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HALOMETHYL-METAL COMPOUNDS

LIX*. AN IMPROVED PREPARATION OF PHENYL(FLUORODICHLOROMETHYL)MERCURY, A USEFUL FLUOROCHLOROCARBENE PRECURSOR

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SUMMARY

An improved procedure for the preparation of phenyl(fluorodichloromethyl)mercury involving the reaction of phenylmercuric chloride with sodium methoxide and fluorodichloromethane in THF/methanol at −40° is described. This procedure gives $PhHgCCl_2F$ in high yield and generally of about 85% purity. Reactions of the $PhHgCCl_2F$ thus prepared with olefins [cyclohexene, cyclooctene, allyltrimethylsilane, vinyltriethylsilane, $Me_2C{=}CHCl$, $Me_2C{=}C(Br)Me$, $CH_2{=}C(Me)CH_2Cl$ and norbornene] gave the expected cyclopropanes in high yield. This mercury reagent added CClF to the C=N bond of $PhN{=}CCl_2$, giving 1-phenyl-2-fluoro-2,3,3-trichloroaziridine and to the C=S bond of thiobenzophenone. The thiirane formed was, however, unstable to the reaction and work-up conditions and only its desulfurization product, $Ph_2C{=}CClF$, could be isolated. Fluorochlorocarbene transfer from this mercurial also could be induced by the action of sodium iodide in DME. Evidence is presented which shows that $PhHgCCl_2F$ is more reactive in CClF transfer than indicated in a previous report.

INTRODUCTION

In a previous paper of this series,[2] it was reported that phenyl(fluorodichloromethyl)mercury could be prepared by reaction of phenylmercuric chloride, fluorodichloromethane and potassium tert-butoxide in diethyl ether at −25°. The yields of this mercurial were low (≈35%) and the product obtained usually was contaminated with substantial (≈20%) amounts of diphenylmercury. The fluorination of phenyl(bromodichloromethyl)mercury with $PhHgF \cdot HF$ provided $PhHgCCl_2F$ in good yield and purity[3,4], but this procedure required the prior synthesis of $PhHgCCl_2Br$ and $PhHgF \cdot HF$ and was time-consuming as a result. Phenyl(fluorodichloromethyl)mercury reacts with olefins at 80° to give *gem*-fluorochlorocyclopropanes in

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[*] For Part LVIII see ref. 1 (Part LVIII was inadvertently labelled LVII).

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high yield, but in order for this reagent to receive recognition as a useful CClF transfer agent, its synthesis required substantial improvement. We report here an improved synthesis of this mercurial. In addition, we note that it is more reactive as a CClF reagent than had previously been supposed.

RESULTS AND DISCUSSION

The change in the basic procedure which resulted in substantial improvement of the preparation of $PhHgCCl_2F$ was a simple one. Replacement of potassium tert-butoxide with sodium methoxide was the deciding factor. The addition of sodium methoxide in methanol/THF to a mixture of phenylmercuric chloride and fluorodichloromethane in THF at −40° to −35° gave $PhHgCCl_2F$ in high yield. As in the potassium tert-butoxide based procedure, the product was contaminated with diphenylmercury (up to 15 %) which could not be separated easily by fractional crystallization or column chromatography. However, this impurity was an innocuous one which did not interfere with the CClF transfer chemistry of $PhHgCCl_2F$. Although the amount of diphenylmercury contaminant can be assessed reasonably accurately by analytical brominolysis[2], we used a simpler procedure to obtain an indication of the purity of each batch of phenyl(fluorodichloromethyl)mercury prepared. After each preparation, a sample of the product was used in a reaction with cyclooctene in benzene at 80° to produce 9-fluoro-9-chlorobicyclo[6.1.0]nonane, a reaction which we know $PhHgCCl_2F$ undergoes in nearly quantitative yield[2] [eqn. (1)]. From the yield of the organic product obtained, and from the amount of phenylmercuric chloride isolated, we obtained a reasonable estimate of the purity of that particular batch of $PhHgCCl_2F$, an estimate which certainly was sufficient for the purpose of conducting further reactions with the batch of mercurial in question.

(1)

During the course of this study we discovered that phenyl(fluorodichloromethyl)mercury is a more reactive source of CClF than our previous study[2] had indicated. In our earlier work with $PhHgCCl_2F$, we had tried to follow the course of its reactions with olefins and other substrates by means of thin layer chromatographic analysis for the $PhHgCCl_2F$ as yet unconsumed, and had concluded that reaction times of about 48 h at 80° are required for complete consumption of the mercurial. Present research has shown such TLC analysis to be unreliable due to the presence of the diphenylmercury.

In order to define the optimum reaction time for olefins of average carbenophilic reactivity, we carried out reactions of $PhHgCCl_2F$ (all samples from the same batch) with cyclohexene in benzene under standard conditions for variable lengths of time. The results, summarized in Table 1, show that reaction times of 10–12 h are sufficient. In other words, $PhHgCCl_2F$ is about four times more reactive then we had originally supposed.

This greater reactivity of $PhHgCCl_2F$ is not surprising in the light of our recent work with $PhHgCBr_2F$[5]. This mercurial was found to be considerably more reactive than $PhHgCBr_3$. For instance, at room temperature its reaction with cyclooctene

TABLE 1

REACTION OF PHENYL(FLUORODICHLOROMETHYL)MERCURY WITH CYCLOHEXENE: EFFECT OF REACTION TIME ON PRODUCT YIELDS[a]

Reaction time (h)	*7-Fluoro-7-chloronorcarane yield (%)*	*PhHgCl, yield (%)*
1	25	32
3	52	59
5	64	75
7	74	80
9	77	83
12	79	85
20	82	87

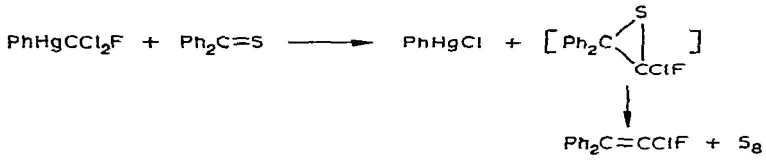
[a] Each entry represents a separate reaction. The mercurial [between 6.63 and 7.07 g (17.4 and 18.6 mmol) assuming pure $PhHgCCl_2F$] and 52–62 mmol of cyclohexene in 35 ml of dry benzene were stirred and heated at 82–83° under nitrogen.

was complete within 3 days. In contrast, the room temperature reaction between $PhHgCBr_3$ and this olefin required 15–16 days[6]. Our finding that $PhHgCCl_2F$ is more reactive than $PhHgCCl_3$ is in line with this observation. The factors responsible for this enhanced reactivity when a fluorocarbene is extruded from a phenyl(trihalomethyl)mercurial will be discussed at length in our paper concerning $PhHgCBr_2F$[7].

The phenyl(fluorodichloromethyl)mercury prepared during the course of this study was used to prepare various *gem*-fluorochlorocyclopropanes including five that are new. The preparative work is summarized in Table 2. An arbitrary reaction time of 20 h was used in most cases, although shorter reaction times probably would have sufficed. The reported product yields are high and in actual fact are even higher since they were calculated by assuming that the mercurial charged (which was the limiting reagent) was pure $PhHgCCl_2F$.

The first example of CClF addition to a C=N bond also was demonstrated [eqn. (2)]. In contrast, the product of the addition of CClF to the C=S bond of thiobenzophenone was not stable at 80° [eqn. (3)]. In earlier studies we had shown that $PhHgCCl_2Br$ derived dichlorocarbene adds to $PhN{=}CCl_2$ to give 1-phenyl-2,2,3,3-tetrachloroaziridine[8] and to thiobenzophenone to produce 2,2-dichloro-3,3-diphenylthiirane[9]. The latter differed from 2-fluoro-2-chloro-3,3-diphenylthiirane in stability; it was not decomposed at 80° and could be isolated as the pure substance. Thermolysis at 200° gave 1,1-dichloro-2,2-diphenylethylene[9].

(2)



(3)

The sodium iodide procedure [eqn. (4)][2,10] served well in the generation of

$$\begin{array}{lr} PhHgCX_3 + Na^+I^- \xrightarrow[80-85^\circ]{MeOCH_2CH_2OMe} PhHgI + \overset{\displaystyle Na^+CX_3^-}{\underset{\displaystyle NaX + CX_2}{\downarrow}} & (4) \end{array}$$

CClF from the $PhHgCCl_2F$ prepared in this study, and it was found to be applicable to CClF transfer to $PhN{=}CCl_2$ and $Ph_2C{=}S$ as well. The products obtained were the same as those produced in the thermal reaction and their yields were comparable. The advantage of the sodium iodide procedure resides in the fact that these high yields can be effected in shorter reaction times (3 h) than in the thermal reaction which effects similar CClF transfer (10–12 h).

The present study has served to change the image of $PhHgCCl_2F$ from that of a difficult-to-prepare and not very reactive reagent to that of a readily accessible, quite reactive CClF transfer agent. The previously demonstrated[2] applicability of phenyl(fluorodichloromethyl)mercury to the synthesis of *gem*-fluorochlorocyclopropanes from base-sensitive or poorly reactive olefins thus becomes of greater significance in preparative chemistry.

EXPERIMENTAL

General comments

All reactions were carried out in flame-dried glassware under an atmosphere of prepurified nitrogen. Infrared spectra were recorded using a Perkin–Elmer Model 457A grating infrared spectrophotometer, proton NMR spectra using a Varian Associates T60 spectrometer. Chemical shifts are reported in δ units, ppm downfield from internal TMS. Fluorine NMR spectra were obtained using a Perkin–Elmer–Hitachi R20B spectrometer at 56.446 MHz. Gas chromatography (GLC) was used extensively in the analyses of reaction mixtures, determination of product yields and isolation of products. The units employed were Hewlett–Packard F&M model 700 and 5754 and MIT isothermal gas chromatographs. Two columns served in this study: (*1*) 4 ft. 10% UCW 98 silicone rubber on Chromosorb W and (*2*) 7 ft. 25% SE 30 silicone rubber gum on Chromosorb W.

Preparation of phenyl(fluorodichloromethyl)mercury

A flame-dried 2 liter, three-necked flask equipped with a paddle stirrer, a pressure equalizing addition funnel with a nitrogen inlet tube and a low temperature thermometer was charged with 85.2 g (0.272 mol) of phenylmercuric chloride, 130 g (1.26 mol) of fluorodichloromethane (Matheson) and 1200 ml of dry tetrahydrofuran (THF). The addition funnel was charged with a solution of sodium methoxide prepared by dissolving 7.54 g (0.36 g-atom) of sodium in 85 ml of absolute methanol and diluting with 100 ml of THF. The flask contents were cooled to −40° and the NaOMe solution was added slowly over a period of 1 h. The reaction temperature was not allowed to rise above −35° during the addition. After the addition had been completed, the temperature was allowed to rise to 0°. The reaction mixture then was transferred to a 3 liter round-bottom flask and the solvent was removed at reduced pressure. The viscous gray residue was dissolved in 1400 ml of benzene and extracted with 50 ml of water. (It is important to keep the amount of water used to a minimum). In many instances at this point, a two-phase system containing a small amount of solid resulted.

When this is so, further work-up involving filtration through a paper towel or a layer of Celite is satisfactory. However, in other reactions carried out in this manner treatment of the nonvolatiles with benzene and water as described above gave a gray emulsion which was virtually unfilterable, rather than an easily handled two-phase system. We found that centrifugation (International centrifuge, using 1 quart centrifuge bottles) was the best means of resolving the difficulty. Centrifugation gave an easily separable two-layer system.

Whichever procedure was used, further work-up involved separation and drying of the organic layer over anhydrous $MgSO_4$ and evaporation of the solvent under reduced pressure. In this particular reaction, the yield of crude $PhHgCCl_2F$ obtained was 90.4 g (88 %, calculated on the basis of pure material). In the many reactions carried out, these product yields usually ranged between 85 and 90 %.

The material thus obtained can be used without further purification for CClF transfer chemistry. It is "pure" by thin layer chromatography but it does contain variable amounts of diphenylmercury. An estimate of the purity, based on elemental analysis and on the yield of PhHgCl obtained when the material is allowed to react with cyclooctene at 80°, indicates that the material is about 85–92 % $PhHgCCl_2F$. The purity of each batch was assessed by carrying out a reaction of a sample with cyclooctene in benzene at 80°. A typical analysis for such crude $PhHgCCl_2F$ was: C, 25.47; H, 1.75 %.

^{19}F NMR (in $HCCl_3$): singlet, 114.7 ppm downfield from internal hexafluorobenzene, mercury satellites with $J(^{199}Hg–^{19}F)$ 660 Hz. IR (in CCl_4); 3095 m, 3074 s, 3040 s, 2930 m, 1955 m, 1883 w, 1868 w, 1812 m, 1750 w, 1576 (sh), 1545 m, 1478 s, 1430 s, 1254 (sh), 1244 m, 1064 (sh), 1022 s, 1000 s, 907 w, 721 m, 698 s, 677 s cm^{-1}.

The m.p. of the product of the reaction described above was 98–100°. The m.p. of analytically pure $PhHgCCl_2F$ is 98–100° [2], so we feel than the melting points of the product are not a reliable guide to purity.

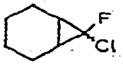
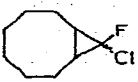
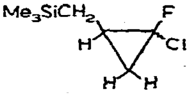
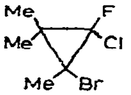
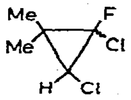
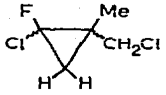
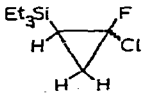
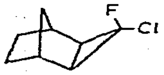
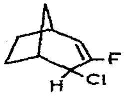
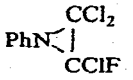
Thermolysis of phenyl(fluorodichloromethyl)mercury in the presence of carbenophiles

A three-necked flask of appropriate size equipped with a magnetic stirring assembly, a thermometer and a reflux condenser topped with a nitrogen inlet tube was flame-dried under nitrogen and allowed to cool to room temperature under a positive pressure of nitrogen. The flask was charged with one molar equivalent of the crude mercury reagent, three molar equivalents of the carbenophile and dry benzene (about 2 ml per mmol of mercury reagent). The reaction mixture was stirred and heated at reflux under nitrogen for an appropriate length of time (usually 20 h), cooled and filtered to remove the precipitated PhHgCl. The filtrate was trap-to-trap distilled at reduced pressure into a cold (−78° to −196°) receiver and the distillate analyzed by GLC. Product yields were determined by GLC using the internal standard procedure. The products were isolated by GLC or by fractional distillation. Known compounds were identified by comparison of their GLC retention times, IR spectra, NMR spectra and/or refractive indices with those of authentic samples or with literature values. Table 2 indicates mmols of reactants used, reaction times, products formed and their yields and phenylmercuric chloride yields. Further comments concerning product identification follow below.

As in our previous study[2], we noted the formation of both *syn* and *anti* isomers when such were possible, but in no case was resolution of the isomer mixture attempted.

TABLE 2

REACTIONS OF PHENYL(FLUORODICHLOROMETHYL)MERCURY[a]

Reactant	*(mmol)*	*$PhHgCCl_2F$ (mmol)*	*Product*	*(Yield %)*[b]	*PhHgCl yield (%)*
	(62)	18.6		(84)	86
	(650)	216.0		(78)	85
$Me_3SiCH_2CH{=}CH_2$	(32)	14.0		(91)	90
$Me_2C{=}C(Br)CH_3$	(60)	18.2		(80)	92
$Me_2C{=}CHCl$	(74)	17.3		(72)	85
$CH_2{=}C(CH_3)CH_2Cl$	(75)	16.1		(80)	86
$Et_3SiCH{=}CH_2$	(24.4)	15.1		(65).	81
	(65)	20.3		(42)	88
			+		
				(42)	
Et_3SiH	(50)	18.3	$Et_3SiCHClF$	(80)	84
$PhN{=}CCl_2$	(39)[c]	21.2	 $PhN\langle{}^{CCl_2}_{CClF}$	(74)	87
$Ph_2C{=}S$	(15.7)[d]	14.8	$Ph_2C{=}CClF$	(75)	85

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[a] Reactions carried out in benzene solution at reflux, with stirring under nitrogen, for 20 h unless otherwise specified. [b] Yields were calculated assuming that the mercurial charged was pure $PhHgCCl_2F$; because of the inert Ph_2Hg contaminant in the mercurial, these yields are somewhat lower than the actual yields based on $PhHgCCl_2F$ in the mercury starting material. [c] 40 h reaction time. [d] 48 h reaction time.

Their peaks in the gas chromatograms always overlapped. The products derived from cyclohexene, cyclooctene and triethylsilane are known compounds and samples were available from our previous study[2]. The $Et_3SiCHClF$ isolated had n_D^{25} 1.4344 (lit.[2] n_D^{25} 1.4351). Its ^{19}F NMR spectrum (in CCl_4) showed a doublet (J_{HF} 44.6 Hz) at 4.9 ppm upfield from internal hexafluorobenzene; its proton NMR spectrum agreed with the published[2] spectrum. The products derived from CClF addition to norbornene also have been reported previously[11]: 3-chloro-3-*syn*-fluorotricyclo[3.2.1.$0^{2,4}$]octane, n_D^{25} 1.4814 (reported[11] n_D^{25} 1.4800) and 3-fluoro-4-*exo*-chlorobicyclo-[3.2.1]-2-octene, n_D^{25} 1.4971 (reported[11] n_D^{25} 1.4962). The IR and NMR spectra of these products were in agreement with those reported.

New compounds (Physical and spectroscopic data of syn/anti isomer mixtures)

1-*Fluoro*-1-*chloro*-2-(*trimethylsilylmethyl*)*cyclopropane*. n_D^{25} 1.4241. IR (liquid film): 3006 w, 2959 s, 2924 m, 2896 m, 2805 w, 1448 m, 1423 m, 1373 w, 1278 (sh), 1255 s, 1192 s, 1161 s, 1094 m, 1062 w, 1041 w, 1023 m, 982 s, 960 m, 944 m, 935 m, 920 w, 870 (sh), 835 s, 776 w, 760 m, 695 s cm^{-1}. (Found: C, 46.88; H, 7.97; Cl, 19.59. $C_7H_{14}ClFSi$ calcd.: C, 46.54; H, 7.76; Cl, 19.63%.)

1-*Fluoro*-1-*chloro*-2-*bromo*-2,3,3-*trimethylcyclopropane*, n_D^{25} 1.4687. NMR (in CCl_4): δ 1.23 and 1.38 (s, 6H, Me_2C) and 1.84 ppm (s, 3H, Me–C–Br). (Found: C, 33.42; H, 4.24; Br, 37.64. C_6H_9BrClF calcd.: C, 33.44; H, 4.21; Br, 37.08%.)

The ^{19}F NMR spectrum (in CCl_4) showed two singlets at 26.6 and 14.6 ppm downfield from internal hexafluorobenzene, with intensity ratio 1.4/1.

1-*Fluoro*-1,2-*dichloro*-3,3-*dimethylcyclopropane*. n_D^{25} 1.4329. NMR (in CCl_4): δ 1.21 and 1.27 (s, 6H, Me_2C), 2.93 (s, 1H, H *trans* to F) and 3.10 ppm (d, 1H, J_{HF} 17 Hz, H *cis* to F). (Found: C, 38.40; H, 4.47; Cl, 44.88. $C_5H_7Cl_2F$ calcd.: C, 38.24; H, 4.50; Cl, 45.16%.) (A minor by-product from this reaction of $Me_2C{=}CHCl$ and $PhHgCCl_2F$ was identified as 1-fluoro-1-chloro-2-methyl-2-chloromethylcyclopropane (see below), a consequence of the $CH_2{=}C(Me)CH_2Cl$ impurity in the starting olefin).

1-*Fluoro*-1-*chloro*-2-*methyl*-2-*chloromethylcyclopropane*. n_D^{25} 1.4408. NMR (in CCl_4): δ 1.0–1.7 (m, 2H, ring CH_2), 1.44 (s, 3H, CH_3) and 3.58 ppm (s, 2H, CH_2Cl). (Found: C, 38.40; H, 4.46. $C_5H_7Cl_2F$ calcd.: C, 38.24; H, 4.50%.)

1-*Fluoro*-1-*chloro*-2-*triethylsilylcyclopropane*. n_D^{25} 1.4483. ^{19}F NMR (in CCl_4): multiplets at 40.6 and 29.4 ppm downfield from internal hexafluorobenzene, intensity ratio 1.1/1. (Found: C, 51.81; H, 8.59; Cl, 17.38. $C_9H_{18}ClFSi$ calcd.: C, 51.77; H, 8.69; Cl, 16.98%.)

1-*Phenyl*-2-*fluoro*-2,3,3-*trichloroaziridine*. n_D^{25} 1.5378. IR (liquid film): 3074 w, 3050 w, 1602 s, 1498 s, 1458 m, 1380 s, 1364 (sh), 1246 w, 1104 (sh), 1078 s, 1068 (sh), 1034 w, 980 m, 899 m, 875 s, 759 (sh), 746 s, and 693 cm^{-1}. (Found: C, 40.16; H, 2.11; Cl, 43.89; N, 5.76. $C_8H_5Cl_3FN$ calcd.: C, 39.95; H, 2.10; Cl, 44.23; N, 5.83%.)

1-*Fluoro*-1-*chloro*-2,2-*diphenylethylene*. M.p. 40–41° (lit.[12] m.p. 42–42.5°), was the product isolated from the reaction of $PhHgCCl_2F$ with thiobenzophenone. In this reaction, the filtrate after removal of PhHgCl was concentrated. Attempts to recrystallize the residue from ethanol led to formation of sulfur. The ethanol was evaporated and the residue was chromatographed on a 12 inch alumina column, using hexane as eluant. IR (film): 3096 w, 3078 m, 3050 m, 1638 s, 1601 (sh), 1497 s, 1448 s, 1200 s, 1114 s, 1079 w, 1040 w, 1007 w, 945 m, 920 w, 853 s, 769 (sh), 762 s, 702 s, 681 m, 624 m cm^{-1}.

CClF Transfer from phenyl(fluorodichloromethyl)mercury using the sodium iodide procedure

A flask equipped as described for the thermal reactions was charged with one molar equivalent of the mercurial, one to three molar equivalents of the carbenophile and an appropriate quantity of carefully dried, peroxide-free 1,2-dimethoxyethane (DME). This solution was heated to reflux and then a solution of well-dried (24 h at 150° and 0.02 mmHg) sodium iodide (1.1 molar equivalents) in DME was added slowly with stirring and under nitrogen. As the NaI was added, a bright yellow color developed which was soon discharged and phenylmercuric iodide and sodium chloride precipitated rapidly. The reaction mixture was heated and stirred at reflux for 3 h, cooled and filtered. The solids thus removed were washed with water to remove NaCl and dried to give phenylmercuric iodide. The filtrate was trap-to-trap distilled and the distillate was examined by GLC.

A reaction of 17.3 mmol of $PhHgCCl_2F$ and 20.6 mmol NaI in 30 ml of DME in the presence of 62 mmol of cyclooctene (3 h at 85°) gave phenylmercuric iodide (87%) and 9-fluoro-9-chlorobicyclo[6.1.0]nonane (85%).

A reaction of $PhHgCCl_2F$ (16.2 mmol) and sodium iodide (19.0 mmol) in 75 ml of DME in the presence of 19.5 mmol of $PhN{=}CCl_2$ (3 h at 85°) gave PhHgI in 62% yield and 1-phenyl-2-fluoro-2,3,3-trichloroaziridine in 70% yield.

A reaction of $PhHgCCl_2F$ (15.5 mmol) and sodium iodide (17.5 mmol) in 75 ml of DME in the presence of 15.3 mmol of thiobenzophenone (3 h at 85°) gave PhHgI in 67% yield. Work-up by column chromatography as in the thermal reaction gave 1-fluoro-1-chloro-2,2-diphenylethylene, m.p. 40–41°, in 76% yield, based on $Ph_2C{=}S$. ^{19}F NMR (in CCl_4) showed a singlet at 84.5 ppm downfield from internal hexafluorobenzene.

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