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HALOMETHYL–METAL COMPOUNDS

LI*. AN IMPROVED SYNTHESIS OF α-IODOALKYL DERIVATIVES OF TIN AND MERCURY BY THE ORGANOZINC ROUTE. BIS[(TRIMETHYLSILYL)IODOMETHYL]MERCURY

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SUMMARY

The homogeneous solution of iodomethylzinc iodide produced by reaction of equimolar quantities of ethylzinc iodide and diiodomethane in tetrahydrofuran was used to prepare Me_3SnCH_2I, $Me_2Sn(CH_2I)_2$ and $Hg(CH_2I)_2$ in good yield; this procedure is much superior to that using the reagent prepared from the reaction of diiodomethane with zinc/copper couple. The reaction of ethylzinc iodide with CH_3CHI_2, Me_3SiCHI_2 and Me_3SnCHI_2 in THF gave good yields of $CH_3CHIZnI$, $Me_3SiCHIZnI$ and $Me_3SnCHIZnI$, respectively. These reagents were used to prepare, generally in high yield, the following compounds: $Me_3SnCHICH_3$, $Hg(CHICH_3)_2$, $Me_3SiCHISnMe_3$, $Hg(CHISiMe_3)_2$ and $(Me_3Sn)_2CHI$. The $Hg(CHISiMe_3)_2/Ph_2Hg$ reagent pair was found to transfer Me_3SiCH to cyclohexene and cyclooctene, but the silylcyclopropane product yields were quite low.

INTRODUCTION

In the previous paper of this series[1], we reported that ICH_2ZnI and $BrCH_2ZnBr$ as obtained from reaction of the appropriate dihalomethane with zinc/copper couple served as alkylating agents on reaction with halides of germanium, tin, lead and mercury (eqn. 1). This new synthesis of this potentially useful class of compounds

$$XCH_2ZnX + MY \rightarrow MCH_2X + ZnXY \qquad (1)$$

$$X = Br, I;\ Y = Cl, Br, I;\ M = R_3Sn, R_3Pb, Cl_3Ge, YHg$$

represented a great improvement over the previously available routes to such MCH_2X compounds. However, some features of the experimental procedure made these syntheses somewhat more cumbersome than we would have liked. In particular, the zinc/copper couple had to be prepared under rigorously controlled conditions,

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the XCH_2ZnX reagent solutions had to be filtered for successful application and two molar equivalents of diiodomethane (and four of dibromomethane) were required to ensure good yields of MCH_2X product. We report here a substantially improved synthesis of iodomethyl derivatives of tin and mercury. An added bonus of this new procedure is its applicability to the synthesis of RCHI derivatives of these metals, *e.g.*, where $R=CH_3$, Me_3Si and Me_3Sn.

Some time after the original report of the utility of ICH_2ZnI in cyclopropane synthesis[2], Furukawa and coworkers found that iodomethyl-, as well as substituted iodomethyl-zinc reagents could be prepared by treatment of the respective *gem*-diiodoalkane with diethylzinc (eqn. 2). The exact nature of the species present in such

$$RCHI_2+Et_2Zn \rightleftarrows EtZnI+EtZnCHIR \qquad (2)$$

solutions has not been elucidated. As indicated, zinc–iodine exchange is the first step, but further equilibria involving the compounds in eqn. (2) possibly may add to the number of zinc compounds present. Such reagent solutions served excellently in cyclopropane synthesis[3a–c], but their application as alkylating agents would not be practical since they apparently contain Et–Zn linkages as well as the desired RCHI–Zn bonds. Thus their treatment with MX_n compounds should give a mixture of products containing both ethyl and α-iodoalkyl groups. Indeed, the reaction of Furukawa's reagent ($Et_2Zn+CH_2I_2$) with triethyltin hydride gave tetraethyltin as main product[3d]. The following reaction course was suggested:

$$Et_3SnH+RI \rightarrow Et_3SnI+RH \qquad (3)$$

$$Et_3SnI+EtZnY \rightarrow Et_4Sn+ZnYI \qquad (4)$$

An interesting new variation of this procedure uses ethylzinc iodide rather than diethylzinc as the zinc source[4]. The simplest picture of this system is that shown in eqn. (5). However, the authors suggested that further equilibria (eqns. 6 and 7), whose

$$EtZnI+CH_2I_2 \rightleftarrows EtI+ICH_2ZnI \qquad (5)$$

occurrence would result in retention of Et–Zn linkages, were possible[4]. Furthermore, they suggested[4a], on the basis of very indirect evidence, that the active cyclopropanation

$$EtZnI \rightleftarrows Et_2Zn+ZnI_2 \xrightarrow{CH_2I_2} EtZnCH_2I+ZnI_2+EtI \qquad (6)$$

$$2\ EtZnCH_2I \rightleftarrows Et_2Zn+Zn(CH_2I)_2 \overset{ZnI_2}{\rightleftarrows} Et_2Zn+2\ ICH_2ZnI \qquad (7)$$

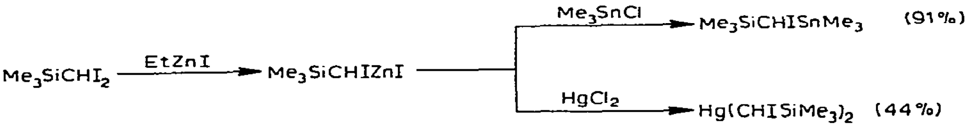
reagent in $EtZnI/CH_2I_2$/olefin reactions is $EtZnCH_2I$. In spite of their conjecture on this point, we felt that it was more likely that the products of eqn. (5) actually were those present in the reagent solution and thus were led to conclude that the $EtZnI/CH_2I_2$ reagent should be a useful alternative to the previously developed $Zn/Cu/CH_2I_2$ system for nucleophilic ICH_2 transfer to heavy metal atoms. The experiments described below provide full confirmation of these ideas.

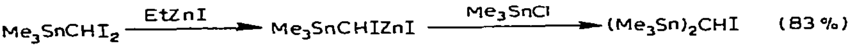
RESULTS AND DISCUSSION

In our initial experiment, a mixture of equimolar quantities of ethylzinc iodide and diiodomethane in THF solution was allowed to equilibrate for 1 h at 35–40°.

The addition of a molar equivalent of trimethyltin chloride followed. After $2\frac{1}{2}$ h the reaction mixture was hydrolyzed. Analysis by GLC showed that trimethyl(iodomethyl)tin had been formed in 78% yield. Of importance was the further observation that *no* ethyltrimethyltin had been produced. Thus it would appear that the reagent system is much simpler than its original developers[4] believed, very probably containing only one major organozinc species, ICH_2ZnI. This variation of our original procedure[1] has several important advantages: the active organozinc reagent is much more easily prepared; the reagent solution is homogeneous, which eliminates the need for filtration under an inert atmosphere; the reagent components need not be used in excess; shorter reaction times can be used. In other experiments the $EtZnI/CH_2I_2$ reagent was used to prepare $Me_2Sn(CH_2I)_2$ (71% yield) and $Hg(CH_2I)_2$ (67% yield).

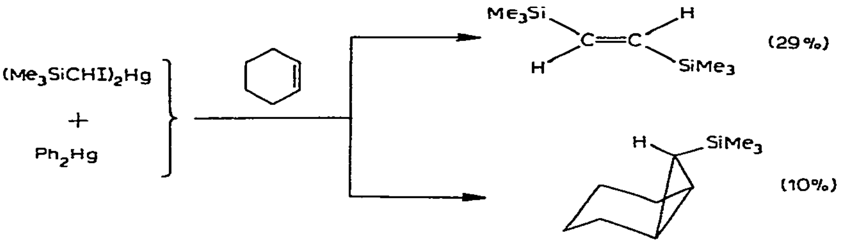
Like diethylzinc, ethylzinc iodide can be used to generate substituted α-iodoalkylzinc species. Reaction of EtZnI with 1,1-diiodoethane resulted in formation of $CH_3CHIZnI$, which on treatment with trimethyltin chloride gave $Me_3SnCHICH_3$ (75%), and with mercuric chloride in 2/1 molar ratio, $Hg(CHICH_3)_2$ (60%). Recently we have prepared Me_3SiCHI_2[5] and Me_3SnCHI_2[6], and these also react with ethylzinc iodide to give $Me_3SiCHIZnI$ and $Me_3SnCHIZnI$, respectively. These reagents show nucleophilic reactivity comparable to that of ICH_2ZnI:



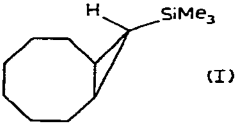


It is obvious that this new approach to the synthesis of iodoalkyl derivatives of heavy metals is capable of further extention. The availability of other *gem*-diiodoalkanes, both wholly organic and organometallic, via simple synthetic routes should make available other new and useful RCHIZnI reagents. Such extentions of scope are receiving our attention.

The mercury compound $(Me_3SiCHI)_2Hg$ prepared during the course of this study was of interest as a potential Me_3SiCH transfer agent. In previous research we had shown $(Me_3SiCCl_2)_2Hg$ and $(Me_3SiCBr_2)_2Hg$ to be moderately good sources of Me_3SiCCl and Me_3SiCBr, respectively[7]. On the other hand, $(Me_3SiCHBr)_2Hg$ proved far too stable to serve as a Me_3SiCH transfer agent[7]. Since α-elimination of Hg–I from an α-iodomethylmercury compound occurs more readily than Hg–Br elimination from the analogous α-bromomethylmercury compound[8], our new $(Me_3SiCHI)_2Hg$ seemed of potential value. However, our experiments showed this compound to be quite stable. A 1/1 molar mixture of $(Me_3SiCHI)_2Hg$ and diphenylmercury did not react with cyclohexene during 3 days at reflux. It required a reaction period of 7 days at 148° (sealed tube) to accomplish decomposition of this mercurial in cyclohexene solution. Phenylmercuric iodide was formed in good yield, but the cyclopropanation reaction was less favored than formal dimerization of Me_3SiCH:



Only the isomers indicated were detected. A similar decomposition of a $(Me_3SiCHI)_2Hg/Ph_2Hg$ mixture in refluxing cyclooctene solution (ca. 140°) during 9 days gave *anti*-9-(trimethylsilyl)bicyclo[6.1.0]nonane, (I), in 10% yield. It would appear that $(Me_3SiCHI)_2Hg$ is a Me_3SiCH transfer agent of only marginal utility.



(I)

EXPERIMENTAL

General comments

For the most part, the general comments of the previous paper of this series[1] apply. Most experiments involving the utilization or formation of iodinated materials (especially the $RCHI_2$ compounds) were carried out in minimum possible light. A General Electric red-filtered infrared lamp was found useful in this connection. Literature procedures were followed in the preparation of 1,1-diiodoethane[9], trimethyl(diiodomethyl)silane[5] and trimethyl(diiodomethyl)tin[6].

Preparation of ethylzinc iodide

A modification of the procedure of Abraham and Rolfe[10] was used. To 32.7 g (0.50 g-atom) of dry zinc/copper couple, prepared by the method of LeGoff[11], in a dry, nitrogen-filled three-necked flask equipped with a mechanical stirrer, reflux condenser, dropping funnel and gas inlet tube was added 78.0 g (0.50 mole) of iodoethane. Following an initial, mildly exothermic reaction, the mixture was heated gently for 1 h, at which time the reaction mixture had beome too thick to stir. The volatile components were removed at reduced pressure, giving 26.5 g of distillate (34% recovery of iodoethane). The residual dry, white powder was extracted with 290 ml of dry THF under nitrogen and the resulting solution was filtered using a Schlenk filter. A 31% recovery (10.2 g) of unconverted metal was realized. The THF solution was transferred to a pint centrifuge bottle, using a canulla, and centrifuged. The resulting clear, water-white solution of EtZnI was transferred to a dry "pop" bottle under argon or nitrogen for storage. Titration of an aliquot of this solution with aqueous H_2SO_4 and measurement of the ethane evolved by standard techniques indicated the solution to be 1.14 *N* in EtZnI.

Reaction of $EtZnI/CH_2I_2$ with trimethyltin chloride

The procedure is described in detail to provide an example of the techniques used. A flame-dried, nitrogen-filled 100-ml three-necked flask equipped with a magnetic stirring unit, a reflux condenser, dropping funnel, gas inlet tube and no-air stopper was charged (via syringe) with 17.0 ml (19.5 mmole) of 1.14 *N* EtZnI in THF and 5.41 g (20.2 mmole) of diiodomethane. This mixture was allowed to equilibrate at 35–40° for 30–60 min. A solution of 4.0 g (20.1 mmole) of trimethyltin chloride in 15 ml of THF then was added dropwise with stirring. The reaction mixture was stirred at 35–40° for 2–2½ h. Subsequently, the mixture was poured into 100 ml of distilled water and 50 ml of hexane was added. After further treatment with four 100-ml portions of distilled water, the organic phase was dried, concentrated and trap-to-trap distilled at 0.03 mm. The distillate obtained (23.53 g) was analyzed by GLC (column B[1], 130°). A single high-boiling product, trimethyl(iodomethyl)tin, was present and ethyltrimethyltin, an authentic sample of which was available, was absent. Yield determination by GLC showed that Me_3SnCH_2I had been formed in 78% yield. A sample was isolated by distillation, b.p. 58–59° at 10 mm; its IR and NMR spectra were identical with those of authentic material[1].

A reaction carried out in similar fashion between 45 mmole of ICH_2ZnI prepared from EtZnI and CH_2I_2 and 20 mmole of dimethyltin dichloride, in THF, gave the expected $Me_2Sn(CH_2I)_2$[1] in 71% yield.

Reaction of EtZnI/CH_2I_2 with mercuric chloride

The reagent solution prepared from 44.5 mmole of EtZnI and 48.9 mmole of diiodomethane in THF was treated dropwise with stirring with a solution of 5.40 g (19.8 mmole) of mercuric chloride in 20 ml of dry THF. After the reaction mixture had been stirred at 40° for 3 h, it was concentrated at reduced pressure to 50 ml. Cooling of the residue provided 7.29 g of white cubic crystals in two crops. Recrystallization from 100 ml of chloroform gave 6.38 g (67%) of nearly pure $Hg(CH_2I)_2$, long white needles, m.p. 76–77.5°, lit.[10] m.p. 80–81.5°.

Reactions of $Me_3SiCHIZnI$

(*a*). *With trimethyltin chloride.* A solution of 20.0 ml (22.8 mmole) of 1.14 *N* EtZnI in THF and 6.54 g (19.2 mmole) of trimethyl(diiodomethyl)silane was stirred under nitrogen at 40° for 30 min and then treated with 17.9 mmole of trimethyltin chloride in 9 ml of THF. The mixture was stirred for 2 h at 40° and 16 h at 25°. The work-up as described for the Me_3SnCH_2I preparation above provided 6.24 g of distillate, b.p. 74–77° (2.3 mm). A 2 g center cut was better than 95% pure by GLC. GLC analysis of the total distillate showed the presence of 1.36 mmole of the starting silane and 16.3 mmole (91% yield) of product which was isolated by GLC (column D[1], 155°) and characterized as $Me_3SiCHISnMe_3$, n_D^{25} 1.5338. (Found: C, 22.20; H, 5.01; I, 33.76. $C_7H_{19}ISiSn$ calcd.: C, 22.30; H, 5.08; I, 33.67%.) NMR (in CCl_4): δ 0.18 (s, 9 H, Me_3Si), 0.25 [s, 9 H, $J(^{117}Sn–^1H)$ 51 Hz, $J(^{119}Sn–^1H)$ 53 Hz] and 1.73 ppm [s, 1 H, J(Sn–H) not observed, CHI]. IR (liquid film): 2990 sh, 2965 s, 2915 m, 1450 sh, 1405 m (broad), 1310 w, 1265 s, 1250 s, 1205 sh, 1195 m, 1040 m, 985 s, 860 s, 840 s, 775 s, 740 m, and 705 m, cm^{-1}.

(*b*). *With mercuric chloride.* The reagent prepared from 22.0 mmole of EtZnI and 23.8 mmol of Me_3SiCHI_2 in THF was treated with 2.44 g (9.0 mmole) of mercuric chloride in 15 ml of THF. After the reaction mixture had been stirred under nitrogen at 40° for 7 h, TLC[13] showed that two organomercury products were present. The work-up as described in the $Hg(CH_2I)_2$ preparation gave a white crystalline residue which upon recrystallization from methanol (250 ml) yielded 3.23 g (57%) of product mercurial which was pure by TLC, softening at ca. 80°, melting 122–130° with gas evolution. A further recrystallization from hexane provided 2.48 g (44%) of long needles, pure by TLC, m.p. 126–130° (dec). Another two recrystallizations gave an analytical sample, m.p. 129–130.5°, decomposition with gas evolution, of $(Me_3SiCHI)_2Hg$. (Found: C, 15.33; H, 3.27; I, 40.78. $C_8H_{20}I_2Si_2Hg$ calcd.: C, 15.33; H, 3.22; I, 40.49%.) NMR (in $CDCl_3$): δ 0.23 (s, 18 H, Me_3Si) and 2.18 ppm (s, 2H, CHI).

The other organomercuric product which had a shorter TLC retention time was present in only trace amounts in this experiment. However, a pure sample of this compound was obtained from a separate experiment using column chromatography 40 x 200 mm silicic acid (Mallinckrodt) column, hexane separation followed by benzene elution) and subsequent recrystallization from hexane. This material, m.p 89–90°, pure by TLC, was characterized as $Me_3SiCHIHgI$. It decomposed to mercuric iodide readily, upon standing or in solution. (Found: C, 8.96; H, 1.93; I, 47.21. $C_4H_{10}I_2SiHg$ calcd.: C, 8.88; H, 1.86; I, 46.95%.) NMR (in $CDCl_3$): δ 0.28 (s, 9 H, Me_3Si) and 2.77 (s, 1 H, CHI).

In another experiment in which the molar ratio of $Me_3SiCHIZnI$ to $HgCl_2$ was increased from 2.4 to 3.0, $Hg(CHISiMe_3)_2$ was obtained in 53% yield. Shorter reaction times resulted in diminished yields of this product.

Reactions of $CH_3CHIZnI$

(*a*). *With trimethyltin chloride.* A mixture of 22.0 mmole of EtZnI and 19.8 mmole of 1,1-diiodoethane[9] in THF was kept at 40° for 30 min. Subsequently, 18.0 mmole of Me_3SnCl in 9 ml of THF was added and the mixture was stirred under nitrogen at 40° for 2½ h. The usual work-up procedure for liquid products gave 4.29 g (75%) of trimethyl(1-iodoethyl)tin, b.p. 78–80° (15 mm), better than 98% pure by GLC (column A[1], 130°). A center cut, b.p. 79–79.5° (15 mm) had n_D^{25} 1.5410. (Found: C, 18.87; H, 4.10; I, 39.50. $C_5H_{13}ISn$ calcd.: C, 18.84; H, 4.11; I, 39.82%.) NMR (in CCl_4): δ 0.22 [s, 9 H, $J(^{117}Sn-^1H)$ 51 Hz, $J(^{119}Sn-^1H)$ 54 Hz, Me_3Sn], 2.05 (d, J 8 Hz, 3 H, J(Sn–H) 56 Hz, $CHCH_3$) and 3.28 ppm (q, J 8 Hz, 1 H, J(Sn–H) not observed, SnCHI).

(*b*). *With mercuric chloride.* A solution of $CH_3CHIZnI$ prepared from 26.4 mmole of EtZnI and 31.6 mmole of CH_3CHI_2 in THF was treated with 8.8 mmole of mercuric chloride in 15 ml of THF (dropwise). The addition of the second half of the $HgCl_2$ solution was accompanied by the precipitation of a quantity of metallic mercury as a dark gray powder. After the mixture had been stirred at 40° for 165 min, TLC[13] showed the presence of three organomercury products.

During the course of the standard work-up the characteristic odor of diethylmercury became evident, and suitable precautions were taken during subsequent operations. Crystallization of the concentrated residue from 4/1 hexane/chloroform afforded two crops of product, virtually pure by TLC, m.p. 83–86°, 2.72 g (60%). Successive recrystallizations from 4/1 hexane/chloroform and finally hexane alone afforded pure $(CH_3CHI)_2Hg$, m.p. 88.5–90°. (Found: C, 9.36; H, 1.47; I, 49.98. $C_4H_8I_2Hg$ calcd.: C, 9.41; H, 1.58; I, 49.72%.) NMR (in $CDCl_3$): δ 1.95 (d, J 8 Hz, 3 H, CH_3) and 3.74 ppm (q, J 8 Hz, 1H, CHI). In addition to these signals, extraneous signals appeared at δ 1.20 (d, J 8 Hz, 0.3 H) and δ 3.33 ppm (s, 0.5 H), even in the spectrum of a sample of known analytical purity. These may be related to the observed solution instability of this mercurial; a $CDCl_3$ solution had decomposed to a significant extent 30 min after its preparation.

In separate experiments in which the inverse addition procedure (organozinc solution to $HgCl_2$) was employed very similar results were obtained. However, in these experiments the transient formation of molecular iodine could be clearly observed accompanying the reduction to metallic mercury.

Reaction of $Me_3SnCHIZnI$ with trimethyltin chloride

To the deep yellow-orange solution obtained by equilibration of 22.0 mmole of EtZnI and 19.8 mmole of Me_3SnCHI_2[6] in THF under nitrogen at 40° for 60 min was added 17.7 mmole of Me_3SnCl in 9 ml of THF. The resulting mixture, virtually colorless after 60 min, was stirred for $2\frac{1}{2}$ h at 40°.

GLC analysis (column A[1], 160°) of the trap-to-trap distillate obtained in the usual manner showed the presence of 14.6 mmole (83 %) of $(Me_3Sn)_2CHI$. Short-path distillation gave 6.07 g (74 %) of product, b.p. 59–60° (0.18 mm), better than 98 % pure by GLC. A center cut, b.p. 59.5–61° (0.18 mm), had n_D^{25} 1.5682. NMR (in CCl_4): δ 0.23 [s, 18 H, $J(^{117}Sn-^{1}H)$ 51 Hz, $J(^{119}Sn-^{1}H)$ 54 Hz, Me_3Sn] and 1.73 ppm [s, 1 H $J(Sn-H)$ 36 Hz, CHI]. (Found: C, 18.04; H, 4.16; I, 26.96. $C_7H_{19}ISn_2$ calcd.: C, 17.98; H, 4.10; I, 27.14 %.)

Reaction of $(Me_3SiCHI)_2Hg/Ph_2Hg$ with olefins

(*a*). *With cyclohexene.* A mixture of 3.15 g (5.02 mmole) of $(Me_3SiCHI)_2Hg$, 1.79 g (5.05 mmole) of diphenylmercury and 50 ml of cyclohexene was sealed under nitrogen in a heavy walled Pyrex tube and heated at 148° for 7 days with occasional shaking. The resulting reaction mixture was a clear, colorless solution which contained a shiny, platelike precipitate, characteristic of the crystalline habit of phenylmercuric iodide. Filtration gave 2.03 g of solid, m.p. 274–277°. The filtrate was trap-to-trap distilled at 0.08 mm (pot temperature to 50°), leaving essentially no residue. Concentration of the distillate using an 8 inch Vigreux column removed much of the cyclohexene. The remaining liquid was analyzed by GLC (F & M 700, 6 ft. 20 % UC-W98 silicone oil, 120°) and samples of the components were isolated by GLC. These were identified as *trans*-1,2-bis(trimethylsilyl)ethylene (29 % yield, based on Me_3Si groups) and *anti*-7-(trimethylsilyl)norcarane (10 % yield) by comparison of their GLC retention times, IR and NMR spectra with those of authentic samples available from another study[14].

(*b*). *With cyclooctene.* A flame-dried 100 ml one-necked flask equipped with a reflux condenser, thermometer and magnetic stirring assembly was charged under nitrogen with 1.77 mmole of $(Me_3SiCHI)_2Hg$, 1.80 mmole of diphenylmercury and 20 ml of dry cyclooctene. The solution was stirred at reflux (ca. 140°) for 9 days, until the Me_3Si resonance at 0.3 ppm due to the starting mercurial no longer was observed in the NMR spectrum of an aliquot of the reaction mixture. Further work-up of the reaction mixture followed the procedure in (*a*) above. GLC analysis (as above, at 160°) established the presence of *anti*-9-(trimethylsilyl)bicyclo[6.1.0]nonane in 10 % yield. The large amount of cyclooctene present made detection of *trans*-1,2-bis(trimethylsilyl)ethylene impossible.

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