6.60–7.30 (m, 5 H, C_6H_5); ^{13}C NMR ($CDCl_3$, 67.9 MHz) δ 10.96 (q, *J* = 128.3 Hz, $CH_3CH_2C{=}NPh$), 22.71 (q, *J* = 139.2 Hz, SCH_3), 39.51 (t, *J* = 129.3 Hz, $CH_3CH_2C{=}NPh$), 121.24 (d, *J* = 161.0 Hz, C_6H_5), 126.42 (d, *J* = 161.7 Hz, C_6H_5), 129.44 (d, *J* = 161.0 Hz, C_6H_5), 152.83 (s, ipso C_6H_5), 210.04, 210.34, and 213.46 (all s, FeCO), 236.06 (s, C=N). Anal. Calcd for $C_{16}H_{13}Fe_2NO_6S$: C, 41.86; H, 2.85. Found: C, 41.76; H, 2.86.

(μ-MeC=NPh)(μ-EtS)$Fe_2(CO)_6$ (8d): red solid; 34% yield; mp 81.0–82.5 °C (pentane); IR, terminal carbonyl region (pentane), 2062 (s), 2045 (vw), 2020 (vs), 1990 (vs), 1972 (m), 1965 (m), 1940 (vw) cm^{-1}; ^1H NMR (CD_2Cl_2, 90 MHz) δ 1.50 (t, *J* = 7.32 Hz, 3 H, SCH_2CH_3), 1.96 (s, 3 H, $CH_3C{=}NPh$), 2.61 (q, *J* = 7.32 Hz, 2 H, SCH_2CH_3), 6.51–7.65 (m, 5 H, C_6H_5); ^{13}C NMR ($CDCl_3$, 67.9 MHz) δ 18.37 (q, *J* = 128.1 Hz, SCH_2CH_3), 33.88 (t, *J* = 140.0 Hz, SCH_2CH_3), 34.55 (q, *J* = 127.8 Hz, $CH_3C{=}NPh$), 121.52 (d, *J* = 160.4 Hz, C_6H_5), 126.43 (d, *J* = 162.0 Hz, C_6H_5), 129.25 (d, *J* = 161.8 Hz, C_6H_5), 152.29 (s, ipso C_6H_5), 209.94, 210.07, and 213.91 (all s, FeCO), 230.83 (s, C=N). Anal. Calcd for $C_{16}H_{13}Fe_2NO_6S$: C, 41.86; H, 2.85. Found: C, 41.83; H, 2.87.

Reaction of [Li][(μ-CO)(μ-MeS)$Fe_2(CO)_6$] with Ph(Cl)-C=NPh. A 200-mL Schlenk flask equipped with a stir bar and rubber septum was charged with 1.51 g (3.00 mmol) of $Fe_3(CO)_{12}$, degassed by three evacuation/nitrogen-backfill cycles and then charged with 20 mL of THF by syringe. A cloudy solution of 3.00 mmol of lithium methanethiolate in 20 mL of THF (prepared in a separate flask at 0 °C by the reaction of an excess of methyl mercaptan with 3.00 mmol of *n*-butyllithium) was subsequently added by cannula. An immediate reaction ensued (with brisk gas evolution) and a gradual color change from dark green to brown-red was observed. The resulting [Li](μ-CO)(μ-MeS)$Fe_2(CO)_6$] reagent solution was stirred for 20 min at room temperature and subsequently used in situ without further purification.

To the previously prepared [Li][(μ-CO)(μ-MeS)$Fe_2(CO)_6$] reagent solution (3.00 mmol) was added by cannula 0.65 g (3.01 mmol) of *N*-phenylbenzimidoyl chloride dissolved in a separate flask in 20 mL of THF. After the resulting reaction mixture had been stirred for 60 h at room temperature, the solvent was removed in vacuo to yield a brown-red tar which was purified by filtration chromatography. Pentane eluted an orange band which gave 0.21 g (0.56 mmol, 37% based on S) of $(\mu\text{-MeS})_2Fe_2(CO)_6$ identified by its ^1H NMR spectrum.[28] Pentane eluted a second orange band which gave 0.94 g (1.84 mmol, 61%) of (μ-PhC=NPh)(μ-MeS)-$Fe_2(CO)_6$ (**8a**) identified by comparison of its ^1H NMR spectrum to that of an authentic sample.

Synthesis of (μ-PhC=NPh)(μ-*t*-BuS)$Fe_2(CO)_6$. A 250-mL Schlenk flask equipped with a spin bar and rubber septum was charged with 1.51 g (3.00 mmol) of $Fe_3(CO)_{12}$ and degassed by three evacuation/nitrogen-backfill cycles. The flask was then charged with 50 mL of THF, 0.42 mL (3.00 mmol) of triethylamine, and 0.34 mL (3.00 mmol) of *tert*-butyl mercaptan by syringe. The reaction mixture was stirred for 20 min at room temperature during which time slow gas evolution as well as a color change from dark green to brown-red were observed. The resulting $[Et_3NH]$[(μ-CO)(μ-*t*-BuS)$Fe_2(CO)_6$] reagent solution subsequently was used in situ without further purification.

To the previously prepared $[Et_3NH]$[(μ-CO)(μ-*t*-BuS)$Fe_2(CO)_6$] reagent solution (3.00 mmol) cooled to −78 °C was added by cannula 0.65 g (3.01 mmol) of *N*-phenylbenzimidoyl chloride dissolved in a separate flask in 20 mL of THF (also cooled to −78 °C). The reaction mixture was stirred for 15 min at −78 °C, warmed to room temperature, and then stirred for 40 h at room temperature. Subsequently, the solvent was removed in vacuo to yield a red, oily solid which was purified by filtration chromatography. Pentane eluted an orange band which was not collected. Pentane eluted a second orange band which gave 1.40 g (2.54 mmol, 85%) of (μ-PhC=NPh)(μ-*t*-BuS)$Fe_2(CO)_6$ (**8e**) as a red, air-stable solid: mp 124.0–126.0 °C after recrystallization from pentane: IR, terminal carbonyl region (pentane), 2068 (m), 2025 (vs), 1998 (vs), 1990 (vw), 1980 (vw) cm^{-1}; ^1H NMR (CD_2Cl_2, 90 MHz) δ 1.55 (s, 9 H, $SC(CH_3)3)$, 6.55–7.22 (m, 10 H, C_6H_5); ^{13}C NMR ($CDCl_3$, 67.9 MHz) δ 34.18 (q, *J* = 126.2 Hz, $SC(CH_3)_3$), 49.22 (s, $SC(CH_3)_3$), 120.84–129.92 (m, C_6H_5), 146.75 (s, ipso C_6H_5), 153.18 (s, ipso C_6H_5), 209.23, 210.96, and 213.75 (all s, FeCO), 232.88 (s, C=N). Anal. Calcd for $C_{23}H_{19}Fe_2NO_6S$: C, 50.30; H, 3.49. Found: C, 50.46; H, 3.61.

Acknowledgment. We are grateful to the National Science Foundation for generous support of this work.

Registry No. 8a, 111905-37-4; **8b**, 111905-38-5; **8c**, 111905-39-6; **8d**, 111905-40-9; **8e**, 111905-41-0; $Fe_3(CO)_{1i}$, 17685-52-8; PhC(SMe)=NPh, 6780-43-4; MeC(SMe)=NPh, 58276-22-5; EtC(SMe)=NPh, 69079-30-7; MeC(SEt)=NPh, 87876-89-9; PhC(Cl)=NPh, 4903-36-0; methyl mercaptan, 74-93-1; *tert*-butyl mercaptan, 75-66-1.

Supplementary Material Available: IR and mass spectral data for new compounds (4 pages). Ordering information is given on any current masthead page.

Dimethylsilanone ($Me_2Si{=}O$) Generation by Retroene Decomposition of Allyloxysilanes

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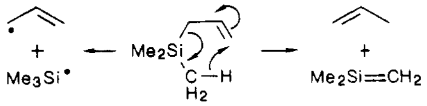
Department of Chemistry, Iowa State University, Ames, Iowa 50011

Received August 19, 1987

Gas-phase thermolysis of (allyloxy)dimethylsilane cleanly affords dimethylsilanone by retroene decomposition (log *A* = 12.6, E_a = 54.6 kcal/mol). However, substituting the ether-linked carbon with two methyl groups (3-(dimethylsiloxy)-3-methyl-1-butene) introduces a competition between C–O bond homolysis and retroene decomposition.

Introduction

The thermolysis of allylsilanes (e.g., **1**) has proved to be an attractive route to silenes (**2**) via retroene elimination of propene.[1] After more than a decade of controversy, it was finally established[2] that the unimolecular decomposition of allyltrimethylsilane (**1**) proceeds by two competitive processes—a concerted retroene reaction and Si–C bond homolysis.



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2

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(1) For example: Block, E.; Revelle, L. K. *J. Am. Chem. Soc.* **1978** *100*, 1630; Barton, T. J.; Burns, G. T. *J. Am. Chem. Soc.* **1978**, *100*, 5246; Barton, T. J.; Burns, G. T.; Arnold, E. V.; Clardy, J. *Tetrahedron Lett.* **1981**, *22*, 7; Burns, G. T.; Barton, T. J. *J. Organomet. Chem.* **1981**, *209*, C25; Barton, T. J.; Burns, G. T. *Organometallics* **1982**, *1*, 1455.

(2) Barton, T. J.; Burns, S. A.; Davidson, I. M. T.; Ijadi-Maghsoodi, S.; Wood, I. T. *J. Am. Chem. Soc.* **1984**, *106*, 6367.

0276-7333/88/2307-0528$01.50/0

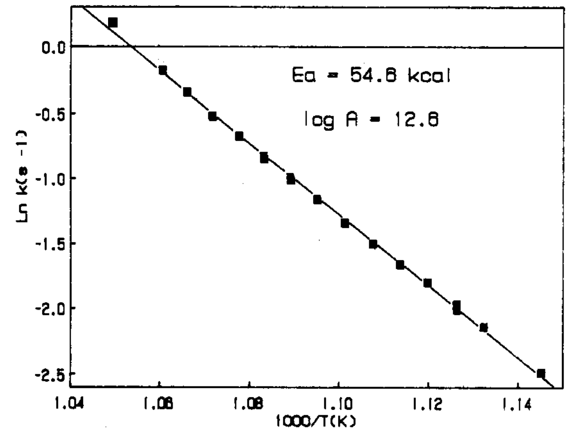
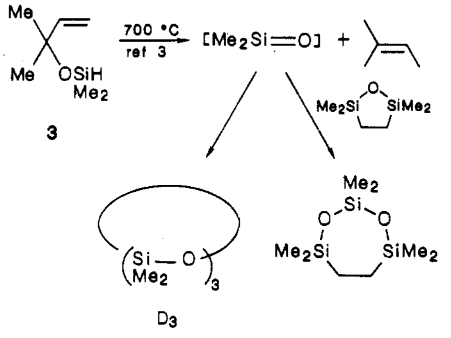


Figure 1. Arrhenius plot for the formation of propene in the thermolysis of 4.

To date there has been only a single mention in the literature of extension of this reaction to the generation of systems containing silicon multiply bonded to elements other than carbon. In 1979 it was briefly reported by Lane and Frye[3] that when 3-(dimethylsiloxy)-3-methyl-1-butene (3) was distilled through a ketene generator heated to 700 °C, the products were 2-methyl-2-butene and hexamethylcyclotrisiloxane (D_3). It was reasonably suggested

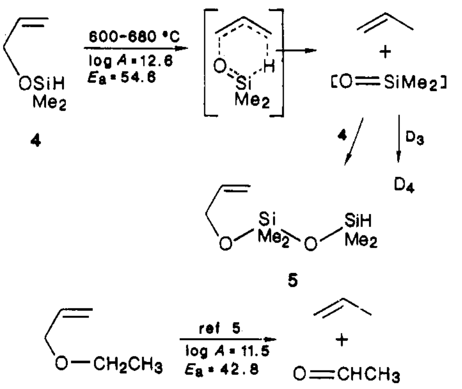


that the decomposition of 3 involved extrusion of dimethylsilanone, $Me_2Si{=}O$, although no mechanistic suggestions were proffered. Indeed, strong evidence for the intermediacy of $Me_2Si{=}O$ was obtained through trapping by insertion into Si—O bonds of established silanone traps.

As part of an extensive study of the production of multiply bonded silicon via thermal decompositions, we herein report our results on the formation of dimethylsilanone from the retroene reaction.

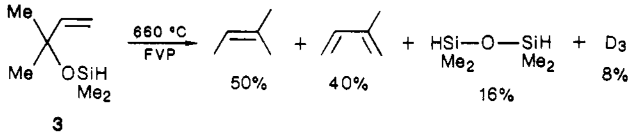
Results and Discussion

As we were concerned (vide infra) about complications due to the two C-substituted methyls in 3, the parent system, allyl dimethylsilyl ether (4), was first synthesized and studied. The gas-phase pyrolysis of 4 was examined in a stirred flow reactor (SFR) modeled after the design of Davidson.[4] Over the temperature range of 600–680 °C, the pyrolysis of 4 cleanly produced propene, D_3, and disiloxane 5. All products are consistent with the extrusion of $Me_2Si{=}O$ from 4, with 5 being presumably formed by insertion of $Me_2Si{=}O$ into the Si—O bond of 4. In order to eliminate this source of removal of 4, the pyrolyses were conducted with a 50-fold excess of D_3, which efficiently



trapped $Me_2Si{=}O$ to produce octamethylcyclotetrasiloxane (D_4) and eliminate the formation of 5. Kinetics were conveniently obtained by following the formation of propene, and a 20-point plot of the rate constants (Figure 1) fit the Arrhenius relationship log k (s^{-1}) = 12.57 ± 0.11 (54.62 ± 0.45 kcal/mol)/2.303RT. The preexponential factor of $10^{12.6}$ ($\Delta S^{\ddagger} = -5.2$ cal/(mol deg)) is consistent with the six-center transition state of a retroene decomposition of 4. For comparison, the retroene decomposition of ethyl allyl ether (6) has Arrhenius parameters[5] of log A = 11.5 and E_a = 42.8 kcal/mol. The somewhat larger A factor for 4 may be speculatively attributed to a more polar and less synchronous reaction. Clearly, neither Arrhenius parameter for 4 is consistent with decomposition initiated by homolysis of the C–O bond, which should require >70 kcal/mol. Further evidence that propene did not arise from allyl radical intermediacy was obtained by copyrolysis of 4 and toluene-d_8 (21-fold excess) with no resulting deuterium incorporation in the propene product.[6]

Even though the decomposition of 4 appeared to be a clean retroene reaction, it could not be assumed that the same was true for its dimethyl derivative 3. In 3, the possibility of C–O homolysis is considerably enhanced by the resulting formation of a tertiary carbon-centered radical. Thus, a more extensive examination of the thermolysis of 3 was undertaken. Flash vacuum pyrolysis (FVP) of 3 (660 °C, ca. 10^{-4} Torr, 75% decomposition) afforded, in addition to the reported[3] 2-methyl-2-butene (50%) and D_3 (8%), isoprene (40%) and *sym*-tetramethyldisiloxane (16%).



While the formation of 2-methyl-2-butene and D_3 can be taken as prima facie evidence for retroene extrusion of $Me_2Si{=}O$, both isoprene and tetramethyldisiloxane suggest initial homolysis of the C–O bond. The resulting two radicals, 7 and $Me_2HSiO^{\bullet}$, might be expected to disproportionate to produce isoprene and dimethylsilanol, the latter of which can self-condense to afford the observed disiloxane. An attempt to trap 7 by copyrolysis of 3 and excess toluene-d_8 was unsuccessful in that there was no deuterium incorporation in the methylbutene, and the ratio of isoprene to methylbutene was unchanged. Thus, not surprisingly, hydrogen abstraction by 7 cannot compete

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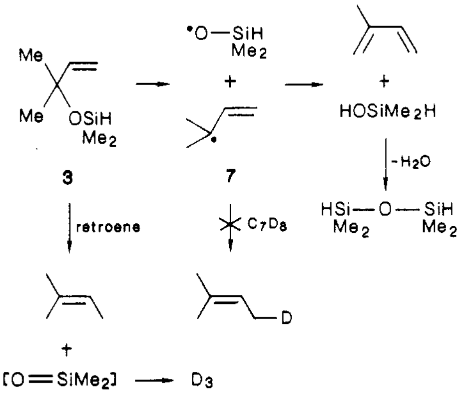
[3] Lane, T. H.; Frye, C. L. *J. Organomet. Chem.* **1979**, *172*, 213.

[4] Baldwin, A. C.; Davidson, I. M. T.; Howard, A. V. *J. Chem. Soc., Faraday Trans. 1* **1975**, *71*, 972.

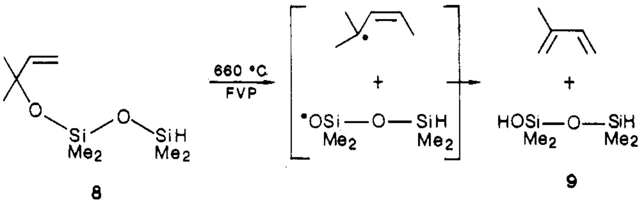
[5] Egger, K. W.; Vitins, P. *Int. J. Chem. Kinet.* 1974, *6*, 429.

[6] Additional evidence against C–O homolysis in 4 is our observation that $Me_3SiOCH_2CH{=}CH_2$ does not decompose over the temperature range (600–680 °C) employed in the pyrolysis of 4.



with hydrogen loss to form the conjugated system.

An additional test of the radical route to isoprene was found in the pyrolysis of disiloxane **8**.[3] Compound **8** contains all of the elements of **3** but cannot undergo a retroene reaction. FVP of **8** (670 °C, 90% decomposition) afforded isoprene and silanol **9** as virtually exclusive products.



In our SFR system, which exits into a packed-column gas chromatograph, the two C_5 products from the pyrolysis of **3** were not cleanly separated, and thus our attempts to obtain kinetic parameters for the formation of each of these products are fraught with some indeterminable error. Irregardless, the Arrhenius parameters (Figures 2 and 3) obtained over a rather narrow temperature range (540–590 °C) for 2-methyl-2-butene ($\log A = 13.0 \pm 0.2$, $E_a = 51.6 \pm 0.7$ kcal/mol) and isoprene ($\log A = 17.2 \pm 0.3$, $E_a = 69.1 \pm 1.3$ kcal/mol) clearly point to different mechanisms of formation: the former, concerted; the latter, homolysis.

Summary

(Allyloxy)dimethylsilane (**4**) was found to thermally decompose to dimethylsilanone by a concerted retroene elimination of propene. However, due to greater radical stability, homolytic cleavage competes with retroene decomposition in 3-(dimethylsiloxy)-3-methyl-1-butene (**3**). Thus, for the production of dimethylsilanone, only **4** is a suitable precursor. We will soon report our results on the formation of silicon–nitrogen and silicon–sulfur double bonds via retroene decompositions.

Experimental Section

General. NMR spectra were recorded on a Nicolet Model NT-300 spectrometer at 300 MHz. GC/MS data were obtained on a HP 5970 mass selective detector coupled with a HP 5890 GC. Preparative gas chromatographic (GC) separations were performed on a Varian Model 920 instrument. Flash vacuum pyrolyses (FVP) were carried out by slowly distilling compounds through a heated, seasoned horizontal quartz pyrolysis tube (16-mm i.d., 200 mm long) packed with quartz chips, with product collection in a trap cooled with liquid N_2. Pressures were measured by an ion gauge placed behind a liquid N_2 trap. The stirred flow reactor (SFR) system is modeled after that described by Baldwin.[4] Our SFR system uses a 60 mL/min He flow to sweep the material through the reactor into a Varian 6000 gas chromatograph (flame ionization detector (FID)) and has the option of diverting the separated products into a VG SX-300 quadrupole mass spectrometer (MS) for mass analysis.

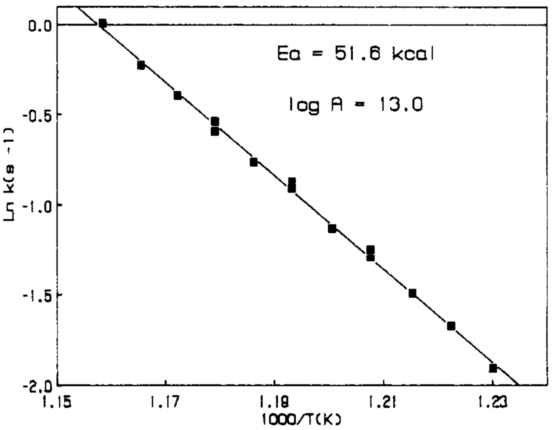


Figure 2. Arrhenius plot for the formation of 2-methyl-2-butene from the thermolysis of 3.

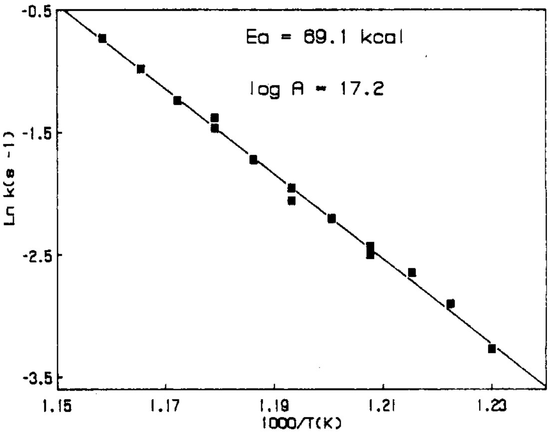


Figure 3. Arrhenius plot for the formation of isoprene from the thermolysis of 3.

Synthesis of Allyl Dimethylsilyl Ether (4). A 100-mL three-neck flask, equipped with magnetic stirrer and N_2 inlet, was charged with Me_2SiHCl (5.2 g, 54.7 mmol), CaH_2 (3.2 g, 76 mmol), and Et_2O (40 mL). After the mixture was cooled to 0 °C, 2.1 g (36.1 mmol) of allyl alcohol was added dropwise (syringe, 5 min). The mixture was warmed to room temperature and stirred for 24 h. Trap-to-trap distillation was followed by fractional distillation (17 cm Vigreux) to afford 1.9 g (16.6 mmol, 46%) of **4** (bp 87–90 °C, literature bp 83–84 °C[7]), which contained (by GC) a very small amount of allyl alcohol. Pure **4** for kinetic studies was obtained by preparative GC and characterized by GC/MS *m/z* (% relative intensity) 116 (M^+, 1.4), 115 (11), 101 ($M - CH_3$, 50), 99 (59), 75 ($M - C_3H_5$, 97), 61 (41), 59 (MeSiO or Me_2HSi, 94), 45 (HSiO, 100), 43 (MeSi, 55), 41 (C_3H_5, 43) and ^1H NMR ($CDCl_3$) δ 0.21 (6 H, d, $J = 2.8$ Hz), 4.16 (2 H, m), 4.63 (1 H, heptane, $J = 2.8$ Hz, SiH), 5.09 (1 H, m), 5.21 (1 H, m), 5.91 (1 H, m).

Synthesis of 3-(Dimethylsiloxy)-3-methyl-1-butene (3). Ether **3** was synthesized by the method of Lane and Frye.[3] Characterization was by GC/MS and ^1H NMR.

Synthesis and FVP of 1-(Vinyldimethylcarbinoxy)-1,1,3,3-tetramethyldisiloxane (8). Ether **8** was prepared by the method of Lane and Frye[3] and characterized by GC/MS and ^1H NMR. FVP of **8** was carried out at 670 °C with a conversion of ca. 90%. Isoprene and 1,1,3,3-tetramethyldisiloxanol (**9**) were almost the exclusive products with only trace amounts of other products detectable by capillary GC. Disiloxane **9**: GC/MS *m/z* (% relative intensity) 150 (M^+, 1.8, 149 ($M - 1$, 13), 135 ($M - CH_3$, 100), 133 (48), 119 (58), 89 (10), 75 (38), 61 (20), 47 (19), 45 (34).

Copyrolysis of 4 and Toluene-d_8. The copyrolysis of **4** and C_7D_8 was conducted in the SFR at 640 °C with a 60 mL/min He

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(7) Mironov, V. F.9; Kozlikov, V. L.; Fedotov, N. S. *Zh. Obshch. Khim.* **1969**, *39*, 966.

flow. A mixture of 0.7 Torr 4 and 15 Torr C_7D_8 was employed, and the production of propene after GC separation was monitored by a quadrupole mass spectrometer. The relative intensities of the m/z 41, 42, and 43 ions were identical with those of the propene derived from SFR pyrolysis of 4 in the absence of toluene-d_8.

Copyrolysis of 3 and Toluene-d_8. Copyrolysis of 3 (0.2 Torr) and toluene-d_8 (8.3 Torr) at 550 °C was carried out in the SFR. No deuterium incorporation into 2-methyl-2-butene was detected in the mass spectrum nor did the ratio of isoprene to 2-methyl-2-butene differ from that observed in the pyrolysis of 3 in the absence of C_7D_8.

FVP of 3. Ether 3 was pyrolyzed by distillation through the FVP unit (vide supra) at 660 °C. The products were collected in a liquid N_2-cooled trap and *n*-decane was added as a GC standard. The major products, isoprene (40%), 2-methyl-2-butene (50%), and *sym*-tetramethyldisiloxane (16%), were isolated by preparative GC and characterized by GC/MS and ^1H NMR. D_3 (8%) was solely characterized by GC/MS comparison with authentic material.

Acknowledgment. The National Science Foundation is gratefully acknowledged for funding of this work. The Department of Energy, Division of Materials Sciences, is acknowledged for funds utilized in the construction of the stirred flow reactor. Dr. S. Ijadi-Maghsoodi, of the Ames Laboratory is acknowledged for his invaluable aid in the kinetic experiments.

Registry No. 3, 23483-22-9; 4, 23483-20-7; 8, 71210-10-1; $Me_2Si{=}O$, 47956-45-6; Me_2SiHCl, 1066-35-9; allyl alcohol, 107-18-6.

Oxygen Atom Transfer Reactions to Metal Carbonyls. Kinetics and Mechanism of CO Substitution Reaction of $Cr(CO)_6$ in the Presence of C_6H_5IO

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Received August 24, 1987

Reported are rates of reaction and activation parameters for the reaction of $Cr(CO)_6$ with CH_3CN in the presence of C_6H_5IO to form $Cr(CO)_5(NCCH_3)$. The rate of reaction is first order in concentrations of $Cr(CO)_6$ and of C_6H_5IO but zero order in CH_3CN concentration. Two possible mechanisms are suggested for the reaction, one is similar to that proposed earlier for corresponding reactions using $(CH_3)_3NO$ as the oxygen atom transfer reagent. A quantitative comparison between the reactivities of C_6H_5IO and $(CH_3)_3NO$ toward $Cr(CO)_6$ was not possible, but qualitatively the reaction using C_6H_5IO is orders of magnitude faster than that using $(CH_3)_3NO$. In order to dissolve polymeric $(C_6H_5IO)_n$, it was allowed to react with CH_3OH to form $C_6H_5I(OCH_3)_2$, which is soluble. However, its tetrahydrofuran solution does not react with $Cr(CO)_6$ unless H_2O is added. The rate of reaction increases with increasing H_2O concentration, and the rate varies inversely with the square of CH_3OH concentration. This behavior is explained in terms of a rapid preequilibrium that generates free reactive molecular C_6H_5IO.

Introduction

Considerable research effort has recently been devoted to the use of iodosobenzene (PhIO) as an oxygen atom transfer reagent to organic compounds, particularly in the presence of transition-metal porphyrins.[1] An example of the use of PhIO in organometallic chemistry is provided by its use[2] for the synthesis of $[\eta^5\text{-}C_5(CH_3)_5ReCONOPPh_3]BF_4$. The reaction of PhIO with metal carbenes is said[3] to be a convenient and rapid chemical method for the characterization of carbenoid metal complexes. Schardt and Hill[4] call attention to the fact that PhIO is a polymer insoluble in most solvents, but the polymer reacts with methanol to readily form $PhI(OCH_3)_2$ which is soluble in a variety of solvents. Their article also references several reports of the use of PhIO as an oxygen atom transfer reagent.

In spite of all the research activity with PhIO, we know of no kinetic study on its reactions with metal carbonyls. Our studies[5] on the kinetics and mechanisms of reactions of metal carbonyls with Me_3NO prompt us to want to investigate other oxygen atom transfer reagents. This paper presents the results of the reaction of $Cr(CO)_6$ with CH_3CN in the presence of PhIO to afford $Cr(CO)_5(NCCH_3)$.

Experimental Section

Compounds and Solvents. The carbonyl $Cr(CO)_6$ was obtained from Strem Chemicals and purified by sublimation. Iodosobenzene was synthesized by a literature method.[6] Methanol and tetrahydrofuran (THF) were dried with $Mg(OCH_3)_2$ and sodium benzophenone ketyl, respectively, and distilled prior to use.

Kinetic Measurements. Infrared spectra were recorded on a Nicolet-5DX FT-IR that uses a 0.5-mm NaCl cell. UV-visible

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[†] Lanzhou University.

[‡] Northwestern University.

(1) Groves, J. T.; Nemo, T. E.; Myers, R. S. *J. Am. Chem. Soc.* **1979**, *101*, 1032. Groves, J. T. In *Metal Ion Activation of Dioxygen*, Spiro, T. G., Ed.; Wiley: New York, 1980; p 125.

(2) Patton, A. T.; Strouse, C. E.; Knobler, C. B.; Gladysz, J. A. *J. Am. Chem. Soc.* **1983**, *105*, 5804.

(3) Lukehart, C. M.; Zeile, J. V. *J. Organomet. Chem.* **1975**, *97*, 421.

(4) Schardt, B. C.; Hill, C. L. *Inorg. Chem.* **1983**, *22*, 1563.

(5) Shi, Y. L.; Gao, Y. C.; Shi, Q. Z.; Kershner, D. L.; Basolo, F. *Organometallics* **1987**, *6*, 1528.

(6) Saltzman, H.; Sharefkin, J. G. *Org. Synth.* **1963**, *43*, 60.