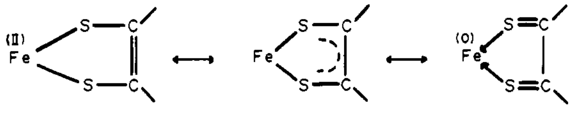
cupying the apical position is much less satisfactory. Thus the atoms S(1) and P(1) lie well below and Fe, S(2) and C(1) well above a best plane through these five atoms (S(1) -0.426, P(1) -0.398, Fe +0.309, S(2) +0.222, C(1) + 0.294 Å) while the angles S(1)-Fe-P(2) = 124.9 (0)° and S(1)-Fe-P(1) = 140.9 (0)° differ markedly from idealized values of 90 and 180°, respectively. The structure contrasts sharply with the structures of orange and violet $Ru[S_2C_2(CF_3)_2](CO)(PPh_3)_2$ where the coordination about the central atom is demonstrably square pyramidal with the carbonyl group respectively axial and equatorial in the two isomers.[13] In the ruthenium systems no evidence was found for trigonal-bipyramidal species either in solution or in the solid state. A number of iron complexes of the type $Fe[S_2C_2(CF_3)_2](CO)L_2$ (L = tertiary phosphine or phosphite) have been prepared by Lewis base additions to $\{Fe[S_2C_2(CF_3)_2](CO)_3\}_2$,[12] but no structural data are available. While it might be tempting to deduce structures analogous to that of **3g**, it seems clear from a comparison of **3g** with the ruthenium complexes that very subtle electronic and steric differences are sufficient to modify solid-state configurations in these systems. Thus any structural generalizations are probably hazardous. In solution compounds **3** do not retain the rigid trigonal bipyramid of the solid state for, even at low temperature, the ^{31}P nuclei are equivalent and in **3a** ^{13}C NMR shows that the two backbone carbon atoms of the dithiolene ligand are indistinguishable.

The dithiolene skeleton defined by the atoms S(3), S(2), C(2), and C(3) in $Fe[S_2C(COMe)C(C_6H_4NO_2)](CO)[P(OMe)_3]_2$ is strictly planar as is invariably found for these ligands. Nevertheless there are some interesting features of the FeS_2C_2 ring that reflect the unsymmetrical nature of the substituents and the stereochemistry of the metal. Thus the Fe-S(2) (trans to CO) bond length (2.196 (1) Å) is significantly longer than the equatorial Fe-S(1) distance (2.155 (1) Å). Both of these bond lengths are however markedly shorter than the Fe-S(1) distances in $Fe(\eta^2\text{-}CS_2)(CO)_2(PPh_3)(PMe_3)$ (2.334 (2) Å)[16] or $Fe[C(S)OMe][Ph_2PCH{=}C(t\text{-}Bu)S](CO)[P(OMe)_3]$ (2.307 (2) Å)[4] where six ligand atoms are present. Comparison of the Fe-S bond lengths (average 2.175 Å) in **3g** with the sum of covalent radii for iron (~1.26 Å) and sulfur (1.04 Å) suggests that the Fe-S bonds are slightly shortened single bonds. The C(3)-S(2) and C(3)-S(1) distances (1.722 (3) and 1.736 (5) Å), which are marginally different, compare favorably with corresponding distances in other dithiolate complexes[24] and are close to the expected value of 1.75 Å for a C(sp^2)-S single bond.[25] The C(2)-C(3) bond length (1.355 (5) Å) corresponds to that expected for a double bond differing from the sp^2-sp^2 single bond length found in the related dithiolate derivative $(C_5H_5)Co[S_2C_2(CF_3)_2]$ (C-C = 1.48 Å)[26] but being rather similar to the corresponding distance (1.364 (10) Å) in $(C_5H_5)[CoS_2C_2(CN)_2]$.[27] There has been considerable discussion in the literature concerning metal-dithiolate bonding and in particular whether the ground states of these molecules are best described in terms of the dithiolate A or the dithioketone B.[24] The structural parameters for **3g** suggest that while there is some delocalization over the S_2C_2 portion of the chelate ring, the canonical from A predominates.



A B

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Registry No. **1**, 64424-66-4; **2a**, 67228-20-0; **2b**, 67228-18-6; **2c**, 77357-83-6; **3a**, 78803-84-6; **3b**, 78790-98-4; **3c**, 78790-99-5; **3d**, 78791-00-1; **3e**, 78791-01-2; **3f**, 78791-02-3; **3g**, 78791-03-4; **4**, 78853-55-1; **5g**, 78791-04-5; **6g**, 78791-05-6; **7**, 64424-68-6; **9a**, 26314-39-6; **9b**, 50615-12-8; **10b**, 78791-06-7; **11**, 360-91-8.

Supplementary Material Available: Listings of hydrogen atom coordinates and isotropic thermal parameters (Table S1) and structure factor amplitudes (14 pages). Ordering information is given on any current masthead page.

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(Trimethylsilyl)methylidene and (Trimethylsilyl)methylidyne Compounds of Molybdenum and Tungsten: $(Me_3SiCH_2)_3M{\equiv}CSiMe_3$ (M = Mo, W) and $(Me_3SiCH_2)_3Mo{=}CHSiMe_3$

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From the reaction between $MoCl_5$ and $LiCH_2SiMe_3$ (5 equiv) at 0 °C in hexane/ether solvent, a purple distillate (70 °C, 10^{-3} torr) has been obtained, in addition to the previously characterized $Mo_2(CH_2SiMe_3)_6$. The purple distillate has been shown to contain the carbene compound $(Me_3SiCH_2)_3Mo{=}CHSiMe_3$ and the carbyne compound $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$ by analytical and spectroscopic studies. When the above reaction is carried out at -78 °C, a yellow distillate (70 °C, 10^{-3} torr) is obtained, which is the pure carbyne compound. Analogous reactions employing WCl_6 and $LiCH_2SiMe_3$ (6 equiv) at -78 °C lead to $(Me_3SiCH_2)_3W{\equiv}CSiMe_3$ as the sole volatile tungsten-containing product. The new compounds have been characterized by elemental analyses and IR, NMR (^1H and ^{13}C), and mass spectroscopy.

Introduction

The reactions between early-transition-metal halides and alkyllithiums or Grignard reagents, in which the alkyl groups lack β-hydrogen atoms, have allowed the syntheses of extensive series of homoleptic alkyl transition-metal complexes.[2,3]

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However, reactions involving halides of Ta(V) and Nb(V) have been found to more commonly yield alkyl-metal alkylidene or alkylidyne compounds when bulky alkyl ligands such as CH_2CMe_3[4] and CH_2SiMe_3[5,6] are involved. Similarly, for molybdenum and tungsten, though it has been possible to isolate homoleptic alkyls such as WMe_6[7,8] and M_2R_6[9-11] ($M{\equiv}M$), complexes containing various combinations of alkyl, alkylidene, and alkylidyne ligands have been isolated.[12-15] We now report further products obtained from reactions between ((trimethylsilyl)methyl)lithium and molybdenum and tungsten chlorides.

Results and Discussion

Syntheses. The reaction between WCl_6 and $LiCH_2SiMe_3$ (6 equiv) at −78 °C in hexane/ether solvent mixtures yields $(Me_3SiCH_2)_3W{\equiv}CSiMe_3$ as a distillable (+70 °C, 10^{-3} torr) yellow liquid. This is the only volatile tungsten-containing compound isolated from this reaction, though its yield is only ca. 10% based on tungsten.

The reaction between $MoCl_5$ and $LiCH_2SiMe_3$ (5 equiv), when carried out in hexane/ether solvent mixtures at −78 °C, yields $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$ as a distillable (70 °C, 10^{-3} torr) yellow liquid, in addition to the previously characterized dimolybdenum compound $Mo_2(CH_2SiMe_3)_6$ ($M{\equiv}M$), which is a yellow solid that sublimes at 100–120 °C, 10^{-4} torr.

When the reaction between $MoCl_5$ and $LiCH_2SiMe_3$ (5 equiv) is carried out in ether/hexane solvent mixtures at ca. 0 °C, a purple distillate is obtained at 70 °C, 10^{-3} torr, in addition to $Mo_2(CH_2SiMe_3)_6$. The purple distillate, which could have been formulated as $Mo(CH_2SiMe_3)_4$ on the basis of elemental analyses, is shown below to consist of a mixture of $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$ and $(Me_3SiCH_2)_3Mo{=}CHSiMe_3$. We have not, however, been able to isolate the pure carbene compound from this mixture.

The reaction between $MoCl_3$ and $LiCH_2SiMe_3$, when carried out in hexane/ether solvent mixtures at either −78 or 0 °C, yields $Mo_2(CH_2SiMe_3)_6$ as the only volatile molybdenum-containing compound.

Properties. $(Me_3SiCH_2)_3M{\equiv}CSiMe_3$ compounds (M = Mo or W) are yellow liquids at room temperature, which solidify on cooling to ca. 5 °C to waxy crystalline solids. The compounds are air sensitive but appear indefinitely stable when stored either under a nitrogen atmosphere or in vacuo. They are extremely soluble in hydrocarbons at room temperatures. In the mass spectrum, they show molecular ions, M^+, together with $[M - 2H]^+$ ions. No M_2-containing ions were observed.

The ^1H and ^{13}C [^1H] NMR spectra in toluene-d_8 are temperature independent in the range −60 to +60 °C and are as expected for discrete molecular species. The methylene protons in $(Me_3SiCH_2)_3W{\equiv}CSiMe_3$ show coupling to ^{183}W, which has $I = {}^1/_2$ and a natural abundance of 14.4%. (However, coupling to the methyne carbon was not resolved.) Analytical data and NMR data are given in the Experimental Section.

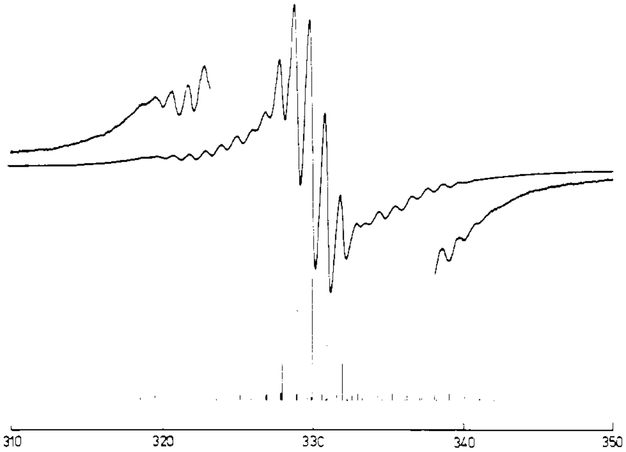

Figure 1. ESR spectrum of a fresh toluene solution of the purple liquid (inset at gain × 5); scale B/mT. Temperature = −30 °C; frequency = 9.127 GHz; microwave power = 200 mW; 100-kHz modulation = 0.2 mT. Stick diagram represents best fit according to equations in ref 17 with $g = 1.977$, $a_{Mo} = 0.00342$ cm^{-1}, and $a_H = 0.00092$ cm^{-1}.

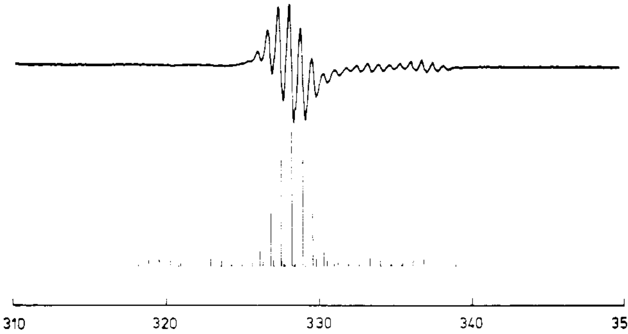

Figure 2. ESR spectrum of a fresh benzene solution of the purple liquid to which has been added a good excess of a petroleum ether solution of $LiCH_2SiMe_3$; scale B/mT. Temperature = 20 °C; frequency = 9.132 GHz; microwave power = 0.2 mW; 100-kHz modulation = 50 μT. Stick diagram represents best fit according to equations in ref 17 with $g = 1.986$, $a_{Mo} = 0.00322$ cm^{-1}, and $a_H = 0.00064$ cm^{-1}.

$(Me_3SiCH_2)_3Mo{=}CHSiMe_3$, which has not been separated from the carbyne compound, is formulated as a purple oxygen-sensitive liquid or low-melting solid on the following grounds.

1. Elemental analyses of the purple distillate are consistent with the presence of four Me_3SiCH_2 groups or related carbene or carbyne ligands per molybdenum.

2. Addition of D_2SO_4 to the purple liquid liberated the following: Me_4Si-d_1, 83%; Me_4Si-d_2, 8.3%; Me_4Si-d_3, 8.3%. These values were determined by mass spectroscopy after correcting for ^{13}C natural abundances. Analogous deuterolysis experiments involving the bridged carbyne complexes gave ratios of Me_4Si-d_1:d_2:d_3 of 8.6:1.0:1.6 and 8.4:1.0:1.8, for M = Ta with W, respectively. We conclude that in the present instance, the Me_4Si-d_1:d_2:d_3 ratio of 10:1:1 is good evidence for the existence of the $Mo{=}CHSiMe_3$ and $Mo{\equiv}CSiMe_3$ moieties, though the ratio may not serve as an accurate measure of the relative concentrations of alkyl, carbene, and carbyne ligands present in the purple distillate.

3. With the assumption of a formula weight equivalent to $Mo(CH_2SiMe_3)_4$, magnetic susceptibility studies, using the Evans method,[16] yield $\mu_{eff} = 1.1$ μ_B. This was not temperature dependent in the range +60 to −60 °C in toluene as solvent.

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4. Hydrocarbon solutions of the purple liquid show both sharp ESR signals and well-resolved NMR spectra; the latter are identical to those of $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$. Toluene, benzene, and decalin solutions of the purple liquid gave ESR signals at room temperature that showed seven lines with relative intensities typical of an incompletely resolved interaction with six equivalent hydrogen atoms. The pattern was repeated at lower intensity for the less abundant isotopes ^{95}Mo and ^{97}Mo, which have $I = {}^5/_2$. The best resolved spectrum was obtained in toluene at −30 °C and is shown in Figure 1, together with the simulated spectrum:[17] $g_{iso} = 1.977$, $a_H = 0.00092$ cm^{-1}, $a_{Mo} = 0.00342$ cm^{-1}. When the liquid was frozen at −150 °C, all proton and molybdenum hyperfine structure was lost, yielding only a sharp, slightly asymmetric signal whose points of maximum slope corresponded to $g_{\parallel} = 1.998$ and $g_{\perp} = 1.967$. Thus, g_{iso}(calcd) = 1.978, which is in close agreement with that observed at room temperature.

Addition of a petroleum solution of $LiCH_2SiMe_3$ (in excess) to a freshly prepared benzene solution of the purple liquid gave the ESR spectrum shown in Figure 2. The spectrum is interpretable in terms of a singlet ground state, $S = {}^1/_2$, and interaction of the electron with eight equivalent hydrogen atoms and ^{95}Mo and ^{97}Mo isotopes, $I = {}^5/_2$, and may be so simulated:[17] $g_{iso} = 1.986$, $a_H = 0.00064$ cm^{-1}, $a_{Mo} = 0.00322$ cm^{-1}. With time, the signal decayed.

We assign the ESR spectrum of the purple liquid (Figure 1) to the carbene species $Mo(CHSiMe_3)(CH_2SiMe_3)_3$ and the spectrum obtained upon adding excess $LiCH_2SiMe_3$ (Figure 2) to the anion $[Mo(CHSiMe_3)(CH_2SiMe_3)_4]^-$.

The ESR spectra (Figures 1 and 2) are incompatible with a d^2 ion and may not be attributed to $Mo(CH_2SiMe_3)_4$; cf. $Cr(CH_2SiMe_3)_4$.[18] For $Mo(CH_2SiMe_3)_3(CHSiMe_3)$ and $Mo(CH_2SiMe_3)_4(CHSiMe_3)^-$, the highest symmetry point groups to which the MoC_6C' and MoC_4C' moieties may belong are C_{3v} and C_{4v}, respectively. In both groups, the unpaired electron would occupy the degenerate pair (d_{xz}, d_{yz}). However, metal-to-carbene π-bonding would remove this degeneracy leading to the observed orbital singlet ground state. Proton hyperfine structure would be expected through a spin-polarization mechanism in the C–H bonds, its magnitude being proportional in each case to the unpaired spin density on the carbon. The observation of hfs from methylene protons, but not from the carbene proton, may then be rationalized if it is assumed that the unpaired electron on the molybdenum creates spin density on the four attached carbons approximately equally but that the carbene carbon loses spin density back to the molybdenum through its π bond. The proton hfs from this C–H bond is thus presumed too small to be resolved.

Concluding Remarks. This work provides further examples of the complexities that arise during metathetic reactions involving the chlorides of molybdenum and tungsten and organolithium reagents. The alkylmetal complexes are only formed in low yields, though once isolated in pure states, they are thermally quite stable. The reaction pathways leading to their formation are unknown; $Mo_2(CH_2SiMe_3)_6$, $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$, and $(Me_3SiCH_2)_3Mo{=}CHSiMe_3$ are all formed in the reaction between $MoCl_5$ and $LiCH_2SiMe_3$ (5 equiv).

Experimental Section

Dry and oxygen-free atmospheres (N_2) and solvents were used throughout. Bench-top manipulations involved standard Schlenk and vacuum techniques. Sampling procedures were carried out in a Vacuum Atmospheres Co. Dri-Lab assembly.

$(Me_3SiCH_2)_3Mo{=}CHSiMe_3$ + $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$. $MoCl_5$ (13.2 g, 48.3 mmol) was added slowly with stirring to a solution of $LiCH_2SiMe_3$ (21.0 g, 221 mmol) in hexane (200 mL) and ether (100 mL) at 0 °C under N_2. A dark-red solution was formed, together with insoluble salts. The solution was allowed to warm to room temperature and stirred for 3 h. Solvent was removed in vacuo, and the residue was extracted with pentane (150 mL) and separated by filtration. The pentane filtrate was taken to dryness in vacuo, and the brown residue was heated (10^{-3} torr, 60–80 °C). A purple liquid was collected on a liquid-nitrogen-cooled cold finger. (The residue after distillation contained $Mo_2(CH_2SiMe_3)_6$, which was sublimed at 100–120 °C, 10^{-3} torr.) The purple liquid (ca. 5 mL) was collected from the cold finger and redistilled (70 °C, 10^{-3} torr). Anal. Calcd for $Mo(CH_2SiMe_3)_4$: C, 43.21; H, 9.97. Found: C, 43.37; H, 9.80; Li < 30 ppm.

$(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$. $MoCl_5$ (8.0 g) was added slowly ($1^1/_2$ h) with stirring to a solution of $LiCH_2SiMe_3$ (11 g, 5 equiv) in ether (200 mL)/hexane (100 mL) cooled in a dry ice/acetone slush bath (−78 °C). When the addition was complete, the solution was allowed to warm to room temperature slowly. The solvent was stripped, and the residues were extracted with pentane (150 mL). The brown filtrate was collected, pentane was stripped, and the sticky solid was heated in vacuo. At 70 °C, 10^{-3} torr, a yellow liquid distilled: $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$ (0.87 g, 7.5% yield based on Mo). Anal. Calcd for $MoSi_4C_{16}H_{42}$: C, 43.39; H, 9.56. Found: C, 43.63; H, 9.45. ^1H NMR data obtained in toluene-d_8 at 220 MHz, 16 °C: $\delta(CH_2Si\mathit{Me}_3) = 0.15$ (9 H), $\delta(CSi\mathit{Me}_3) = 0.42$ (3 H), $\delta(C\mathit{H}_2SiMe_3) = 1.16$ (2 H) (δ in ppm relative to Me_4Si). ^{13}C NMR data obtained in toluene-d_8 at 25.2 MHz, 20 °C: $\delta(CH_2Si\mathit{Me}_3) = 2.2$, $\delta(CSi\mathit{Me}_3) = 0.06$, $\delta(\mathit{C}H_2SiMe_3)$ 68.8, $\delta(\mathit{C}SiMe_3) = 363.9$ (δ in ppm relative to Me_4Si).

$(Me_3SiCH_2)_3W{\equiv}CSiMe_3$. WCl_6 (22.2 g) was added in small portions over a period of $1^1/_2$ h to a solution of $LiCH_2SiMe_3$ (32 g, 6 equiv) in ether (200 mL)/hexane (200 mL) cooled to −78 °C with constant stirring. The solution was allowed to warm slowly to room temperature, whereupon the solvent was stripped. Pentane (200 mL) was added. The insoluble species were collected by filtration and discarded. The brown filtrate was stripped to give a dark brown sticky solid from which a yellow liquid distilled at 70 °C, 10^{-3} torr: $(Me_3SiCH_2)_3W{\equiv}CSiMe_3$ (4.7 g, 16% yield based on tungsten). Anal. Calcd for $WSi_4C_{16}H_{42}$: C, 36.21; H, 7.97. Found: C, 36.90; H, 8.15. ^1H NMR data obtained in toluene-d_8 at 220 MHz, 16 °C: $\delta(CH_2Si\mathit{Me}_3) = 0.19$ (9 H); $\delta(CSi\mathit{Me}_3) = 0.43$ (3 H); $\delta(C\mathit{H}_2SiMe_3) = 0.71$ (2 H), $^2J(^{183}W\text{–}H) = 10.2$ Hz (δ in ppm relative to Me_4Si). ^{13}C NMR data obtained in toluene-d_8 at 25.2 MHz, 20 °C: $\delta(CH_2Si\mathit{Me}_3) = 2.1$; $\delta(CSi\mathit{Me}_3) = -0.1$; $\delta(\mathit{C}H_2SiMe_3) = 56.2$, $J(^{183}W\text{–}^{13}C) = 81$ Hz; $\delta(\mathit{C}SiMe_3) = 344.6$ (δ in ppm relative to Me_4Si).

$Mo_2(CH_2SiMe_3)_6$. $MoCl_3$ (21.0 g) was added in small portions over a period of $^1/_2$ h to a solution of $LiCH_2SiMe_3$ (28 g, 3 equiv) in ether (100 mL)/hexane (200 mL), cooled to 0 °C with constant stirring. The solution was warmed to room temperature and the solvent stripped. Extraction with hexane (200 mL) gave a dark brown solution, which was stripped to a dark solid residue. Sublimation at 120 °C, 10^{-3} torr, gave orange crystals of $Mo_2(CH_2SiMe_3)_6$ (5.53 g, 25% based on molybdenum).

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Registry No. $(Me_3SiCH_2)_3Mo{=}CHSiMe_3$, 78638-60-5; $(Me_3SiCH_2)_3Mo{\equiv}CSiMe_3$, 78638-61-6; $Mo_2(CH_2SiMe_3)_6$, 34439-17-3; $(Me_3SiCH_2)_3W{\equiv}CSiMe_3$, 78638-62-7; $MoCl_5$, 10241-05-1; WCl_6, 13283-01-7; $MoCl_3$, 13478-18-7; $LiCH_2SiMe_3$, 1822-00-0.

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