2\,OH^- + CH_3CH_2OH \rightarrow Hg + 4\,X^- + 2\,H_2O + CH_3CHO \tag{4}$$

Reduction of tetraiodomercurate(II) ions to mercury in alkaline aqueous ethanol has been independently demonstrated, the number of hydroxide ions consumed per tetraiodomercurate(II) ion being 2.2, consistent with occurrence of reaction (4) and some contribution from reaction (5).

$$HgI_4^{2-} + 3\,OH^- + CH_3CHO \rightarrow Hg + 4\,I^- + 2\,H_2O + CH_3CO_2^- \tag{5}$$

Reduction of mercuric oxide by ethanol at 150° has been reported [25], but does not occur under the present conditions, hence a halogenomercury(II) species is essential for reduction to occur. The yields of base from cleavage in methanol (Table 1) show a pattern similar to those in ethanol, hence solvent oxidation again occurs, though the reaction is slower than for ethanol. By contrast, cleavage proceeds straightforwardly in aqueous tert-butanol giving near quantitative yields of base and HgI_4^{2-}. Decomposition by iodide ions is accelerated

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*Subsequent to our preliminary communication [11], cleavage of the mercurial by iodide ions in *N,N*-dimethylformamide has been briefly reported [24].

markedly on addition of hydrochloric acid (Table 1). The mercurial is stable to acid cleavage in the absence of added halide ions (see Experimental, and ref. 19).

Cleavage with bromide ions [reaction (3); X = Br] is very incomplete in aqueous tert-butanol, consistent with the preparation of bis(pentafluorophenyl)-mercury in good yield by the reverse reaction (1). In aqueous ethanol, tetrabromomercurate(II) and hydroxide ions obtained from cleavage are consumed by reaction (4), which is faster for X = Br than X = I. With added acid, cleavage is virtually quantitative in both solvents.

Removal of highly electronegative groups from mercury by halide ions appears to be a fairly general reaction, and has also been observed for perfluoroalkylmercurials [24, 26, 27], phenyl(trihalogenomethyl)mercurials [27, 28], some mercurated phenols [29], and α-mercurated sulphones [30], aldehydes [31], carboxylic acid esters [24, 31], and ketones [31].

(c). *Cleavage of (pentafluorophenyl)phenylmercury with iodide ions*

(Pentafluorophenyl)phenylmercury is rapidly cleaved by an equivalent amount of sodium iodide in aqueous acetone.

$$PhHgC_6F_5 + I^- + H_2O \rightarrow PhHgI + OH^- + C_6F_5H \qquad (6)$$

However, decomposition with a large excess of iodide ions (mol. ratio, NaI/-$PhHgC_6F_5$ = 67/1) in aqueous ethanol gives tetraiodomercurate(II) ions and diphenylmercury as major products, due to disproportionation (7) followed by the cleavage reaction (3) (X = I).

$$2\,PhHgC_6F_5 \rightarrow Ph_2Hg + (C_6F_5)_2Hg \qquad (7)$$

The alternative route, reaction (6) followed by (8), can be ruled out since phenylmercuric iodide is not symmetrized by iodide ions [32].

$$2\,PhHgI + 2\,I^- \rightarrow HgI_4^{2-} + Ph_2Hg \qquad (8)$$

No disproportionation of (pentafluorophenyl)phenylmercury in refluxing aqueous acetone or aqueous ethanol was observed with reaction times similar to those for cleavage, but catalysis of reaction (7) by halide ions is likely [see e.g. halide induced disproportionation of (pentachlorophenyl)(pentafluorophenyl)mercury [33]), and would be far more pronounced with the large excess of iodide used in aqueous ethanol.

(d). *Possible mechanisms for mercuration and cleavage*

Since mercuration under basic conditions [reactions (1) and (2)] is the reverse of cleavage by halide ions [reactions (3) and (6) respectively], the mechanisms must be closely related. In basic alcoholic media, pentafluorobenzene yields pentafluorophenyl carbanions [34]*, which can react with a halogenomercury(II) species in the mercuration step. This is represented in Scheme 1 for $(C_6F_5)_2Hg$.

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[*] Deprotonation is far faster than nucleophilic displacement of fluoride [34], perhaps explaining the the absence of significant competition from fluoride elimination in the present syntheses [section (*a*)].

SCHEME 1

$$C_6F_5H + OH^- \rightleftharpoons C_6F_5^- + H_2O$$

$$HgX_4^{2-} \rightleftharpoons X^- + HgX_3^- \rightleftharpoons 2\ X^- + HgX_2 \quad (X = Br \text{ for mercuration})$$

$$C_6F_5^- + HgX_2 \rightleftharpoons C_6F_5HgX_2^- \rightleftharpoons C_6F_5HgX + X^-$$

$$C_6F_5^- + C_6F_5HgX \rightleftharpoons (C_6F_5)_2HgX^- \rightleftharpoons (C_6F_5)_2Hg + X^-$$

Perfluoroalkylmercurials have been prepared from mercuric salts and perfluoroalkyl carbanions (derived from perfluoro-olefins and fluoride ions)[35]. In cleavage (X = Br or I), the steps are reversed, and a similar mechanism has already been proposed for decomposition of some mercurials in *N*,*N*-dimethylformamide [24]. Bis(pentafluorophenyl)mercury does not give isolable $(C_6F_5)_2HgX^-$ complexes [36], and disproportionation [37] complicates attempts to obtain $C_6F_5HgX_2^-$. However, some analogous halogeno(perfluoroalkyl)mercurate(II) complexes have been detected [26, 38], and halogeno-organomercurate(II) species are well-established kinetic intermediates [39]. Cleavage by direct hydrolysis of $(C_6F_5)_{3-n}HgX_n^-$ (n = 1 or 2) complexes, analogous to proposals for (pentafluorophenyl)tin compounds [40], may be ruled out, since such a process is probably irreversible and since cleavage has also been effected in anhydrous tert-butanol. However, acceleration of cleavage on acidification [section (*b*)] may be due to direct protolysis of $Hg(C_6F_5)_{3-n}X_n^-$. Mercuration syntheses and iodide cleavage of aryl(pentafluorophenyl)mercurials probably proceed via reactions (9) (R = Ph or *p*-MeC_6H_4; X = Cl) and (10) (R = Ph; X = I), in view of mechanisms for analogous reactions involving phenyl(trihalogenomethyl)mercurials [5, 28]:

$$C_6F_5^- + RHgX \underset{(10)}{\overset{(9)}{\rightleftharpoons}} C_6F_5HgR + X^-$$

Experimental

Microanalyses were by the Australian Microanalytical Service, Melbourne. Infrared spectra were recorded on Nujol and hexachlorobutadiene mulls with Perkin—Elmer 257 and Unicam SP 200 spectrophotometers. Ultraviolet spectra were obtained with Unicam SP 700 and Shimadzu QV-50 spectrophotometers. Gas—liquid chromatography was carried out with an Aerograph Autoprep A700 instrument fitted with a silicone gum SE30 column.

Pentafluorobenzene was obtained from Imperial Smelting Corporation and Bristol Organics. Mercuric chloride (A.R.) was from B.D.H.. Phenylmercuric chloride (B.D.H.) was recrystallized from benzene and *p*-tolylmercuric chloride was prepared as reported [41]. Other reagents and solvents were laboratory reagent grade and were used without purification unless indicated otherwise. Lithium bromide hydrate contained ca. 15% water by weight. Aqueous tert-butanol, aqueous ethanol, and aqueous acetone contained 60, 60, and 75% respectively, of the appropriate organic solvent by volume.

Synthesis of bis(pentafluorophenyl)mercury by mercuration under basic conditions

Optimum conditions, (*a*) and (*b*), were established on the basis of a range of small scale preparations (using 2.0 mmole of mercuric chloride).

(a). Using an excess of sodium hydroxide. A soln. of lithium bromide hydrate (30g, ca. 290 mmole), mercuric chloride (5.4 g, 20 mmole), sodium hydroxide (4.9 g, 122 mmole), and pentafluorobenzene (6.8 g, 40 mmole) in 200 ml aqueous tert-butanol was stirred vigorously and heated under reflux for 4 h. (The mercuric and lithium salts were dissolved before addition of sodium hydroide.) The reaction mixture was filtered, and the filtrate was poured into a large volume of water precipitating bis(pentafluorophenyl)mercury, which was recrystallized from aqueous methanol (yield 7.9 g, 75% based on $HgCl_2$), m.p. 141 - 142°, lit. [19] m.p. 142°, mixed m.p. (with a sample obtained by the Grignard method [19]), 141 - 142°. The infrared spectrum of the product was identical with that of the authentic compound obtained by the Grignard method [19]. The yield was not increased by a longer reaction time or a larger excess of lithium bromide.

(b). Using an excess of pentafluorobenzene. A soln. of lithium bromide hydrate (22.5 g, ca. 220 mmole), mercuric chloride (4.9 g, 18 mmole), sodium hydroxide (1.6 g, 40 mmole), and pentafluorobenzene (9.0 g, 54 mmole), in 200 ml aqueous tert-butanol was stirred vigorously and heated under reflux for 4 h, further pentafluorobenzene (4.5 g, 27 mmole) being added after 2 h. Treatment and purification as in (*a*) gave the mercurial (69%), identified as above. The preparation can also be effected using mercuric oxide in place of mercuric chloride and sodium hydroxide.

(c). Effect of omission of tert-butanol. A mixture of pentafluorobenzene (4.0 mmole) and an aqueous solution (20 ml) of mercuric chloride (2.0 mmole), sodium hydroxide (4.75 mmole), and lithium bromide hydrate (ca. 25 mmole) was heated under reflux for 4 h giving bis(pentafluorophenyl)mercury (17%).

Synthesis of (pentafluorophenyl)phenylmercury by mercuration under basic conditions

A suspension of phenylmercuric chloride (6.3 g, 20 mmole) in aqueous tert-butanol (100 ml) containing sodium hydroxide (0.97 g, 24 mmole) and pentafluorobenzene (4.4 g, 26 mmole), was stirred and heated under reflux for 45 min, the arylmercuric halide dissolving after 5 min. The reaction mixture was cooled and poured into a large volume of water precipitating (pentafluorophenyl)phenylmercury, which was recrystallized several times from carbon tetrachloride (yield 3.0 g, 33% based on PhHgCl), m.p. 161 - 162°, lit. [19] m.p. 164°, mixed m.p. (with a sample m.p. 161 - 162° obtained by the Grignard method [19]), 161 - 162°. The infrared spectrum of the product was identical with that of the authentic compound obtained by the Grignard method [19].

Synthesis of (pentafluorophenyl)-p-tolylmercury by mercuration under basic conditions

Similarly, *p*-tolylmercuric chloride (0.66 g, 2.0 mmole), sodium hydroxide (0.50 g, 12.5 mmole), and pentafluorobenzene (0.34 g, 2.0 mmole) in 20 ml of aqueous tert-butanol gave (reaction time, 15 min) (pentafluorophenyl)-*p*-tolylmercury (0.55 g, 60%, from hexane), m.p. 171 - 172°, lit. [23] m.p. 170 - 172°. (Found: C, 34.6; H, 1.9; F, 20.3. $C_{13}H_7F_5Hg$ calcd.: C, 34.0; H, 1.5; F, 20.7%.) The infrared spectrum was identical with that of an authentic sample obtained by decarboxylation [23].

Cleavage of bis(pentafluorophenyl)mercury with halide ions

Yields and products were generally unaltered when the reactions were carried out under nitrogen. A nitrogen atmosphere was used to prevent water absorption during cleavage in anhydrous t-butanol.

(a). Reactions with iodide ions. For reactions in Table 1, a soln. of bis(pentafluorophenyl)mercury (0.50 mmole) and sodium iodide (33 mmole) in 20 ml of the appropriate solvent was heated under reflux. [When acidic conditions were required (Table 1), HCl (1.0 mmole) was added.] The reaction mixture was then cooled in ice and the alkali formed (or unreacted acid) was determined volumetrically. After filtration to remove mercury (reactions in ethanol or methanol) or unreacted mercurial, the yield of tetraiodomercurate(II) ions was determined by precipitation as bis(methyltriphenylarsonium) tetraiodomercurate(II) [42]. Identities of products from representative reactions were confirmed by melting points [42] and infrared spectra [43]. The precipitation procedure was found to be near quantitative ($\geqslant$ 96%) in the solvents used. Yields of pentafluorobenzene were determined by ultraviolet spectroscopy and gas—liquid chromatography, the compound being distilled from the reaction mixtures during the final 5 min of reaction time. No other volatile fluorocarbons were detected. Cleavage of the mercurial in ethanol for 30 min yielded acetaldehyde, which was swept from the reaction mixture with nitrogen and was isolated as the 2,4-dinitrophenylhydrazone [44] (identified by thin-layer chromatography). Reaction of the mercurial (0.50 mmole) with dried (120°, then silica gel) sodium iodide (2.0 mmole) in 20 ml of refluxing anhydrous tert-butanol (twice distilled from calcium hydride) for 30 min gave base (27%) and HgI_4^{2-} (29%). The corresponding reaction in aqueous tert-butanol gave base (62%).

Bis(pentafluorophenyl)mercury was recovered unchanged after being heated under reflux in aqueous ethanol, with or without added acid, using conditions similar to those of the cleavage reactions. When pentafluorobenzene (1.0 mmole), sodium hydroxide (1.0 mmole), and sodium iodide (33 mmole) in 20 ml of aqueous tert-butanol were heated under reflux for 3½ h, base (9%) was consumed.

(b). With bromide ions. Cleavage reactions were carried out in a similar manner to those with iodide ions. In aqueous ethanol, mercury was deposited, and yields of base were 3—12% for reaction times of 10 min to 4 h. The yield of pentafluorobenzene was 53% after 1 h. With added HCl, 96% of the acid was consumed in 30 min. In aqueous tert-butanol, no mercury was deposited, and base (24%) and tetrabromomercurate(II) ions [26%, determined gravimetrically as $(MePh_3As)_2HgBr_4$ [37]; 85—90% of $HgBr_4^{2-}$ is precipitated] were obtained after 2 h. The yield of pentafluorobenzene was 35% after 1 h. There was little change in the yield of base from 30 min to 6 h. With added acid, 87% was consumed in 15 min, and 96% in 30 min.

Cleavage of (pentafluorophenyl)phenylmercury with iodide ions

(a). In aqueous acetone. (Pentafluorophenyl)phenylmercury (0.50 mmole) and sodium iodide (0.50 mmole) in 20 ml aqueous acetone were heated under reflux for 10 min yielding base (77%). Addition of water precipitated phenylmercuric iodide (80%, after being washed with carbon tetrachloride to remove $PhHgC_6F_5$), m.p. 270°, lit. [45] m.p. 264 - 266°, the infrared spectrum being identical with that of an authentic sample prepared from phenylmercuric acetate

[46]. Under similar conditions, bis(pentafluorophenyl)mercury (0.25 mmole) and sodium iodide (0.50 mmole) gave base (54%).

(b). In aqueous ethanol. (Pentafluorophenyl)phenylmercury (0.50 mmole) and sodium iodide (33 mmole) in refluxing aqueous ethanol (20 ml) for 10 min gave base (90%) and tetraiodomercurate(II) ions (68%), determined after removal of the white precipitate obtained during titration of base. Extraction of the precipitate with boiling hexane gave a residue of phenylmercuric iodide (ca. 20%; infrared identification), m.p. 267 - 268° (after vacuum sublimation), and evaporation of the solvent to crystallization gave diphenylmercury (50%), m.p. 124°, lit. [47] m.p. 125° (infrared identification [48]).

Reaction of tetrahalogenomercurate(II) ions with alkaline aqueous ethanol

(a). Tetraiodomercurate(II) ions. Mercuric iodide (0.50 mmole), sodium iodide (33 mmole) and sodium hydroxide (1.0 mmole) in 20 ml aqueous ethanol were heated under reflux giving mercury metal. After 1 h, base (34%) and mercuric iodide (42%, as HgI_4^{2-}) remained, and after 4 h, 7% and 17% respectively, remained. The amounts consumed were not significantly affected when a nitrogen atmosphere was used, and no reaction occurred on omission of ethanol or base. In the absence of sodium iodide, base was consumed and mercury and mercurous salts were obtained. No reaction occurred between mercuric oxide (commercial, freshly precipitated, or precipitated "in situ") and refluxing aqueous ethanol in the absence of halide ions. Qualitative tests showed that alkaline tetraiodomercurate(II) ions oxidise methanol.

(b). Tetrabromomercurate(II) ions. On reaction of mercuric bromide (0.50 mmole), lithium bromide (33 mmole), and sodium hydroxide (1.0 mmole) in 20 ml refluxing aqueous ethanol, 93% of the base was consumed in 30 min.

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