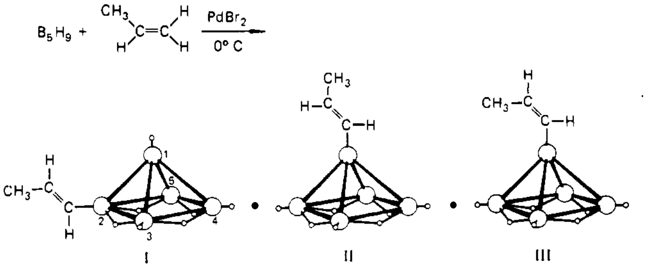

$PdBr_2$. Also isolated in smaller quantities were 2-propylpentaborane (4.7%) and 1-propylpentaborane (2.6%). No hydrogen was produced in the reaction, but GC analysis[9] of the higher volatile materials from the reaction revealed that 10.7 mmol of propylene had been consumed and that 5.5 mmol of propane had been produced. Also produced in the reaction was a small amount of lower volatile material which has not yet been characterized.

$PdBr_2$ was also found to catalyze the reactions of ethylene and 1-butene with pentaborane(9) under mild conditions to give good yields of alkenylpentaboranes: ethylene, 10 h, 0 °C, 43% 2-vinylpentaborane,[4] 13% 2-ethylpentaborane,[10] 2.3 equiv of vinylpentaboranes/$PdBr_2$; 1-butene, 8 h, 0 °C, 2-(*trans*-1-but-1-enyl)pentaborane[11] (50.4%),[12] 1-(*trans*-1-but-1-enyl)pentaborane[13] (26.9%), 2-(*trans*-1-but-2-enyl)pentaborane[14] (21.7%), 2-butylpentaborane (1%), 5.1 equiv of butenylpentaboranes/$PdBr_2$.

The procedures outlined above provide a number of significant improvements over our previously reported metal-catalyzed syntheses of alkenylboranes, in terms of higher yields, avoidance of side reactions, and ease of product separation and purification. Although the reaction gives a mixture of 1-alkenyl- and 2-alkenyl-substituted pentaboranes, we have now shown[15] that pyrolysis of the unseparated reaction products, using conditions similar to those previously described,[3,4] gives monocarbon carborane products based on the $2\text{-}CB_5H_9$ cage system in high yields. Thus, separation of the alkenylpentaborane isomers before the conversion to monocarbon carboranes is not necessary.

Palladium salts have of course been used as stoichiometric reagents for arene–olefin coupling reactions[16] which can be made pseudocatalytic in the presence of a suitable oxidant, such as oxygen or cupric chloride. The reactions described above with pentaborane(9) were found to be *catalytic* in the absence of an additional oxidant and are to our knowledge the first examples of transition-metal-catalyzed olefin substitution reactions. The mechanism of this reaction is at present unknown; however, the formation of nearly equivalent amounts of alkenylpentaboranes and propane in the propylene reaction suggests that the hydrogenation of a second equivalent of olefin is an important step in the catalytic cycle. In addition, the formation of both 1-alkenyl- and 2-alkenyl-substituted pentaboranes suggest the possibility that two different reaction pathways, involving either oxidative addition of a basal B–H unit (2-position) of pentaborane(9) to the palladium or electrophilic attack of the metal reagent at the apex boron (1-position) in pentaborane(9), may be important in the reaction. Indeed, we have previously proposed[1] similar types of reactivities to account for the platinum bromide catalyzed dehydrodimerization of pentaborane(9) which leads to the selective formation of the boron–boron coupled compound $1{:}2'\text{-}[B_5H_8]_2$. Further studies are now in progress aimed at elucidating the mechanism(s) of these unique reactions.

In conclusion, we feel it is significant that alkenylboranes can now be prepared from olefins, rather than more difficult to handle acetylenes, and that these reactions can be carried out by using mild, low-temperature conditions. Furthermore, these palladium-promoted reactions appear to have great potential for the large scale syntheses of alkenylboranes and -carboranes and we are presently investigating the production of a variety of such species using these procedures.

Acknowledgment. We thank the Army Research Office and the National Science Foundation for support of this research.

Registry No. I, 78837-93-1; II, 87261-36-7; III, 87261-37-8; $PdBr_2$, 13444-94-5; pentaborane(9), 19624-22-7; propylene, 115-07-1; 2-vinylpentaborane, 78837-91-9; 2-ethylpentaborane, 23753-62-0; 2-(*trans*-1-but-1-enyl)pentaborane, 87281-27-4; 1-(*trans*-1-but-1-enyl)pentaborane, 87308-13-2; 2-(*trans*-1-but-2-enyl)pentaborane, 87281-28-5; ethylene, 74-85-1; 1-butene, 106-98-9.

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[9] Durapak phenyl isocyanate/Poracil C, 80–100 mesh, ambient temperature.

[10] Onak, T.; Dunks, G. B.; Searcy, I. W.; Spielman, J. *Inorg. Chem.* **1967**, *6*, 1465.

[11] ^{11}B NMR (ppm, C_5H_{12}, C_6D_6): 1.2 (s, B_2), −13.5 (d, 2, $B_{3,5}$, J_{B-H} = 153 Hz), −18.2 (d, B_4, J_{B-H} = 161 Hz), −51.6 (d, B_1, J_{B-H} = 165 Hz). ^1H NMR (δ, C_6D_6, ^{11}B spin decoupled) 6.00 (m, C–H), 5.40 (d of m, C–H, J = 17.5 Hz), 2.50 (br q, 3, B_{3-5}–H), 1.90 (m, 2, CH_2), 0.80 (t, 3, CH_3), −1.50 (br s, BHB), −2.60 (br s, BHB).

[12] Only relative yields are given for the 1-butene reaction owing to the difficulty in quantitatively separating 1-butene from pentaborane(9).

[13] ^{11}B NMR (ppm, C_5H_{12}, C_6D_6): −12.8 (d, 4, B_{2-5}, J_{B-H} = 166 Hz, $J_{B_{apex}-B_{base}}$ = 20 Hz), −45.2 (s, B_1). ^1H NMR (δ, C_6D_6, ^{11}B spin decoupled): 6.20 (m, C–H), 5.84 (d of m, C–H, J = 19.5 Hz), 2.40 (br q, 4, B_{2-5}–H), 2.10 (m, 2, CH_2), 1.00 (t, 3, CH_3), −2.40 (br s, BHB).

[14] ^{11}B NMR (ppm, C_5H_{12}, C_6D_6): 2.5 (s, B_2), −13.5 (d, 2, $B_{3,5}$, J_{B-H} = 153 Hz), −18.2 (d, B_4, J_{B-H} = 161 Hz), −51.6 (d, B_1, J_{B-H} = 165 Hz). ^1H NMR (δ, C_6D_6, ^{11}B spin decoupled): 5.40 (m, 2, C–H), 2.40 (br q, 3, B_{3-5}–H), 1.60 (d, 3, CH_3), 1.10 (m, 2, CH_2), −2.10 (br s, BHB), −2.80 (br s, BHB).

[15] Davan, T.; Corcoran, E. W., Jr.; Sneddon, L. G., to be submitted for publication.

[16] (a) Heck, R. F. *J. Am. Chem. Soc.* **1968**, *90*, 5518. (b) Fujiwara, Y.; Moritani, I.; Danno, S.; Asano, R.; Teranishi, S. *Ibid.* **1969**, *91*, 7166. (c) Shue, R. S. *J. Catal.* **1972**, *26*, 117.

Hydrogen Peroxide Oxidation of the Silicon–Carbon Bond in Organoalkoxysilanes[1]

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Summary: The silicon–carbon bond in organoalkoxysilanes is readily cleaved by 30% H_2O_2, with retention of configuration at carbon, to give the corresponding alcohols. The new methodology is applicable to one-pot synthesis of anti-Markownikoff alcohols from terminal olefins and of ketones from acetylenes via a sequence of hydrosilylation with $HSiMe(OEt)_2$ and oxidation.

Described herein is the first successful result on the oxidative cleavage of the silicon–carbon bond with 30%

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[1] Silafunctional Compounds in Organic Synthesis. 20. For part 19, see: (a) Tamao, K.; Ishida, N.; Kumada, M. *J. Org. Chem.* **1983**, *48*, 2120. For part 18, see: (b) Tamao, K.; Akita, M.; Kumada, M. *J. Organomet. Chem.*, in press. For part 17, see: ref 5. The present series title has been numbered consecutively from the former series "Organofluorosilicates in Organic Synthesis".

Table I. Oxidative Cleavage of Octyl-SiMe(OEt)$_2$ with Various Kinds of Oxidant[a]

$$n\text{-}C_8H_{17}\text{-SiMe(OEt)}_2 \xrightarrow{[O]} n\text{-}C_8H_{17}\text{-OH}$$

entry	oxidant (equiv)	additive (equiv)	solvent	temp, °C (time, h)[e]	yield, %
1	30% H_2O_2 (12)	none	DMF	60 (7)	2[b]
2	30% H_2O_2 (12)	KF (4)	DMF	60 (7)	72
3	30% H_2O_2 (12)	KHF_2 (2)	DMF	60 (7)	82
4	30% H_2O_2 (12)	KHF_2 (2)	MeOH/THF[c]	60 (7)	83
5	30% H_2O_2 (12)	$KHCO_3$ (1)	MeOH/THF[c]	rfx (3)	96
6	30% H_2O_2 (12)	$NaHCO_3$ (1)	MeOH/THF[c]	rfx (3)	95
7	30% H_2O_2 (12)	K_2CO_3 (1)	MeOH/THF[c]	rfx (3)	72
8	30% H_2O_2/Ac_2O (12)[d]	KHF_2 (2)	DMF	rt (4)	87
9	90% H_2O_2 (6)	KHF_2 (2)	DMF	60 (4)	88
10	70% *t*-BuOOH (6)	KHF_2 (2)	DMF	60 (4)	54
11	MCPBA (2.5)	KHF_2 (2)	DMF	rt (2)	91

[a] Carried out on a 1-mmol scale in a given solvent (5 mL). To a mixture of the octylsilane, an additive, and a solvent was added an oxidant at room temperature with stirring. After being stirred under a given reaction condition, the mixture was poured into water and extracted with ether five times. The combined extract was washed with 10% $NaHSO_3$ and saturated $NaHCO_3$, dried over Na_2SO_4, and then analyzed by GLC to determine the yield of octanol. [b] Recovered 67%. [c] A 1:1 (volume) mixture. [d] Acetic anhydride and 30% H_2O_2 (1:1 molar ratio) were successively added. [e] Rfx = reflux and rt = room temperature.

hydrogen peroxide. Thus, we have found that the silicon–carbon bond in organoalkoxysilanes is readily cleaved by H_2O_2 under mild conditions to give the corresponding alcohols (eq 1).

$$\text{R-}si \xrightarrow{30\%\ H_2O_2} \text{R-OH} \qquad (1)$$

$$si = \text{alkoxysilyl group}$$

Table I summarizes the results on the oxidative cleavage of *n*-octylmethyldiethoxysilane to *n*-octanol by using several types of oxidant under various conditions. While almost no reaction occurs with 30% H_2O_2 alone in DMF, addition of KF or KHF_2 dramatically facilitates the oxidative cleavage (entries 1–3).[2] The reaction occurs also in methanol/THF (entry 4). These KHF_2-induced oxidations proceed under a neutral condition.[3] In the MeOH/THF mixed solvent a weakly alkaline condition can also be employed by using $NaHCO_3$ or $KHCO_3$ as an additive (entries 5–7), with no fluoride ions being required. A 1:1 mixture of 30% H_2O_2 and acetic anhydride is more effective (entry 8), where peracetic acid formed in situ might be the real oxidant.[4] The oxidizing ability of 90% H_2O_2 seems to be comparable with that of 30% H_2O_2 (entry 9), and commercial 70% *t*-BuOOH is less active (entry 10). Although MCPBA is yet one of the most powerful oxidants (entry 11),[5] the 30% H_2O_2 oxidation has the advantage of cheapness of the reagent and mildness and cleanness of the reaction.

We have examined the structure–reactivity correlation in the oxidation of a series of *n*-octylalkoxysilanes, $n\text{-}C_8H_{17}\text{–Si(OR)}_n\text{Me}_{3-n}$ (n = 1–3; R = Me, Et, *i*-Pr, and/or *t*-Bu), as model compounds under three typical conditions: neutral condition A (cf. entry 3), acidic condition B (cf. entry 8), and basic condition C (cf. entry 5). Condition A affords satisfactory results (82–100% yield) with all substrates, except for a few cases: octyl–Si(OMe)$_3$ and –Si(OEt)$_3$ give viscous polysiloxanes predominantly and octyl–SiMe$_2$(OBu-*t*) requires the prolonged heating. These problems have been overcome by the acidic condition B. Under both conditions, however, octyl–SiMe(OBu-*t*)$_2$ is extremely reluctant to react for steric reasons. The basic condition C appears to be more sensitive to the steric factor and suitable for octyl–SiMe(OEt)$_2$, –SiMe$_2$(OEt), or –SiMe$_2$(OPr-*i*), but not for octyl–SiMe(OPr-*i*)$_2$ or –SiMe$_2$(OBu-*t*). These results clearly indicate that the H_2O_2 oxidation proceeds smoothly if only the substrate carries at least one alkoxy group on silicon and that the reactivity of the silicon–carbon bond can be controlled by changing the nature of alkoxy group(s).

The H_2O_2 oxidation has been observed to proceed with retention of configuration at carbon to be oxidized, as exemplified by oxidation of *exo*- and *endo*-2-norbornyl–SiMe(OEt)$_2$.[6] Under condition A, the exo isomer (exo 100%) gave *exo*-norbornanol (65% yield, exo ≥98%), while the endo isomer (endo 80%) formed the endo alcohol (60% yield, endo 77%). Thus, the oxygen functionality is introduced stereospecifically, as well as regioselectively, onto the originally silicon-bearing carbon atom.

The present oxidation offers one of the most efficient methods for the cleavage of an "unactivated"[5] ordinary alkyl–silicon bond[7] and is of interest from both mechanistic[8] and synthetic points of view. It should be stressed here that the present new development promises many synthetic possibilities which have not been realized with the more common trimethylsilyl compounds,[11,12] in view

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(2) The reaction conditions have not been optimized, and excess amounts of H_2O_2 (usually 3–6 equiv/one Si–C bond) have been used.

(3) The reaction mixture becomes neutral according to the stoichiometry:

$$\gt\text{Si–OR} + \text{KHF}_2 \rightarrow\ \gt\text{Si–F} + \text{HOR} + \text{KF}$$

(4) (a) Hawthorne, M. F.; Emmons, W. D.; McCullum, K. S. *J. Am. Chem. Soc.* **1958**, *80*, 6393. (b) Greenspan, F. P. *Ibid.* **1946**, *68*, 907.

(5) Tamao, K.; Kakui, T.; Akita, M.; Iwahara, T.; Kanatani, R.; Yoshida, J.; Kumada, M. *Tetrahedron* **1983**, *39*, 983.

(6) The exo and endo isomers were prepared in a similar manner to the corresponding fluorosilicates. Tamao, K.; Yoshida, J.; Yamamoto, H.; Kakui, T.; Matsumoto, H.; Takahashi, M.; Kurita, A.; Murata, M.; Kumada, M. *Organometallics* **1982**, *1*, 355.

(7) Eaborn, C.; Bott, R. W. In "Organometallic Compounds of the Group IV Elements"; MacDiarmid, A. G., Ed.; Marcel Dekker: New York, 1968; Vol. 1.

(8) Although the mechanism has not been clarified yet, the most plausible one consistent with the experimental data is as follows. The alkoxy group on silicon may be replaced by H_2O_2 to give a silyl hydroperoxide species that undergoes intramolecular rearrangement to form an alkoxysilane intermediate.[9] The intermediate is finally hydrolyzed to give the observed alcohol and a silanediol species that eventually should end in a mixture of inorganic silicic acid derivatives. A role of fluoride ions may be ascribed to the "fluoride ion assisted rearrangement of silyl peroxide" via a supervalent silicon species.[5,10]

(9) To our best knowledge, there has been no report on the preparation of silyl hydroperoxides from alkoxysilanes and hydrogen peroxide. Cf.: (a) Hahn, W.; Metzinger, L. *Makromol. Chem.* **1956**, *21*, 113. (b) Dannley, R. L.; Lalics, G. *J. Org. Chem.* **1965**, *30*, 3848.

(10) Razubaev, G. A.; Yablokov, V. A.; Ganyushkin, A. V.; Yablokova, N. V.; Kalinina, G. S. *J. Organomet. Chem.* **1979**, *165*, 281.

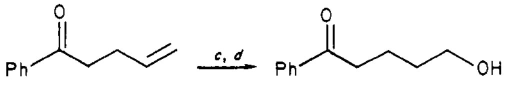
(11) (a) Colvin, E. "Silicon in Organic Synthesis"; Butterworths: London, 1981. (b) Weber, W. P. "Silicon Reagents for Organic Synthesis"; Springer-Verlag: Berlin, Heidelberg, 1983.

of the ready availability of various kinds of sila functional compounds.[13]

By way of such applications, we present here a few preliminary results of the one-pot transformation of certain terminal olefins to the corresponding anti-Markownikoff alcohols via the hydrosilylation–oxidation sequence, as shown by the following examples (eqs 2–4). Isolated yields based on olefins are given.

$$\mathrm{Br(CH_2)_9CH{=}CH_2} \xrightarrow{a,b} \mathrm{Br(CH_2)_{11}OH} \quad 73\% \qquad (2)$$

$$\mathrm{MeO_2C(CH_2)_8CH{=}CH_2} \xrightarrow{a,b} \mathrm{MeO_2C(CH_2)_{10}OH} \quad 79\% \qquad (3)$$

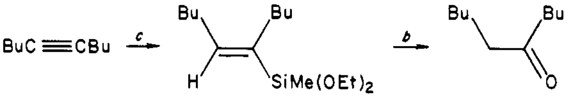


73% (4)

[a] $HSiMe(OEt)_2$ (1.2 equiv), $RhCl(PPh_3)_3$ (0.5 mol %), 80 °C, 3.5–9 h. [b] 30% H_2O_2 (condition A, see text). [c] $HSiMe(OEt)_2$ (1.2 equiv), $H_2PtCl_6 \cdot 6H_2O$ (0.1 mol %), room temperature, 0.5 h. [d] 30% H_2O_2 (condition C, see text).

The most outstanding feature resides in the simplicity, high regioselectivity, and wide functional group compatibility. First, commercially available, air-stable $HSiMe(OEt)_2$ can be used for the Pt-[14] and Rh-catalyzed[15] hydrosilylation, which proceeds smoothly without solvent under air. Rather, an inert-gas atmosphere should not be employed.[16] This makes the present procedure very simple, in contrast to the existing ones that require special techniques to use air-sensitive metal hydrides.[17] Second, these hydrosilylations introduce a silicon atom, and in turn the hydroxy group, onto the terminal carbon atom exclusively.[18] Third, both the hydrosilylation and the H_2O_2 oxidation are compatible with some functional groups such as halogen, ester, and ketone. Of particular interest is the tolerance of the ketone functionality: none of the hydroboration, hydroalumination, and hydrozirconation reactions are compatible with the ketone group.[17,19] It should be also noted that the isolation of the desired alcohol is quite easy because the byproducts are only water-soluble lower alcohols arising from the other ligands on silicon and inorganic silicic acid derivatives.

Finally, the one-pot procedure is also applicable to the conversion of acetylene to ketone, as shown by eq 5 (see eq 2–4 for conditions). The present method involves the direct conversion of a vinylsilane to the corresponding ketone, not via the traditional epoxidation–acidolysis sequence.[20] Further applications of the present new methodology are currently under investigation.



82%

(5)

Acknowledgment. We thank Shin-etsu Chemical Industrial Co., Ltd., for partial support and a gift of chlorosilanes and Mitsubishi Gas-Chemical Industrial Co., Ltd., for a gift of 90% hydrogen peroxide.

Registry No. *n*-$C_8H_{17}SiMe(OEt)_2$, 2652-38-2; H_2O_2, 7722-84-1; Ac_2O, 108-24-7; *t*-BuOOH, 75-91-2; MCPBA, 937-14-4; KF, 7789-23-3; HKF_2, 7789-29-9; $KHCO_3$, 298-14-6; $NaHCO_3$, 144-55-8; K_2CO_3, 584-08-7; *n*-C_8H_{17}–OH, 111-87-5; *n*-C_8H_{17}–$Si(OMe)_3$, 3069-40-7; *n*-C_8H_{17}–$Si(OEt)_3$, 2943-75-1; *n*-C_8H_{17}–$SiMe_2$(OBu-*t*), 87261-97-0; *n*-C_8H_{17}–SiMe(OBu-*t*)$_2$, 87281-30-9; *n*-C_8H_{17}–$SiMe_2$(OEt), 87281-31-0; *n*-C_8H_{17}–$SiMe_2$(OPr-*i*), 87261-98-1; *n*-C_8H_{17}–SiMe(OPr-*i*)$_2$, 87261-99-2; $Br(CH_2)_9CH{=}CH_2$, 7766-50-9; $MeO_2C(CH_2)_8CH{=}CH_2$, 111-81-9; $Br(CH_2)_{11}OH$, 1611-56-9; $MeO_2C(CH_2)_{10}OH$, 24724-07-0; $RhCl(PPh_3)_3$, 14694-95-2; H_2PtCl_6, 16941-12-1; $HSiMe(OEt)_2$, 2031-62-1; *exo*-2-norbornyl–SiMe(OEt)$_2$, 87262-00-8; *endo*-2-norbornyl–SiMe(OEt)$_2$, 87262-01-9; *exo*-norbornanol, 497-37-0; *endo*-norbornanol, 497-36-9; 1-phenyl-4-penten-1-one, 3240-29-7; 5-hydroxy-1-phenyl-1-pentanone, 1011-62-7; dibutylacetylene, 1942-46-7; 5-(5-decenyl)diethoxymethylsilane, 87281-32-1; 5-decanone, 820-29-1.

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[12] We have already developed (*i*-PrO)$_2$MeSiCH$_2$MgCl as a new nucleophilic hydroxymethylating agent based on the aspects presented here.[1a]

[13] E.g., see: Annual Surveys by Corey, J. Y. *J. Organomet. Chem. Libr.* **1982**, *13*, 1.

[14] Speier, J. L. *Adv. Organomet. Chem.* **1979**, *17*, 407.

[15] Cf.: Chalk, A. J. *J. Organomet. Chem.* **1970**, *21*, 207.

[16] The role of oxygen or hydroperoxide in the $RhCl(PPh_3)_3$-catalyzed hydrosilylation: Kickers, H. M.; Haszeldine, R. N.; Malkin, L. S.; Mather, A. P.; Parish, R. V. *J. Chem. Soc., Dalton Trans.* **1980**, 308. We have often observed also in the platinum-($H_2PtCl_6 \cdot 6H_2O$) catalyzed hydrosilylation that no reaction starts under an inert-gas atmosphere, but once the reaction mixture is exposed to air for a while, a rapid reaction results. There might be an acceleration effect by oxygen. See also: Harrod, J. F.; Chalk, A. J. In "Organic Syntheses via Metal Carbonyls"; Wender, I., Pino, P., Eds.; Wiley: New York, 1977; Vol. II, pp 673–704.

[17] Hydroboration: (a) Brown, H. C. "Organic Syntheses via Boranes"; Wiley: New York, 1975. Hydroalumination: (b) Sato, F.; Sato, S.; Kodama, H.; Sato, M. *J. Organomet. Chem.* **1977**, *142*, 71.. (c) Isagawa, K.; Tatsumi, K.; Otsuji, Y. *Chem. Lett.* **1977**, 1117. Hydrozirconation: (d) Schwartz J.; Labinger, J. A. *Angew. Chem., Int. Ed. Engl.* **1976**, *15*, 333.

[18] For example, in the hydroboration, appropriate structural modifications are necessary to attain the high regioselectivity.[17a]

[19] Cf.: (a) Brown, H. C.; Subba Rao, B. C. *J. Am. Chem. Soc.* **1960**, *82*, 681. (b) Brown, H. C.; Karytnyk, W. *Ibid.* **1960**, *82*, 3866.

[20] (a) Stork, G.; Colvin, E. *J. Am. Chem. Soc.* **1971**, *93*, 2080. (b) Hudrlik, P. F.; Hudrlik, A. M.; Rona, R. J.; Misra, R. N.; Withers, G. P. *Ibid.* **1977**, *99*, 1993.

Unexpected Rearrangement of the Deprotonation Product of a Thioformic Acid Ester–Hexacarbonyldiiron Complex: Formation of an Exocluster Sulfonium Function[†]

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Summary: The carbanion formed by lithium diisopropylamide deprotonation of (μ-SCHSMe)Fe$_2$(CO)$_6$ undergoes intramolecular nucleophilic attack at iron to give a C–Fe bond with concomitant Fe–S bond cleavage to form a new anion. Alkylation then produces an exocluster sulfonium function. The structure of the alkylation product obtained by reaction with MeI was determined by X-ray crystallography.

In a previous communication[2] we have reported the novel rearrangement of the anion formed by the deprotonation of (μ-CH$_2$S$_2$)Fe$_2$(CO)$_6$, 1 (eq 1). This sequence,

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[†] Dedicated to Professor Ernst Otto Fischer on the occasion of his 65th birthday on November 10, 1983.

[1] (a) Massachusetts Institute of Technology. (b) University of Alberta. (c) Natural Sciences and Engineering Research Council Postdoctoral Fellow.

[2] Seyferth, D.; Womack, G. B.; Song, L.-C.; Cowie, M.; Hames, B. W. *Organometallics* **1983**, *2*, 928.

0276-7333/83/2302-1696$01.50/0