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# NEW DEOXYGENATIVE TRANSFORMATIONS; REACTIONS OF $(CO)_4Fe[Si(CH_3)_3]_2$ WITH ACYCLIC ETHERS

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## Summary

The reaction of *cis*-$(CO)_4Fe[Si(CH_3)_3]_2$ (I) with $CH_3OSi(CH_3)_3$ and $C_6H_5CH_2OSi(CH_3)_3$ at 80°C affords good yields of $[(CH_3)_3Si]_2O$ and the deoxygenation products $RSi(CH_3)_3$ ($R = CH_3$, $C_6H_5CH_2$). These reactions are proposed to occur via $(CO)_4Fe(R)Si(CH_3)_3$ intermediates. This is supported by the observed formation of *cis*-$(CO)_4Fe(CH_3)Si(CH_3)_3$ (II) during the more rapid reaction of I with $(CH_3)_2O$; subsequent $(CH_3)_4Si$ elimination occurs. With $(C_6H_5CH_2)_2O$, I reacts at 80°C to yield $C_6H_5CH_2Si(CH_3)_3$ and $C_6H_5CH_2OSi(CH_3)_3$ as primary products. With $C_6H_5CH_2OCH_3$, I effects regioselective benzyl—oxygen bond cleavage.

## Introduction

Our interest in developing new methods for metal—carbon bond formation has prompted us to undertake a systematic study of the reactions of transition metal trialkylsilanes with organic compounds [1—4]. Since main group element $(CH_3)_3Si—X$ compounds have been shown to enter into numerous carbon—X bond forming reactions with oxygen-containing organic compounds that their H—X counterparts do not [5—8], transition metal trimethylsilanes would appear to have untapped potential for organometallic synthesis. In this paper, we report that the iron silane *cis*-$(CO)_4Fe[Si(CH_3)_3]_2$ (I) [9] reacts with a variety of acyclic ethers to form labile organometallic intermediates. These intermediates subsequently decompose to deoxygenated organic molecules, effecting an overall transformation with scant precedent in organic synthesis.

## Results

A toluene solution which was 0.40 *M* in $(CO)_4Fe[Si(CH_3)_3]_2$ (I) and $CH_3OSi(CH_3)_3$ was heated at 80 ± 2°C for 24 h. Subsequent GLC analysis indicated the presence of some unreacted $CH_3OSi(CH_3)_3$ (22%), and products $(CH_3)_4Si$ (52%, or 67% based upon unconsumed $CH_3OSi(CH_3)_3$) and $[(CH_3)_3Si]_2O$ (83%), as shown in eq. 1. These yields (as well as all others in this paper) represent mmol product/mmol reactant, and are judged to be accurate within 5%. When eq. 1 was $^1H$ NMR monitored in $C_6D_6$, <5% reaction occurred over the course of 18 h at 4°C.

$$(CO)_4Fe[Si(CH_3)_3]_2 + CH_3OSi(CH_3)_3 \xrightarrow[24\ h]{80^\circ C} (CH_3)_4Si + [(CH_3)_3Si]_2O \qquad (1)$$

(I) 52%
(67% conversion yield)

An identical experiment was conducted using $C_6H_5CH_2OSi(CH_3)_3$ instead of $CH_3OSi(CH_3)_3$ (eq. 2). After 24 h at 80°C, GLC analysis indicated a 74% yield of $C_6H_5CH_2Si(CH_3)_3$; some $C_6H_5CH_2OSi(CH_3)_3$ was recovered (2%), and $[(CH_3)_3Si]_2O$ also formed (117%). In a second, similar experiment, the $C_6H_5CH_2Si(CH_3)_3$ produced was isolated in 44% yield by preparative TLC.

$$(CO)_4Fe[Si(CH_3)_3]_2 + C_6H_5CH_2OSi(CH_3)_3 \xrightarrow[24\ h]{80^\circ C} C_6H_5CH_2Si(CH_3)_3 + [(CH_3)_3Si]_2O$$

(I) 74% (2)

To gain insight into the above transformations, I was treated with a more reactive substrate, dimethyl ether. When approximately equimolar quantities of I and $(CH_3)_2O$ were treated at 5°C in toluene-$d_8$, numerous species were detectable by $^1H$ NMR. Figure 1 shows a series of spectra recorded over the course of 7 h. As illustrated, new resonances indicated the presence of $CH_3OSi(CH_3)_3$ ($\delta$ 3.26, 0.05), $(CH_3)_4Si$ ($\delta$ 0.00) and $[(CH_3)_3Si]_2O$ ($\delta$ 0.08); these assignments were confirmed by GLC. When a similar reaction was allowed to proceed for 4 days at 2°C, a 7/67/57 ratio of products was obtained (eq. 3).

$$(CO)_4Fe[Si(CH_3)_3]_2 + CH_3OCH_3 \xrightarrow[4\ days]{2^\circ C} CH_3OSi(CH_3)_3 + (CH_3)_4Si + [(CH_3)_3Si]_2O$$

(I) 7% 67% 57% (3)

Another new $^1H$ NMR resonance which appeared early in the reaction of I with $(CH_3)_2O$ ($\delta$ 0.37, Fig. 1) was nearly coincident with the $\delta$ 0.34 absorption previously reported for the $Si(CH_3)_3$ protons of *cis*-$(CO)_4Fe(CH_3)Si(CH_3)_3$ (II) [3]. The $FeCH_3$ resonance of this compound is degenerate with the trimethylsilyl resonance of $CH_3OSi(CH_3)_3$. In a separate experiment using a 1/5 molar ratio of I/$(CH_3)_2O$, volatiles were removed from the reaction after 5 h at 3°C. Petroleum ether extraction, filtration, and solvent removal gave an oil (43% yield vs. hexamethylbenzene NMR standard) whose $^1H$ NMR and IR spectra agreed with those previously reported for II [3]. Furthermore, a $^{13}C$ NMR spectrum of a similar reaction after 4.5 h at 3°C (210.4, 206.5, 204.9, 5.5, −12.7 ppm) [3] indicated II to be the major iron carbonyl compound present.

As expected from earlier work [3], continued standing of any of the above solutions of II resulted in its disappearance and the formation of (additional) $(CH_3)_4Si$. Of relevance to a future mechanistic point, II did not react with either $(CH_3)_2O$ or $CH_3OSi(CH_3)_3$ at a rate faster than its decomposition.

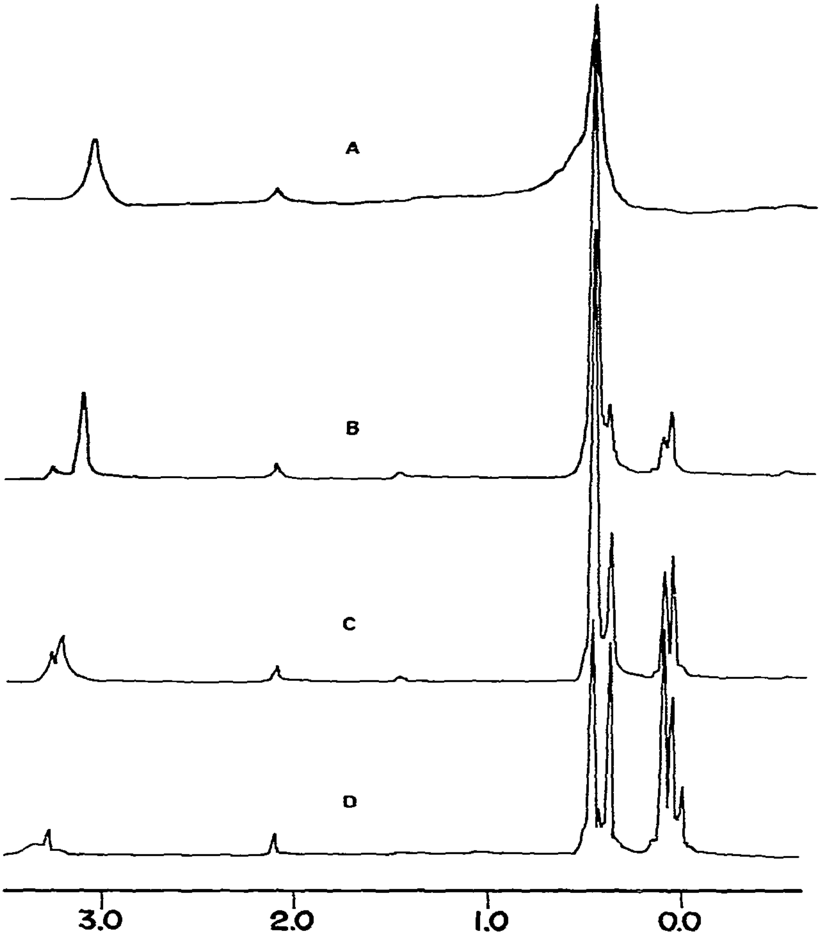


Fig. 1. $^1$H NMR spectra of the initial stages of the reaction of $(CO)_4Fe[Si(CH_3)_3]_2$ with $(CH_3)_2O$: (A) immediately after mixing at $-12°C$; (B) after warming to 5°C; (C) after 2 h at 5°C; (D) after 7 h at 5°C. Peak assignments: $(CH_3)_2O$ appears at $\delta$ 3.05 in A and broadens and moves downfield to $\delta$ 3.33 in D; $\delta$ 2.09 (A—D): hexamethylbenzene standard; $\delta$ 0.45 (A—D): I; $\delta$ 3.26 (B—D): $C\mathit{H}_3OSi(CH_3)_3$; $\delta$ 1.46, $-0.57$ (B, C): side bands; $\delta$ 0.37 (B—D): $(CO)_4Fe(CH_3)Si(C\mathit{H}_3)_3$ (II); $\delta$ 0.08 (B—D): $[(CH_3)_3Si]_2O$; $\delta$ 0.05 (B—D): $CH_3OSi(C\mathit{H}_3)_3$ and $(CO)_4Fe(C\mathit{H}_3)Si(CH_3)_3$; $\delta$ 0.00 (D): $(CH_3)_4Si$. Paramagnetic impurities/by-products impeded the $^1$H NMR monitoring of all reactions in this paper; the spectra in Fig. 1 were the best we could obtain from the reaction of I with $(CH_3)_2O$.

Other aliphatic ethers were treated with I. A toluene solution which was 0.40 *M* in $(C_6H_5CH_2)_2O$ and I was heated at 80 ± 2°C for 24 h. Subsequent GLC analysis indicated the presence of some unreacted $(C_6H_5CH_2)_2O$ (19%) and the products given in eq. 4. When a similar solution of I and $(C_6H_5CH_2)_2O$ was maintained at room temperature, less than 5% reaction occurred over 40 h.

$$\underset{\text{(I)}}{(CO)_4Fe[Si(CH_3)_3]_2} + C_6H_5CH_2OCH_2C_6H_5 \xrightarrow[24\ h]{80°C}$$

$$\underset{89\%}{C_6H_5CH_2Si(CH_3)_3} + \underset{57\%}{C_6H_5CH_2OSi(CH_3)_3} + \underset{19\%}{[(CH_3)_3Si]_2O} \quad (4)$$

A toluene solution (0.40 *M*) of the unsymmetrical ether $C_6H_5CH_2OCH_3$ was treated with I (1 equiv.) at 80 ± 2°C for 24 h. Subsequent GLC analysis indicated the presence of some unreacted $C_6H_5CH_2OCH_3$ (8%) and the products given in eq. 5.

$$\underset{(\mathrm{I})}{(CO)_4Fe[Si(CH_3)_3]_2} + C_6H_5CH_2OCH_3 \xrightarrow[24\ h]{80^\circ C} \qquad (5)$$

$$\underset{87\%}{C_6H_5CH_2Si(CH_3)_3} + \underset{12\%}{(CH_3)_4Si} + \underset{65\%}{CH_3OSi(CH_3)_3} + \underset{1\%}{C_6H_5CH_2OSi(CH_3)_3} + \underset{21\%}{[(CH_3)_3Si]_2O}$$

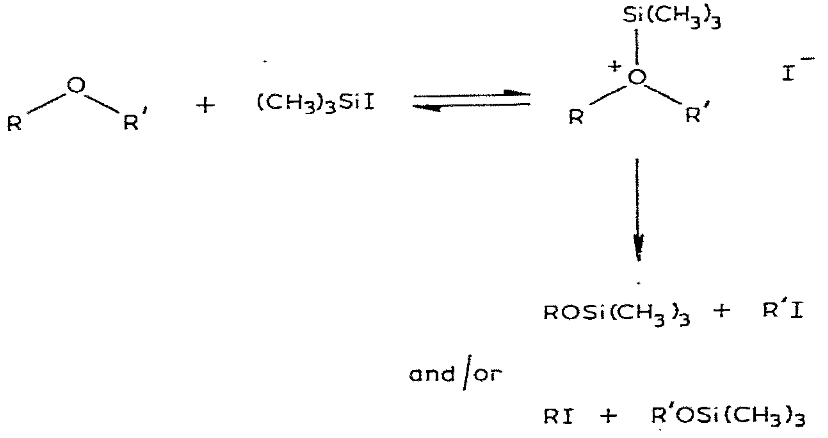
Unfortunately, ethers which contained C—H bonds $\beta$ to the ether oxygen did not afford volatile deoxygenated products when treated with I. Specifically, no $RSi(CH_3)_3$ species were formed from $n\text{-}C_{12}H_{25}OSi(CH_3)_3$, $i\text{-}C_3H_7OSi(CH_3)_3$, $C_2H_5OSi(CH_3)_3$, and $H_2C{=}CHCH_2OSi(CH_3)_3$. In these and all previous reactions, iron carbonyl products ($Fe(CO)_5$, $Fe_3(CO)_{12}$) formed, but were not quantified.

## Discussion

*Mechanistic details.* In order to formulate viable mechanisms for the preceding transformations, comparisons will be made with (1) previously observed reactions of ethers with $(CH_3)_3SiI$ [6b], and (2) reactions of transition metal trimethylsilanes with more basic heteronucleophiles.

Numerous aliphatic ethers undergo ready cleavage when treated with $(CH_3)_3SiI$ in polar solvents at room temperature. These reactions have been suggested by Jung and Lyster to proceed via intermediate oxonium salt ion pairs, as shown in Scheme 1 [6b]. Subsequent carbon—oxygen bond cleavage to alkyl iodide and trimethylsilyl ether products has been shown to occur by both $S_N2$ and $S_N1$-type mechanisms [6b].

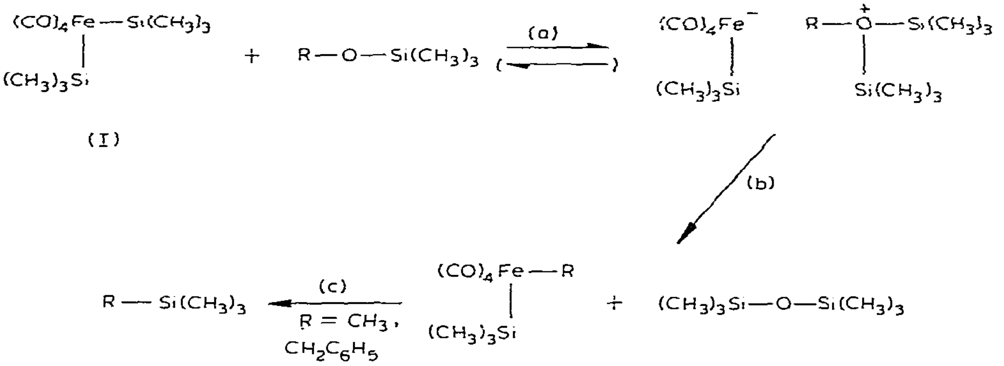
SCHEME 1. Mechanism proposed for ether cleavage by $(CH_3)_3SiI$ [6b].



Isolable ion pairs have been previously observed to form from transition metal trimethylsilanes. For instance, Schäfer and MacDiarmid have reported that $(CH_3)_3P$ and $(CO)_4CoSi(CH_3)_3$ react to yield $(CH_3)_3P^+Si(CH_3)_3(CO)_4Co^-$ [10]. Similarly, $(CH_3)_3N$ and $(CO)_5MnSi(CH_3)_3$ yield the ion pair $(CH_3)_3N^+Si(CH_3)_3$-$(CO)_5Mn^-$ [11].

In light of these precedents, a plausible postulate for the first step of eq. 1 and 2 is given in step a of Scheme 2. The anionic component of the initially formed ion pair, $(CO)_4FeSi(CH_3)_3^-$, should be reasonably nucleophilic [12,13] and capable of attacking the alkyl group of the oxonium cation (step b). This would generate the observed product $[(CH_3)_3Si]_2O$. We recently described an independent synthesis of $M^+(CO)_4FeSi(CH_3)_3^-$ ($M^+ = K^+$, $[(C_6H_5)_3P]_2N^+$) [3], and reported its facile alkylation by electrophiles such as $CH_3SO_3F$ and $C_6H_5$-$CH_2Br$. The resulting *cis*-$(CO)_4Fe(R)Si(CH_3)_3$-alkyls underwent reductive elimination to $RSi(CH_3)_3$ at room temperature (step c), so organometallic intermediates would not be expected to build up under the conditions required for eq. 1 and 2 (80°C).

SCHEME 2: Proposed mechanism for the reaction of I with alkyl trimethylsilyl ethers.

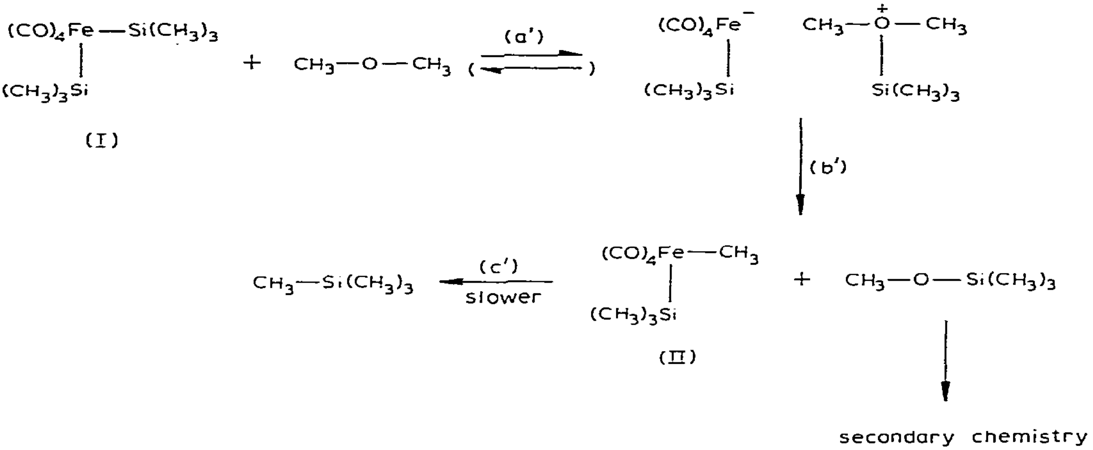


Although Scheme 2 is well supported by both direct and indirect evidence, we sought conditions where additional reaction intermediates would be observable. Accordingly, the organometallic intermediate II was detected in the reaction of I with $(CH_3)_2O$. Our results suggest a mechanism similar to Scheme 2, but with steps a and b faster relative to step c. This is summarized in Scheme 3.

Our data clearly indicate that there is a complicating secondary process occurring in the reaction of I with $(CH_3)_2O$ which consumes the $CH_3OSi$-$(CH_3)_3$ expected to form equimolar with $(CH_3)_4Si$ (Scheme 3). It should be emphasized that control experiments show that the intermediate II (step b′) does not react with any of the organic species known to be present, and that I reacts with $CH_3OSi(CH_3)_3$ only at much higher temperatures. Our best guess is that the oxonium cation $(CH_3)_2O^+Si(CH_3)_3$ might react with $CH_3OSi(CH_3)_3$ as the concentration of the latter builds up. For instance, oxonium salts have been observed to transfer alkyl groups to ethers [14]. Similar trimethylsilyl group transfer could provide a disproportionation pathway, or a lower temperature means of generating the ion pair obtained in step a of Scheme 2.

The reaction of I with $(C_6H_5CH_2)_2O$ (eq. 4) requires higher temperatures and gives more readily interpreted data. The formation of most of the $C_6H_5CH_2Si$-

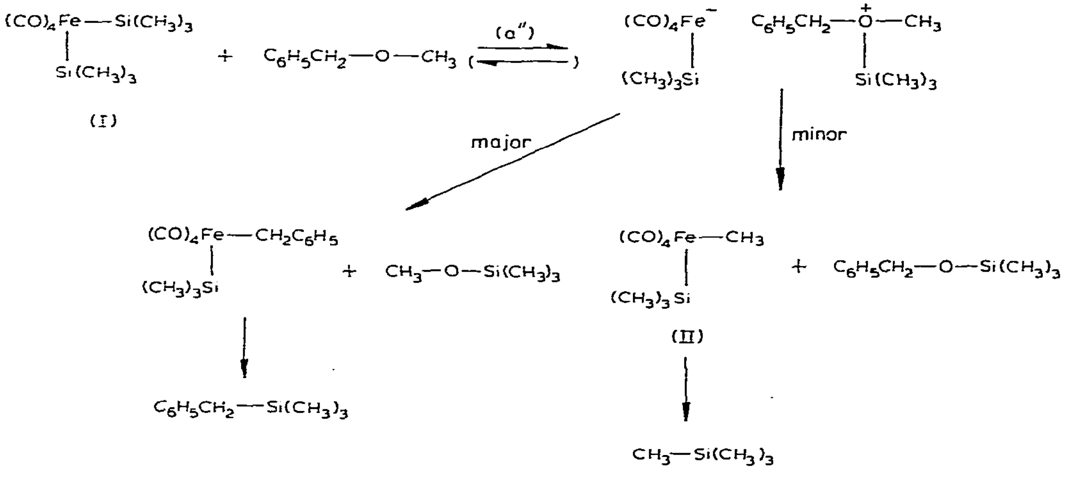
SCHEME 3. Proposed mechanism for the reaction of I with $(CH_3)_2O$.



$(CH_3)_3$ would occur along the lines of steps a′, b′, and c′ of Scheme 3. As the concentration of the by-product $C_6H_5CH_2OSi(CH_3)_3$ builds up, it would be expected to compete effectively at 80°C for unreacted I (eq. 2). This would result in some Scheme 2 chemistry occurring, which would decrease the amount of $C_6H_5CH_2OSi(CH_3)_3$ present, and provide a pathway for $[(CH_3)_3Si]_2O$ formation. This interpretation also predicts the observed presence (19%) of unreacted starting material when equimolar quantities of reactants are employed.

The reaction of I with $C_6H_5CH_2OCH_3$ shows some interesting regioselectivity, as summarized in Scheme 4. Two different alkyl transfer reactions are available to the ion pair generated in step a″, and the products (eq. 5) require a preference (ca. 12/1) for benzyl—oxygen bond cleavage. Under the reaction conditions, the $ROSi(CH_3)_3$ products again appear to undergo some secondary (Scheme 2-type) reactions. Since $(CH_3)_2O$ reacted more rapidly than $(C_6H_5CH_2)_2O$ with I, this regioselectivity suggests that step a′ (or a″) in these reactions is rate determining.

SCHEME 4. Proposed origin of regioselectivity in the reaction of I with $C_6H_5CH_2OCH_3$.



We previously noted that *cis*-$(CO)_4Fe(R)Si(CH_3)_3$ alkyls with $\beta$-CH bonds (or where R = allyl) failed to reductively eliminate $RSi(CH_3)_3$ products [3]. Therefore the lack of such products from the reaction of I with such compounds as i-$C_3H_7OSi(CH_3)_3$ and $C_2H_5OSi(CH_3)_3$ is still consistent with initial reaction via steps a and b of Scheme 2.

*Overview.* Although the organometallic intermediates generated during the course of eq. 1—5 undergo rapid decomposition under the reaction conditions, this work documents (for the first time we believe) that transition metal—carbon bonded species can be made generally available from simple aliphatic alkyl trimethylsilyl ethers. Undoubtedly the oxygenophilic $Si(CH_3)_3$ group plays a key role in the success of these reactions. Current estimates of silicon—oxygen bond dissociation energies are in the 106—112 kcal/mol range [15]. The carbon—oxygen bond dissociation energy in $(CH_3)_2O$ is 82 kcal/mol [16]. The first two steps of Schemes 2—4 also entail metal—silicon bond rupture and metal—carbon bond formation. While precise bond dissociation energies are not available for these molecules, the iron—silicon bond in $(\eta\text{-}C_5H_5)(CO)_2FeSi(CH_3)_2CH_2Br$ has been shown to be no more than 5—7 kcal/mol stronger than the iron—carbon bond in $(\eta\text{-}C_5H_5)(CO)_2FeCH_2Si(CH_3)_2Br$ [17]. Thus it is reasonable to expect the bond metathesis $Fe—Si + C—O \rightarrow Fe—C + Si—O$ to be exothermic by at least 20 kcal/mol. Such thermochemical considerations appear to be underutilized in designing new metal—carbon bond forming reactions.

While the organic transformations $ROSi(CH_3)_3 \rightarrow RSi(CH_3)_3$ accomplished in eq. 1 and 2 is of limited generality, it is worth noting that there is little, if any, precedent for such a conversion. The reductive silylation of $ROSiR'_3$ species with $Mg/(CH_3)_3SiCl$ has been observed, but only in those cases where R is benzylic or allylic and can stabilize a radical center [18]. Furthermore, transition metal catalysts and HMPA were generally required.

One of the principal values of this type of study is in establishing reactivity modes. For instance, by utilizing the monofunctional (but less reactive) silylating agent $(CO)_5MnSi(CH_3)_3$, we have been able to develop useful ring-opening reactions of strained cyclic ethers; when conducted under CO, high yields of organometallic $(CO)_5MnCO(CH_2)_nOSi(CH_3)_3$ ($n$ = 2—4) products are obtained [4].

In conclusion, numerous conceptual links are being established between the chemistry of main group-$Si(CH_3)_3$ compounds (i.e., $(CH_3)_3SiI$, $(CH_3)_3SiCN$) and transition metal-$Si(CH_3)_3$ compounds. By the combination of these with the unique reactions available to transition metal—carbon bonds, important new developments in organic and organometallic synthesis can be expected.

## Experimental

### *General procedures*

All reactions were conducted in 5 mm NMR tubes with the rigorous exclusion of air and water. Benzene and toluene were distilled from Na/benzophenone. Petroleum ether and heptane were distilled from $LiAlH_4$ and sodium, respectively, and then freeze-pump-thaw degassed.

$CH_3OSi(CH_3)_3$, $(C_6H_5CH_2)_2O$, and $C_6H_5CH_2OCH_3$ were obtained from Petrarch, MCB, and CPL Inc., respectively. $C_6H_5CH_2OSi(CH_3)_3$ was prepared from

$C_6H_5CH_2OH$ by the method of Jung [19]. $CH_3OSi(CH_3)_3$ was purified by simple distillation; other substrates were stirred overnight with sodium and then distilled. All were subjected to freeze-thaw-pump degassing after distillation. $(CH_3)_2O$ was obtained from Matheson and was used without purification. Authentic $C_6H_5CH_2Si(CH_3)_3$ was purchased from Petrarch.

Although I can be synthesized from $(CO)_5Fe$ and $Hg[Si(CH_3)_3]_2$ [9], an improved synthesis will be described shortly [20]. Samples of II used in control experiments were prepared as recently described [3].

$^1H$ NMR spectra were obtained on Varian A60 and T60 Spectrometers. $^{13}C$ NMR spectra (50 MHz) were obtained on a Bruker WP-200 Spectrometer. GLC analyses were conducted on a Hewlett—Packard 5720A Gas Chromatograph equipped with either a 5′ or 10′ DC 710 (10% on Chromosorb W) column. Yields were calculated using an electronic integrator and corrected for detector response factor. All product identities were confirmed by coinjection with authentic samples.

### *Reaction of I with $CH_3OSi(CH_3)_3$, $C_6H_5CH_2OSi(CH_3)_3$, $(C_6H_5CH_2)_2O$, and $C_6H_5CH_2OCH_3$*

The ether (0.16 mmol) and I (0.16 mmol; 50—51 mg) were dissolved in 0.40 ml of toluene-$d_8$ in a $^1H$ NMR tube. Heptane was added (0.025 ml), and the tube was tightly sealed by wiring on a latex septum. Samples were then heated at 80 ± 2°C for 24 h, and then cooled overnight before GLC analysis. Quantities of the ethers employed were as follows: $CH_3OSi(CH_3)_3$, 17 mg; $C_6H_5CH_2OSi(CH_3)_3$, 29 mg; $(C_6H_5CH_2)_2O$, 32 mg; $C_6H_5CH_2OCH_3$, 20 mg. Yield data: text.

In a separate experiment, 100 mg (0.32 mmol) of I and 57 mg (0.31 mmol) of $C_6H_5CH_2OSi(CH_3)_3$ were dissolved in 0.40 ml of toluene-$d_8$. After 48 h at 90°C, the solution was applied directly to a preparative TLC plate and chromatographed with petroleum ether (b.p. 20—40°C). Extraction of a fast moving band (visualized by UV) afforded 23 mg (0.14 mmol, 44%) of $C_6H_5CH_2Si(CH_3)_3$.

### *Reactions of I with $(CH_3)_2O$*

Standard solutions of $(CH_3)_2O$: Toluene-$d_8$ or $C_6D_6$ was placed in a $^1H$ NMR tube, capped with a septum, and cooled in an ice bath. After saturation with $(CH_3)_2O$, the solution was warmed to room temperature and (if desired) diluted. Immediately before use, *p*-di-t-butylbenzene or hexamethylbenzene standard was added and the $(CH_3)_2O$ concentration determined by $^1H$ NMR integration.

Toluene-$d_8$ (0.40 ml) containing 0.22 mmol of $(CH_3)_2O$ was added to a 5 mm NMR tube and cooled to 0°C. Using a gas tight syringe, I (70 mg, 0.22 mmol) and heptane (17.3 mg) in 0.30 ml of toluene-$d_8$ were injected. The solution was allowed to stand at 2°C for 4 days, and was then analyzed by GLC. Yield data: see text. The NMR spectra shown in Fig. 1 were obtained from a similar experiment utilizing 2.2 mg of hexamethylbenzene and 0.48 mmol of $(CH_3)_2O$ in 0.40 ml of toluene-$d_8$ and 150 mg (0.48 mmol) I in 0.40 ml of toluene-$d_8$.

In order to obtain more definitive evidence on the intermediacy of II, 132 mg (0.42 mmol) of I in 0.50 ml of $C_6D_6$ was added to 0.80 ml of $C_6D_6$ containing 2.09 mmol of $(CH_3)_2O$ and 12.7 mg *p*-di-t-butylbenzene at 0°C. The solution was kept at 3°C for 5 h; volatiles were then removed under vacuum. The residue was washed with petroleum ether and filtered. The solvent was removed and the residue analyzed. An IR spectrum (hexane) showed absorptions at 2088m, 2026s and 2001vs $cm^{-1}$ (lit. [3] 2088m, 2027s, 2000vs) characteristic of II. A $^1H$ NMR spectrum (after the addition of 5.3 mg of hexamethylbenzene internal standard) showed II to be the major $Si(CH_3)_3$ containing species present (43% yield).

In a similarly directed experiment, 0.80 ml of toluene-$d_8$ containing 1.13 mmol of $(CH_3)_2O$ and 4.5 mg *p*-di-t-butylbenzene was added to a 5 mm NMR tube and cooled to 0°C. Using a gas-tight syringe, 75 mg (0.24 mmol) of I in 0.40 ml of toluene-$d_8$ was added. After 4.5 h at 3°C, the solution was washed into a 10 mm NMR tube containing 9 mg of $Cr(acac)_3$. $^{13}C$ NMR: see text.

### *Control experiments*

To 0.40 ml of $C_6D_6$ were added I (100 mg, 0.32 mmol), $CH_3OSi(CH_3)_3$ (53 $\mu$l, 40 mg, 0.38 mmol), and hexamethylbenzene (3.2 mg). The solution was cooled to 4°C and monitored by periodic $^1H$ NMR spectra at ambient probe temperature. After 18 h, <5% reaction had occurred.

To 0.14 mmol of II in 0.40 ml of $C_6D_6$ (containing 3.5 mg of hexamethylbenzene) was added 18.5 mg (0.18 mmol) of $CH_3OSi(CH_3)_3$. The solution was quickly cooled to 3°C. After 22 h, only $CH_3OSi(CH_3)_3$, II, $(CH_3)_4Si$, and hexamethylbenzene were detectable by $^1H$ NMR.

To 0.06 mmol of II in 0.40 ml of $C_6D_6$ (containing 3.8 mg of hexamethylbenzene) was added 0.21 ml of $C_6D_6$ containing 0.06 mmol of $(CH_3)_2O$. The solution was quickly cooled to 3°C. After 24 h, only $(CH_3)_2O$ (II), hexamethylbenzene, and $(CH_3)_4Si$ were detectable by $^1H$ NMR. GLC analysis indicated only traces of $CH_3OSi(CH_3)$ and $[(CH_3)_3Si]_2O$.

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