HALOMETHYL–METAL COMPOUNDS
XXVII*. THE REACTION OF PHENYL(DIHALOMETHYL)MERCURY COMPOUNDS WITH ORGANOSILICON HYDRIDES: A PREPARATION OF HALOMETHYL–SILICON COMPOUNDS**

DIETMAR SEYFERTH, S. BRIAN ANDREWS AND HARRY D. SIMMONS, JR.***

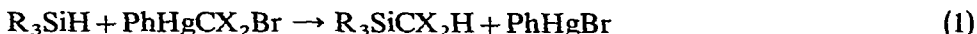*Department of Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139 (U.S.A.)*

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SUMMARY

Phenyl(dihalomethyl)mercury compounds, $PhHgCBr_2H$ and $PhHgCClBrH$, have been found to transfer CHBr and CHCl, respectively, to organosilicon hydrides, forming (bromomethyl)- and (chloromethyl)silanes. Such reactions appear to be of preparative utility only in the case of alkylsilicon hydrides. (Bromomethyl)triethylgermane also was prepared in moderate yield by this procedure. (Dibromomethyl)-mercuric bromide by itself was not a CHBr transfer agent, but did become effective in this application when an equivalent of diphenylmercury was added to it. It was found that diphenylmercury "activates" (dibromomethyl)mercuric bromide by reacting with it to form phenylmercuric bromide and phenyl(dibromomethyl)-mercury.

INTRODUCTION

During our survey of the CX_2 transfer chemistry of phenyl(trihalomethyl)-mercury compounds, we discovered their utility in the preparation of (dihalomethyl)-silanes and -germanes via dihalocarbene insertion into the Si–H and Ge–H bonds (eqns. 1 and 2)[3,4,5]. Although (dihalomethyl)silanes are now beginning to find use as

$$R_3SiH + PhHgCX_2Br \rightarrow R_3SiCX_2H + PhHgBr \qquad (1)$$
$$R_3GeH + PhHgCX_2Br \rightarrow R_3GeCX_2H + PhHgBr \qquad (2)$$

intermediates in the preparation of novel halogenated organosilicon derivatives[6,7], they are not generally useful materials. On the other hand, (monohalomethyl)silanes are valuable intermediates in preparative organosilicon chemistry[8,9]. For this reason, when phenyl(dihalomethyl)mercury compounds, especially $PhHgCBr_2H$ and $PhHg$-$CClBrH$, became available[10-13], we began an investigation of their possible use in the synthesis of (monohalomethyl)silanes and -germanes.


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RESULTS AND DISCUSSION

The reactions of phenyl(bromodichloromethyl)mercury and phenyl(dibromochloromethyl)mercury with triethylsilane proceed[3,4] rapidly and essentially quantitatively in benzene solution at 80°. Such is not the case for phenyl(dibromomethyl)-mercury and phenyl(bromochloromethyl)mercury reactions with this silane. A typical preparation, that of (bromomethyl)triethylsilane (eqn. 3) required heating at 130° in chlorobenzene for *ca.* 35 h in order to obtain the yields indicated. The reaction did

$$Et_3SiH + PhHgCBr_2H \rightarrow PhHgBr + Et_3SiCH_2Br \qquad (3)$$
$$(85\%) \qquad (61\%)$$
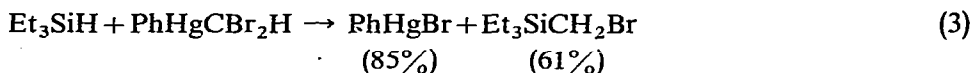
not proceed at a usefully rapid rate at 80°. Phenyl(bromochloromethyl)mercury is even less reactive, a reaction time of 142 h being required in its halomethylenation of triethylsilane (eqn. 4).

$$Et_3SiH + PhHgCBrClH \rightarrow PhHgBr + Et_3SiCH_2Cl \qquad (4)$$
$$(80\%) \qquad (72\%)$$
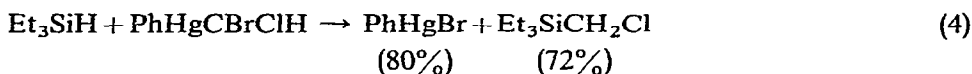
Furthermore, a complication was introduced by the formation as well of (bromomethyl)triethylsilane (7%) in this reaction. Since the bromochloromethane used in the preparation of the PhHgCBrClH used was free of dibromomethane, this formation of the bromomethyl–silane must involve the process shown in eqn. (5).

$$Et_3SiH + PhHgCBrClH \rightarrow PhHgCl + Et_3SiCH_2Br \qquad (5)$$
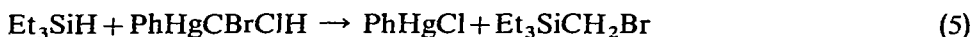
The relatively high reaction temperature of 130° used in the present instance very probably is responsible for the observed lack of specificity. Note also the case of PhHgCClBrCF$_3$[14].

Further reactions of these phenyl(dihalomethyl)mercurials with organosilicon hydrides and with triethylgermane were carried out in order to assess the general applicability of these mercurials in the synthesis of monohalomethyl derivatives of silicon and germanium. The results are presented in Table 1.

The yields listed in Table 1 show clearly that only the conversion of trialkylsilanes to (halomethyl)trialkylsilanes is effected in good yield with the PhHgCXBrH reagents. Reactions with arylsilanes were very unsatisfactory. In addition to the experiment with triphenylsilane listed in Table 1, attempts were made to bromomethylenate diphenylsilane. However, in these reactions only trace amounts of liquids with infrared spectroscopic properties characteristic of the expected products were obtained. With diethylsilane the product yields also were low, and noteworthy is the low yield of Et$_2$Si(CH$_2$Br)$_2$ realized in a reaction of phenyl(dibromomethyl)mercury and diethylsilane in a molar ratio of two. While these low yields may be of significance in terms of the reaction mechanism involved, the picture is further complicated by the high reaction temperatures and the long reaction times required and the fact that some of the starting materials (particularly R$_2$SiH$_2$ and RSiH$_3$ compounds) and some of the products are not stable to phenylmercuric bromide and/or phenyl(dihalomethyl)mercurials. Thus, only 40% of the originally charged (bromomethyl)triphenylsilane could be recovered after it had been heated with one-half molar equivalent each of PhHgCBr$_2$H and PhHgBr in refluxing chlorobenzene for 24 h. A similar treatment of Et$_2$HSiCH$_2$Br resulted in massive decomposition, the formation of
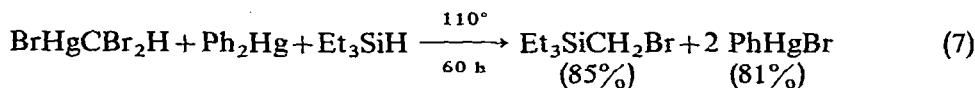
TABLE 1

REACTIONS OF PhHgCBr$_2$H AND PhHgCClBrH WITH ORGANOSILICON HYDRIDES AND TRIETHYLGERMANE IN CHLOROBENZENE AT 130°

Mercurial	Hydride	Ratio mercurial-to-hydride	Reaction time (h)	Product (% yield)	PhHgBr(%)
PhHgCBr$_2$H	Et$_3$SiH	1/1.9	34.5	Et$_3$SiCH$_2$Br (61)	85
PhHgCBr$_2$H	Et$_3$SiH	1/3 (in toluene at 109°)	27	Et$_3$SiCH$_2$Br (65)	79
PhHgCBr$_2$H	Et$_3$SiH	Et$_3$SiH as solvent (at 109°)	27	Et$_3$SiCH$_2$Br (72)	85
PhHgCClBrH	Et$_3$SiH	1/1.94	142	{Et$_3$SiCH$_2$Cl (72) {Et$_3$SiCH$_2$Br (7)	80
PhHgCBr$_2$H	n-Bu$_3$SiH	1/1.9	23.5	n-Bu$_3$SiCH$_2$Br (80)	85
PhHgCClBrH	n-Bu$_3$SiH	1/1.65	144	{n-Bu$_3$SiCH$_2$Cl (76) {n-Bu$_3$SiCH$_2$Br (3)	
PhHgCBr$_2$H	Et$_2$SiH$_2$	1.1/1	36	{Et$_2$HSiCH$_2$Br (42) {Et$_2$Si(CH$_2$Br)$_2$ (2)	
PhHgCBr$_2$H	Et$_2$SiH$_2$	2/1	36	{Et$_2$HSiCH$_2$Br (44) {Et$_2$Si(CH$_2$Br)$_2$ (13)	65
PhHgCBr$_2$H	n-BuSiH$_3$	1.1/1	36	{n-BuH$_2$SiCH$_2$Br (28) {n-BuHSi(CH$_2$Br)$_2$ (3)	54
PhHgCBr$_2$H	Ph$_3$SiH	1/1	20	Ph$_3$SiCH$_2$Br (4)	34
PhHgCBr$_2$H	Et$_3$GeH	1/1	66	Et$_3$GeCH$_2$Br (28)	70

Et$_2$Si(CH$_2$Br)$_2$ in 14% yield representing the only nondestructive process which took place. Triethylgermane also appears to be quite reactive toward phenylmercuric bromide, since the (bromomethyl)triethylgermane produced was accompanied by an equal amount (30% yield) of triethylgermane produced was accompanied by an equal amount (30% yield) of triethylbromogermane. The reaction shown in eqn. (6) no doubt is responsible for the formation of the latter.

$$Et_3GeH + PhHgBr \longrightarrow Et_3GeBr + \tfrac{1}{2} Ph_2Hg + \tfrac{1}{2} Hg + \tfrac{1}{2} H_2 \qquad (6)$$
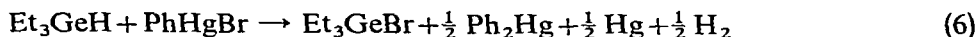
In addition to phenyl(dibromomethyl)mercury, (dibromomethyl)mercuric bromide[12] was examined as a potential CHBr-transfer agent. This compound, however, failed to react under conditions where PhHgCBr$_2$H converted triethylsilane to (bromomethyl)triethylsilane in 73% yield. In a previous study, we had found that (bromomethyl)mercuric bromide, which is unreactive as a CH$_2$ transfer reagent toward cyclohexene, can be used as a methylenating reagent when an equivalent of diphenylmercury is added to the reacting system[15]. A similar "activation" of (dibromomethyl)mercuric bromide could be achieved, as indicated by eqn. (7). The explanation for this "activation" is quite simple: (dibromomethyl)mercuric bromide and diphenylmercury react readily to form phenylmercuric bromide and the active CHBr-transfer agent, phenyl(dibromomethyl)mercury, as a separate experiment in which these products actually were isolated showed.

$$\text{BrHgCBr}_2\text{H} + \text{Ph}_2\text{Hg} + \text{Et}_3\text{SiH} \xrightarrow[\substack{60\text{ h}}]{110°} \underset{(85\%)}{\text{Et}_3\text{SiCH}_2\text{Br}} + \underset{(81\%)}{2\text{ PhHgBr}} \qquad (7)$$

In summary, the PhHgCXBrH reagents are useful in the preparation of halo-methyl–silicon compounds only in their reactions with trialkylsilanes. The mechanism of this halomethylenation of the Si–H and Ge–H bonds is not known at the present time. In particular, in the absence of concrete evidence, we hesitate to assume that insertion of free CHCl or CHBr into these bonds is occurring. Further studies hopefully will provide an answer to this question of mechanism.

EXPERIMENTAL

General comments

All experiments were carried out under an atmosphere of prepurified nitrogen. Gas–liquid partition chromatographic (GLPC) analyses were carried out using an M.I.T. isothermal unit (8 ft or 4 ft, 20% General Electric Co. SE–30 silicone rubber gum on Chromosorb W), an F&M Model 720 gas chromatograph (5 ft, SE–30 or General Electric Co. XF–1150 silicone fluid on Chromosorb W), an F&M Model 700 (3 ft, SE–30 or 10% Dow Corning Corp. DC-200 silicone oil on Chromosorb W) and an F&M Model 5754 (4ft, 10% Union Carbide Corp. UC-W98 silicone rubber or 6 ft DC-200 on Chromosorb W). Infrared spectra were recorded using Perkin–Elmer Infracord Model 237B or 337 spectrophotometers, NMR spectra using Varian Associates A60 or T60 spectrometers. Chemical shifts are reported in ppm downfield from internal tetramethylsilane. Where possible, mercurial reactions were monitored by thin-layer chromatography[16]. Triethylsilane, triphenylsilane, tri-n-butylsilane and diphenylsilane were purchased from Peninsular ChemResearch, Inc. Literature procedures were used to prepare the phenyl(dihalomethyl)mercury compounds[12,13], diethylsilane[17], n-butylsilane[18] and triethylgermane[19].

Reaction of phenyl(dibromomethyl)mercury with triethylsilane

The preparation of (bromomethyl)triethylsilane is described in detail. All other reactions were carried out in similar manner, with any differences noted below.

A 100 ml, three-necked flask equipped with a reflux condenser, magnetic stirring assembly and nitrogen inlet tube was flame-dried under nitrogen and charged with 4.44 g (38.1 mmoles) of triethylsilane, 9.01 g (20.0 mmoles) of phenyl(dibromomethyl)mercury and 60 ml of dry chlorobenzene. The solution was stirred and heated at reflux (130°) for 34.5 h. Filtration gave phenylmercuric bromide, m.p. 274–279°, in 85% yield. Trap-to-trap distillation of the filtrate at 0.05 mm (pot temperature to 80°) followed. A sample of the distillate was saved for GLPC yield analysis; the remainder was concentrated at reduced pressure and the residue was examined by GLPC. A single product, $\text{Et}_3\text{SiCH}_2\text{Br}$, was present, n_D^{25} 1.4672.(Found: C, 40.42; H, 8.12; Br, 38.45. $\text{C}_7\text{H}_{17}\text{BrSi}$ calcd.: C, 40.25; H, 8.20; Br, 38.26%.) The yield, determined by GLPC, was 61%. NMR (in CCl_4): 0.4–1.2 (15 H, Et_3Si–) m; 2.38 (2 H, $-\text{CH}_2\text{Br}$) s. IR (liquid film): 2960s, 2939s, 2912s, 2878s, 2800m, 2724w, 1464m, 1454m, 1413m, 1387m, 1376m, 1256sh, 1237m, 1127m, 1075sh, 1050w, 1015s, 971m, 944sh, 800sh, 760s and 731s cm^{-1}.

Reaction of phenyl(dibromomethyl)mercury with tri-n-butylsilane

The silane (19.0 mmoles) and the mercurial (10.0 mmoles) in 20 ml of chlorobenzene were heated for 23.5 h. Phenylmercuric bromide was isolated in 85% yield. (Bromomethyl)tri-n-butylsilane, n_D^{25} 1.4678, was obtained in 79% yield. (Found: C, 53.20; H, 10.17; Br. 27.44. $C_{13}H_{29}BrSi$ calcd.: C, 53.22; H, 9.96; Br, 27.44%). NMR (in CCl_4): 0.4–1.6 (27 H, Bu_3Si-) m; 2.44 (2 H, $-CH_2Br$) s. A minor product, formed in *ca.* 3% yield, was identified tentatively as tri-n-butylbromosilane.

Reaction of phenyl(dibromomethyl)mercury with triphenylsilane

The silane (10.0 mmoles) and the mercurial (10.5 mmoles) in 20 ml of chlorobenzene were heated at 130° for 20 h. Phenylmercuric bromide was isolated in 37% yield. Column chromatography of the residue obtained on evaporating the filtrate (acid-washed alumina, hexane eluent) gave triphenylsilane (60% recovery) and (bromomethyl)triphenylsilane, m.p. 118–120° (lit.[20] m.p. 120–121°), in 4% yield. An analytical sample, recrystallized from hexane, had m.p. 121–122.5°. (Found: C, 64.45; H, 4.88. $C_{19}H_{17}BrSi$ calcd: C, 64.58; H, 4.85%). NMR (in CCl_4): 3.05 (2 H, $-CH_2Br$) s; 7.2–7.7 (15H, Ph_3Si-) m.

Reaction of phenyl(dibromomethyl)mercury with diethylsilane

The silane (10.7 mmoles) and the mercurial (10.0 mmoles) in 20 ml of dry chlorobenzene were sealed under nitrogen in a 50 ml Pyrex combustion tube and heated in an autoclave rocker for 36 h at 130°. The products obtained were (bromomethyl)diethylsilane (42%), bis(bromomethyl)diethylsilane (7%) and a minor amount of *sym*-tetraethyldisiloxane.

$Et_2(CH_2Br)SiH$. (Found: C, 32.95; H, 6.94; Br, 43.77. $C_5H_{13}BrSi$ calcd: C, 33.14; H, 7.23; Br, 44.11%.) NMR (in CCl_4): 0.4–1.2 (10 H, Et_2Si-) m; 2.42 (2 H, $-CH_2Br$) d (*J* 2 Hz); 3.94 (1 H, SiH) unsym. quintet. In the infrared spectrum (liquid film), $v(Si-H)$ was observed at 2130 cm^{-1}.

$Et_2Si(CH_2Br)_2$. (Found: C, 26.68: H, 5.04; $C_6H_{14}Br_2Si$ calcd.: C, 26.29; H, 5.15%.) NMR (in CCl_4): 0.6–1.3 (10 H, Et_2Si-) m; 2.58 (4 H, $-CH_2Br$) s.

$(Et_2HSi)_2O$. This by-product was identified on the basis of its infrared spectrum (liquid film) which showed $v(Si-H)$ at 2114 cm^{-1} and strong absorption due to Si–O–Si at 1070 cm^{-1}, and which was in good agreement with the IR spectrum reported for this compound by Russian workers[21].

Reaction of phenyl(dibromomethyl)mercury with n-butylsilane

In a similar bomb tube reaction, 11.3 mmoles of the silane and 10.0 mmoles of the mercurial in 20 ml of chlorobenzene were heated for 36 h at 130°. Phenylmercuric bromide was isolated in 54% yield. The major volatile product was (bromomethyl)-n-butylsilane (28% yield). (Found: C, 33.57; H, 7.29. $C_5H_{13}BrSi$ calcd: C, 33.14; H, 7.23%.) NMR (in CCl_4): 0.7–1.7 (9 H, $n-C_4H_9Si$) m; 2.57 (2 H, $-CH_2Br$) t (*J* 3 Hz); 4.10 (2 H, SiH_2) unsym. quintet. In the IR spectrum (liquid film) $v(Si-H)$ was observed at 2159 cm^{-1} (2150, sh). Bis(bromomethyl)-n-butylsilane was a minor (*ca.* 3%) product, the small amount of which only allowed its spectroscopic characterization. NMR (in CCl_4): 1.0–2.0 (9 H, $n-C_4H_9$) m; 2.92 (4 H, $-CH_2Br$) d (*J* 3 Hz); 4.51 (1 H, SiH) unsym. quintet. $v(Si-H)$ was observed at 2147 cm^{-1} in the liquid film IR spectrum. A second minor product (*ca.* 2% yield) was *sym*-di-n-butylsiloxane, $(n-BuH_2-$

Si)$_2$O, n_D^{25} 1.4169 (lit.[18] n_D^{20} 1.4181). Its infrared spectrum (liquid film) showed the Si–H stretch at 2135 cm^{-1} and strong Si–O–Si absorption at 1070 cm^{-1}.

Reaction of phenyl(dibromomethyl)mercury with triethylgermane

A reaction of this germane (7.13 mmoles) with 7.50 mmoles of the mercurial for 66 h gave phenylmercuric bromide in 70% yield. The volatile products were triethylbromogermane (30% yield), identified by comparison of its GLPC retention time and its IR spectrum with those of an authentic sample, and (bromomethyl)triethylgermane (28% yield). Found: C, 33.16; H, 6.60. C$_7$H$_{17}$BrGe calcd: C, 33.13; H, 6.76. NMR (in CCl$_4$): 0.4–1.2 (15 H, Et$_3$Ge–) m; 2.82 (2 H, –CH$_2$Br) s. IR (liquid film): 2939s, 2920sh, 2890s, 2856s, 2800 m, 2708w, 1464s, 1448sh, 1425m, 1389sh, 1377m, 1230w, 1122m, 1050w, 1021s, 969m, 756sh, 740sh, 705s and 684sh cm^{-1}.

Reaction of phenyl(bromochloromethyl)mercury with triethylsilane

A reaction of 18.9 mmoles of the silane and 10.0 mmoles of the mercury compound in 20 ml of chlorobenzene at 130° for 142 h gave phenylmercuric halide in 80% yield. The major volatile product was (chloromethyl)triethylsilane, n_D^{25} 1.4452 (lit.[22] n_D^{25} 1.4452), in 72% yield. NMR (in CCl$_4$): 0.3–1.2 (15H, Et$_3$Si–) m; 2.77 (2H, –CH$_2$-Cl)s. In addition, there were two minor products: triethylchlorosilane, obtained in *ca.* 15% yield and identified by comparison of its GLPC retention time, IR spectrum and mass spectrum with those of an authentic sample, and (bromomethyl)triethylsilane (7%).

Reaction of phenyl(bromochloromethyl)mercury with tri-n-butylsilane

A reaction of the silane (16.5 mmoles) and the mercurial (10.0 mmoles) in 20 ml of chlorobenzene at reflux for 144 h gave (chloromethyl)tri-n-butylsilane in 76% yield. (Found: C, 62.82; H, 11.71. C$_{13}$H$_{29}$ClSi calcd: C, 62.72; H, 11.74%.) NMR (in CCl$_4$): 0.4–1.5 (27 H, Bu$_3$Si–) m; 2.70 (2 H, –CH$_2$Cl) s. In addition, (bromomethyl)-tri-n-butylsilane was obtained in *ca.* 3% yield. Two further products were tri-n-butyl-chlorosilane (*ca.* 15%) and tri-n-butylbromosilane (*ca.* 4%).

Reaction of (dibromomethyl)mercuric bromide/diphenylmercury with triethylsilane

A mixture of 2.27 g (5 mmoles) of BrHgCBr$_2$H and 1.77 g (5 mmoles) of diphenylmercury in 25 ml of triethylsilane was heated at reflux (109°), with stirring and under nitrogen, for 27 h. Phenylmercuric bromide, 2.9 g (81%) was filtered. The yield of (bromomethyl)triethylsilane, determined by GLPC, was 85%.

A similar reaction using (dibromomethyl)mercuric bromide without addition of diphenylmercury gave none of this product, but the formation of a small amount of triethylbromosilane suggested that reduction of Hg–Br had occurred to a minor extent

A reaction of phenyl(dibromomethyl)mercury with neat triethylsilane at reflux for 27 h gave (bromomethyl)triethylsilane in 73% yield.

Reaction of (dibromomethyl)mercuric bromide with diphenylmercury

A mixture of 7.10 g (0.02 mole) of diphenylmercury and 9.06 g (0.02 mole) of BrHgCBr$_2$H in benzene was heated at reflux under nitrogen for 54 h. The yellow reaction mixture was filtered to remove 7.04 g of crude (m.p. 269–278°) phenylmer-

curic bromide (98% yield). Cooling of the filtrate gave 6.16 g of crude phenyl(dibromo-methyl)mercury (70% yield), m.p. 63–68°, identified further by its characteristic infrared spectrum[12]. Small amounts of solid, melting range 140–170° and m.p. 225–228°, and a residual yellow oil were not investigated further.

ACKNOWLEDGEMENTS

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