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The Tungsten–Tungsten Triple Bond. 4.[1] Structural Characterization of Hexakis(trimethylsilylmethyl)ditungsten and Preparation of Bis-μ-(trimethylsilylmethylidyne)-tetrakis(trimethylsilylmethyl)ditungsten

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The reactions of the trimethylsilylmethyl Grignard reagent with WCl_4 and WCl_6 lead respectively to $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$, **1**, and $W_2(CH_2SiMe_3)_6$, **2**. Compound **1** is an air-sensitive, red-brown solid, extremely soluble, volatile at 120 °C under vacuum, and difficult to crystallize. A structure involving Me_3SiC bridges and a W–W bond is proposed but not proved. Compound **2** was reported earlier by Wilkinson and co-workers. It has now been structurally characterized by x-ray crystallography: space group $P2_1$, $a = 12.890$ (1) Å, $b = 18.546$ (2) Å, $c = 18.347$ (2) Å, $\beta = 109.829$ (8)°, $V = 4126.1$ (8) Å^3, $Z = 4$. The two crystallographically independent molecules are essentially identical. On the basis of refinement to $R_1 = 0.066$ and $R_2 = 0.083$, using 3136 reflections having $I > 3\sigma(I)$ collected with Mo Kα radiation and treating only the tungsten and silicon atoms anisotropically, the following average molecular dimensions were found: W–W = 2.255 Å, W–C = 2.14 Å, Si–C = 2.89 Å, W–W–C = 101.7°. The W_2C_6 skeleton has virtual D_{3d} symmetry. The W–W bond in this compound is the shortest one reported to date and is believed to be of order 3.

Introduction

Though molybdenum and rhenium have provided an abundance of compounds with homonuclear metal-to-metal bonds of high order, tungsten has been much less prolific in this respect.[3] Up to this time no compound with a W–W bond of order 4 has been characterized definitively,[4] although genuine $W_2(O_2CR)_4$ compounds appear to exist in noncrystalline form,[5] despite the fact that such bonds are found in scores of molybdenum compounds.[3] In previous papers we described[1,6,7] the preparation and full structural characterization of several compounds containing unbridged W–W triple bonds, the principal ones being $W_2(NMe_2)_6$, $W_2Cl_2(NEt_2)_4$, and $W_2Me_2(NEt_2)_4$. Prior to that, the only compound reported[8,9] which seemed likely to contain an unbridged W–W triple bond was $W_2(CH_2SiMe_2)_6$. However, this had not been structurally characterized beyond the statement that it is isomorphous with the dimolybdenum analogue, $Mo_2(CH_2SiMe_3)_6$, which had been shown to have an Mo_2C_6 skeleton with D_{3d} symmetry and a Mo–Mo bond length (2.167 Å) consistent with the presence of a triple bond.

In this paper we report the full structural characterization of $W_2(CH_2SiMe_3)_6$ and thereby make possible a comparison of M–M triple bonds in the four related compounds M_2L_6, where M = Mo and W and L = CH_2SiMe_3 and NMe_2. We also report the preparation of a new compound which, on the basis of noncrystallographic evidence, we formulate as bis-μ-(trimethylsilylmethylidyne)-tetrakis(trimethylsilylmethyl)ditungsten, $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$.

Experimental Section

Materials. WCl_6 and $TaCl_5$ were purchased from ROC/RIC and stored in glass ampules (ca. 5–40 g each) under vacuum until needed. $W(CO)_6$ was obtained from Strem Chemical Co. WCl_4 was prepared from the reaction $2WCl_6 + W(CO)_6 \rightarrow 3WCl_4$ in refluxing chlorobenzene and then stored in glass vials under N_2 until needed. Chloromethyltrimethylsilane was purchased from Silar Laboratories, Inc., and stored over molecular sieves until used. The Grignard reagent, Me_3SiCH_2MgCl, was prepared in ether (100% yield assumed) and used immediately. Solvents (pentane, hexane, benzene, toluene, ether, and THF) were dried and freed from dissolved molecular oxygen by distilling from a solution of the solvent, benzophenone, sodium, and phenyl ether (Ph_2O was not added to ether or THF), then stored over CaH_2 under nitrogen until used.

Spectroscopic and Analytical Measurements. Elemental analyses were performed by Alfred Bernhardt Mikroanalytisches Laboratorium, Elbach, West Germany, using drybox sampling techniques.

Infrared spectra were obtained from Nujol mulls between CsI plates using a Beckman IR-12 spectrophotometer.

Mass spectra were obtained using an AEI MS9 mass spectrometer and the direct insertion method (100–120 °C).

^1H and ^{13}C NMR measurements were made on Varian Associates A-60 and CFT20 instruments, respectively. Toluene-d_8 was used as the solvent and chemical shifts are reported as ppm downfield from hexamethyldisiloxane (HMDS) (for ^1H NMR data) or as ppm downfield from TMS (^{13}C NMR data).

Table I. ^{1}H and ^{13}C NMR Data for $W_2(CH_2SiMe_3)_6$, $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$, and $Ta_2(CSiMe_3)_2(CH_2SiMe_3)_4$

	$W_2(CSi-Me_3)_2(CH_2-SiMe_3)_4$	$Ta_2(CSi-Me_3)_2(CH_2-SiMe_3)_4$	$W_2(CH_2SiMe_3)_6$
$\delta(CH_2Si-(CH_3)_3)^a$	−0.10 ($J_{183_{W-H}}$ = 7.4 Hz)	0.66	1.74 ($J_{183_{W-H}}$ = 10 Hz)
$\delta(CH_2Si-(CH_3)_3)^a$	0.03	0.25	0.13
$\delta(CSi-(CH_3)_3)^a$	0.28	−0.11	
$\delta(CH_2Si-(CH_3)_3)^b$	68.9 ($J_{183_{W-C}}$ = 74 Hz)	78.9	77.1 ($J_{183_{W-C}}$ = 78 Hz)
$\delta(CH_2Si-(CH_3)_3)^b$	3.0	3.6	2.6
$\delta(CSi-(CH_3)_3)^b$	352.6 ($J_{183_{W-H}}$ = 73 Hz)	404.0	
$\delta(CSi-(CH_3)_3)^b$	2.25	4.9	

^{a} $\delta(^1$H) in ppm relative to hexamethyldisiloxane at 60 MHz, in toluene-d_8. ^{b} $\delta(^{13}$C) in ppm relative to tetramethylsilane at 20 MHz, in toluene-d_8.

Cryoscopic molecular weight determinations were carried out in a jacketed flask using a thermistor and a Wheatstone bridge circuit to monitor the temperature.

General Preparative Procedures. All preparations and other operations were carried out under a dry and oxygen-free nitrogen atmosphere or in vacuo, using standard Schlenk-type techniques.[10] After preparation, the samples were stored and handled in a Vacuum Atmospheres Co. DriLab system.

Preparation of $W_2(CH_2SiMe_3)_4(CSiMe_3)_2$. Me$_3$SiCH$_2$MgCl (103 mmol) in ether (95 ml) was added rapidly to WCl$_4$ (8.41 g, 25.8 mmol) in ether (150 ml), with stirring, at room temperature (water bath). After 30 min very little reaction had occurred so THF (100 ml) was added. The slurry was stirred (4 h) at 25 °C and then refluxed (3 h) yielding a dark brown solution and a copious light brown precipitate. The solvent was stripped (30–40 °C), and the residue was dried thoroughly (50 °C (10^{-3} Torr)) and then extracted with warm hexane (200 ml). The solvent was removed from the filtrate and the residue was purified by sublimation (140 °C (10^{-3} Torr)) to yield ca. 3 g of a reddish brown crystalline solid. Anal. Calcd for W_2-(CSiMe$_3$)$_2$(CH$_2$SiMe$_3$)$_4$: C, 32.50; H, 7.05; Cl, 0.00. Found: C, 32.4; H, 6.91; Cl, 0.00. A cryoscopic molecular weight determination in benzene gave 899 ± 20; calcd mol wt for $W_2(CH_2SiMe_3)_4$-(CSiMe$_3$)$_2$ 886. NMR data (^{1}H and ^{13}C) are reported in Table I.

Infrared data (Nujol mull between CsI plates): 273 (m), 341 (m), 367 (w), 428 (w), 464 (m), 489 (w), 596 (s), 622 (s), 686 (s), 698 (s), 710 (s), 738 (sh), 750 (s), 840 (vvs, vvbr), 893 (sh), 928 (vs), 962 (sh), 1014 (vs), 1152 (vw), 1170 (vw), 1245 (vs), 1259 (s), 1281 (w), 1306 (m) cm^{-1}.

$W_2(CH_2SiMe_3)_4(CSiMe_3)_2$ has a strong peak in its mass spectrum corresponding to the molecular ion (see Figure 1).

Preparation of $W_2(CH_2SiMe_3)_6$. $W_2(CH_2SiMe_3)_6$ was prepared as reported in the literature.[8] Anal. Calcd for $W_2(CH_2SiMe_3)_6$: C, 32.35; H, 7.47. Found: C, 32.4; H, 7.36.

Infrared data (Nujol mull between CsI plates): 233 (w), 248 (w), 278 (m), 458 (sh), 550 (m), 561 (sh), 617 (m), 683 (vs), 695 (vs), 833 (vvs, vvbr), 888 (vs), 938 (vs), 955 (vs), 1015 (w), 1102 (vw), 1157 (vw), 1170 (vw), 1195 (vw), 1246 (vs), 1259 (s), 1304 (w), 1318 (m) cm^{-1}.

Preparation of $Ta(CH_2SiMe_3)_4(CSiMe_3)_2$. $Ta_2(CH_2SiMe_3)_4$-(CSiMe$_3$)$_2$ was prepared as reported in the literature[11,12] and recrystallized from pentane. ^{1}H and ^{13}C NMR data are reported in Table I.

Infrared data (Nujol mull between CsI plates): 267 (m), 312 (m), 373 (m), 407 (m), 445 (m), 472 (m), 496 (m), 612 (m), 622 (m), 675 (sh), 700 (s), 725 (m), 750 (m), 782 (s), 830 (vvs, vvbr), 893 (m), 912 (sh), 933 (vs), 1020 (w), 1088 (w), 1102 (w), 1152 (w), 1170 (w), 1244 (vs), 1257 (m), 1293 (m), 1308 (m) cm^{-1}.

Crystal Selection and Data Collection. Dark reddish brown single crystals, which had been stored in an evacuated sealed tube, were immersed in heavy mineral oil in a nitrogen-filled glovebag for examination under a microscope. A nearly equidimensional crystal, 0.30 × 0.35 × 0.40 mm, was selected for data collection and sealed in a
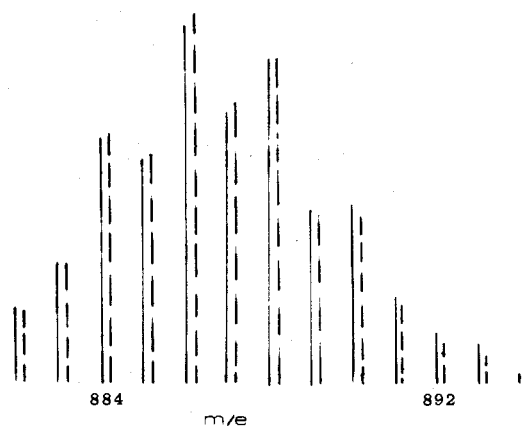

Figure 1. Observed (full lines) and calculated (broken lines, slightly offset) m/e pattern for the parent ion peak in the mass spectrum of $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$.

capillary filled with mineral oil. The crystal appeared to be of good quality based on ω scans for several intense reflections, which had peak widths at half-height <0.20°. General procedures for crystal examination and characterization had been previously reported.[13] The crystal was found to be monoclinic, displaying $2/m$ Laue symmetry. A small shell of data, collected to obtain reflections for accurate crystal centering, revealed systematically absent reflections $0k0$ ($k = 2n + 1$), indicating the space group to be either $P2_1$ (noncentrosymmetric) or $P2_1/m$ (centrosymmetric). The correct space group was subsequently shown to be $P2_1$. The final lattice constants[14] and orientation matrix, used for calculation of the setting angles in data collection, as determined at 22 ± 2 °C by least-squares refinement of the setting angles for 15 high-angle reflections (21.1° < 2θ(Mo Kα) ≤ 34.0° and $\lambda_{MoK\bar\alpha}$ 0.710 73 Å), are a = 12.890 (1) Å, b = 18.546 (2) Å, c = 18.347 (2) Å, β = 109.829 (8)°, and V = 4126.1 (8) Å^3. The observed volume of 4126.1 Å^3 is consistent with that anticipated for $Z = 4$, thus requiring no imposed crystallographic symmetry on the molecules in either $P2_1$ or $P2_1/m$. However, in the acentric space group, $P2_1$, the asymmetric unit must consist of two complete molecules.

Data were collected with Mo Kα radiation at room temperature, 22 ± 2 °C, using a Syntex $P\bar1$ autodiffractometer, equipped with a graphite-crystal monochromator. Intensities were recorded using variable scan rates from 4.0 to 24.0°/min for θ–2θ scans ranging from MoKα_1 − 1.1° to MoKα_2 + 1.0°. A total of 4253 unique reflections having 2θ(Mo Kα) ≤ 40.0° were recorded. During data collection, the three standard reflections, measured every 100 reflections, showed an average drop in intensity of approximately 46%. Further data collection using this crystal was judged to be unjustified. Since the crystal decomposition was essentially linear, a linear correction as a function of reflection number was applied to all data. The data were also corrected for absorption effects, μ = 60.83 cm^{-1}. The minimum and maximum transmission factors are 0.040 and 0.338 with the average absorption correction being 0.218. After corrections for decomposition, absorption, Lorentz, and polarization effects, the data were reduced to a set of relative $|F_o|^2$, and the 1117 reflections having $I < 3\sigma(I)$ were rejected as being unobserved.

Structural Solution and Refinement. The statistical indicators from the computer program FAME[15] clearly indicated the correct space group to be noncentric. Space group $P2_1$ was also supported by the three-dimensional Patterson map, which was devoid of the anticipated Harker vectors, 0, $^1/_2 + 2y$, 0, if the correct space group were $P2_1/m$. The structure was solved and refined in space group $P2_1$ with the asymmetric unit consisting of two independent molecules. Atomic coordinates for two of the tungsten atoms, W(1) and W(2), were derived from the Patterson map and refined in two cycles of least-squares refinement to give discrepancy indices

$$R_1 = \Sigma \||F_o| - |F_c|\|/|F_o| = 0.419$$

$$R_2 = [\Sigma w(||F_o| - |F_c||)^2/\Sigma w|F_o|^2]^{1/2} = 0.490$$

The function minimized during all least-squares refinements was $\Sigma w(|F_o| - |F_c|)^2$, where the weighting factor, w, equals $4F_o^2/\sigma(F_o^2)^2$. A value of 0.07 was used for p in the calculation of σ.[13] The atomic scattering factors used were those of Cromer and Waber.[16] Anomalous

Table II. Refined Positional and Anisotropic Thermal Parameters[a,b] for the Tungsten and Silicon Atoms for Molecules I and II in $W_2[CH_2Si(CH_3)_3]_6$

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
W(1)	0.1455 (1)	0.0000 (0)	0.05221 (9)	0.0076 (1)	0.00310 (6)	0.00249 (5)	−0.0002 (2)	0.0033 (1)	−0.0002 (1)
W(2)	0.3005 (1)	0.0639 (1)	0.10989 (9)	0.0076 (1)	0.00287 (6)	0.00234 (5)	0.0003 (2)	0.0029 (1)	−0.0001 (1)
W(3)	0.6359 (1)	0.4884 (1)	0.37282 (9)	0.0077 (1)	0.00273 (6)	0.00238 (5)	−0.0002 (2)	0.0028 (1)	−0.0000 (1)
W(4)	0.7863 (1)	0.4196 (1)	0.42810 (9)	0.0078 (1)	0.00282 (6)	0.00237 (5)	−0.0001 (2)	0.0026 (1)	0.0003 (1)
Si(1)	−0.051 (1)	−0.0027 (9)	0.1446 (7)	0.010 (1)	0.0051 (6)	0.0042 (4)	0.002 (2)	0.006 (1)	−0.0004 (10)
Si(2)	0.189 (1)	−0.1830 (8)	0.0427 (8)	0.014 (1)	0.0033 (5)	0.0048 (5)	0.001 (1)	0.006 (1)	−0.0010 (9)
Si(3)	0.001 (1)	0.0553 (8)	−0.1372 (7)	0.011 (1)	0.0042 (5)	0.0031 (4)	0.001 (1)	0.002 (1)	−0.0008 (9)
Si(4)	0.293 (1)	0.1763 (8)	0.2602 (6)	0.011 (1)	0.0045 (6)	0.0032 (4)	−0.003 (1)	0.004 (1)	−0.0016 (8)
Si(5)	0.379 (1)	0.1777 (8)	−0.0142 (7)	0.011 (1)	0.0046 (6)	0.0038 (4)	0.000 (1)	0.006 (1)	−0.0006 (9)
Si(6)	0.540 (1)	−0.0357 (8)	0.1921 (8)	0.009 (1)	0.0035 (5)	0.0048 (5)	0.002 (1)	0.001 (1)	0.0009 (9)
Si(7)	0.441 (1)	0.4884 (8)	0.4665 (6)	0.010 (1)	0.0050 (6)	0.0034 (4)	0.001 (1)	0.005 (1)	−0.0013 (9)
Si(8)	0.468 (1)	0.4442 (7)	0.1875 (7)	0.009 (1)	0.0040 (6)	0.0031 (4)	−0.000 (1)	0.003 (1)	−0.0009 (8)
Si(9)	0.698 (1)	0.6708 (8)	0.3568 (8)	0.012 (1)	0.0031 (5)	0.0049 (5)	0.001 (1)	0.003 (1)	0.0009 (9)
Si(10)	0.033 (1)	0.5110 (8)	0.5140 (8)	0.010 (1)	0.0038 (5)	0.0047 (5)	0.000 (1)	0.002 (1)	0.0001 (10)
Si(11)	0.859 (1)	0.3087 (7)	0.3017 (7)	0.012 (1)	0.0032 (5)	0.0038 (4)	0.000 (1)	0.005 (1)	−0.0013 (8)
Si(12)	0.770 (1)	0.3100 (8)	0.5764 (7)	0.012 (1)	0.0048 (6)	0.0032 (4)	0.000 (1)	0.005 (1)	0.0030 (8)

[a] Figures in parentheses are the estimated standard deviations in the least significant figures. [b] The form of the anisotropic thermal parameter is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

dispersion effects[17] were included in the calculated scattering factors for tungsten and silicon.

A difference Fourier map at this point revealed the positions of the remaining two tungsten atoms. The positional and isotropic thermal parameters for the four tungsten atoms were refined to give $R_1 = 0.194$ and $R_2 = 0.275$. From a series of three difference Fourier maps, each following the least-squares refinement of the atoms located up to that point, the remaining 60 atoms composing the asymmetric unit were located. The 64 unique atoms were refined in three cycles of full-matrix least-squares refinement employing first isotropic thermal parameters for all atoms to give $R_1 = 0.091$ and $R_2 = 0.113$ and then employing anisotropic thermal parameters for the 4 tungsten atoms and the 12 silicon atoms to give final values of 0.066 and 0.083 for R_1 and R_2, respectively, for the 3136 reflections having $I > 3\sigma(I)$.

In view of the limited data set, $2\theta \leq 40.0°$, the extensive crystal decomposition during data collection, the large corrections for absorption effects, and the costs of refinement of such a large asymmetric unit, refinement was terminated without attempting to refine anisotropic thermal parameters for the 48 carbon atoms. No attempt was made to determine the absolute configuration of the molecules, since the data did not merit such analysis. During the final cycle of refinement no parameter shifted by more than 0.30σ, where σ is the estimated standard deviation of that parameter, and the average parameter shift was less than 0.05σ. A final difference Fourier map showed no features of structural significance. The choice of the acentric space group $P2_1$ was also supported by the fact that in the final cycle of least-squares refinement only one correlation coefficient was observed to be greater than 0.5.

A table of observed and calculated structure factors for the data used in the final refinement is available elsewhere.[18]

Results and Discussion

Synthetic Considerations. In our previous study[6] of the reactions between tungsten halides and LiNMe₂ we found that $W(NMe_2)_6$, $W_2(NMe_2)_6$, or mixtures of these dimethylamides were obtained depending on the choice of the tungsten halide. For example, the reaction between WCl_6 and LiNMe₂ led to a mixture of $W(NMe_2)_6$ and $W_2(NMe_2)_6$ while reactions involving WBr_5 and WCl_4 led to the isolation of only pure $W(NMe_2)_6$ and $W_2(NMe_2)_6$, respectively. Wilkinson and his co-workers[8,9] reported the preparation of W_2R_6, where R = CH_2SiMe_3 and CH_2CMe_3, from the reaction between WCl_6 and RMgCl. On the basis of our previous experience in metathetic reactions involving tungsten halides, we thought it might be productive to react WCl_4 with 4 equiv of Me_3SiCH_2MgCl with the objective of obtaining W_2-$(CH_2SiMe_3)_6$ in high yield. To our surprise the alkane-soluble product from these reactions did not appear to consist of, nor to contain any, $W_2(CH_2SiMe_3)_6$, irrespective of the mode of addition of the reactants. We have been unable to obtain a crystalline product from these alkane extractions although a thermally stable, volatile, red-brown solid, which we formulate

as $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$ (see later), sublimes at 120 °C and 10^{-4} cmHg. This compound is exceedingly soluble in hydrocarbon solvents and many attempts to crystallize it from solution failed. However, by slow evaporation of pentane at −78 °C we have obtained $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$ as a rust-colored, crystalline solid, but, thus far, no crystals suitable for single-crystal x-ray studies have been obtained.

By following the procedure described by Wilkinson and his co-workers,[8] employing WCl_6 and Me_3SiCH_2MgCl (6 equiv), we isolated $W_2(CH_2SiMe_3)_6$ as an orange crystalline compound. ^1H NMR spectra of the crude alkane extractions from the reaction between WCl_6 and Me_3SiCH_2MgCl (6 equiv) suggest the presence of a mixture of both $W_2(CH_2SiMe_3)_6$ and $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$. The former is, however, readily isolated because it crystallizes easily and is stable during column chromatography employing cellulose.

Previously in the reactions of niobium and tantalum pentachlorides with alkylmagnesium reagents the compounds $M_2(CSiMe_3)_2(CH_2SiMe_3)_4$, where M = Nb and Ta,[11,12] and $Ta(CHCMe_3)(CH_2CMe_3)_3$[19] have been isolated. Thus it seemed possible that reactions involving WCl_4 and Me_3CCH_2MgCl might lead to $W_2(CCMe_3)_2(CH_2CMe_3)_4$ or some alkylidene compound of tungsten (i.e., a compound containing the W–CHCMe₃ moiety). In our present study this has not proved to be the case: only small amounts of $W_2(CH_2CMe_3)_6$ have been isolated. This is not to say that alkylidene compounds of tungsten are not formed in this reaction—merely that we have not detected them. There does, however, appear to be a significant difference between the neopentyl and trimethylsilylmethyl ligands as previously noted in the chemistry of niobium and tantalum peralkyls.[11,19,20] Finally, we note that it is not clear why reactions involving WCl_4 should lead to $W_2(CSiMe_3)_2(CH_2SiMe_3)_4$ when reactions involving WCl_6 lead to $W_2(CH_2SiMe_3)_6$.

$W_2(CSiMe_3)_2(CH_2SiMe_3)_4$. This new compound[21] is a red-brown, extremely air-sensitive solid which is exceedingly soluble in hydrocarbon solvents. A cryoscopic molecular weight determination in benzene showed it to be a dinuclear species. It is volatile and readily sublimes at 120 °C and 10^{-4} cmHg. Its mass spectrum has a parent ion peak corresponding to $W_2(CSiMe_3)_2(CH_2SiMe_3)_4^+$ together with peaks for many other W_2^+-containing ions. The observed vs. calculated m/e distribution for the $W_2(CSiMe_3)_2(CH_2SiMe_3)_4^+$ ion is shown in Figure 1. This is considerably different from simple W_2^+ patterns in $W_2(NMe_2)_6^+$ due to the presence of six silicon atoms. It is interesting to note that $W_2(CH_2SiMe_3)_6$ does not give a simple molecular ion in the mass spectrometer but rather

Table III. Refined Positional and Isotropic Thermal Parameters[a] for the Carbon Atoms in Molecules I and II in $W_2[CH_2Si(CH_3)_3]_6$

Atom	x	y	z	B_{iso}, Å²
C(1)	0.081 (3)	−0.022 (3)	0.144 (2)	4.5 (11)
C(2)	0.208 (3)	−0.088 (2)	0.010 (2)	4.6 (9)
C(3)	0.044 (3)	0.080 (2)	−0.030 (2)	4.2 (10)
C(4)	0.237 (3)	0.155 (3)	0.154 (2)	4.7 (11)
C(5)	0.362 (4)	0.087 (3)	0.016 (3)	6.8 (13)
C(6)	0.389 (4)	−0.006 (3)	0.194 (3)	5.6 (12)
C(1,1)	−0.058 (3)	0.099 (2)	0.172 (2)	5.0 (11)
C(1,2)	−0.169 (4)	−0.024 (3)	0.049 (3)	7.7 (14)
C(1,3)	−0.092 (5)	−0.062 (3)	0.210 (3)	9.3 (16)
C(2,1)	0.036 (4)	−0.204 (3)	0.017 (3)	8.7 (16)
C(2,2)	0.249 (4)	−0.250 (3)	−0.006 (3)	7.4 (13)
C(2,3)	0.273 (4)	−0.196 (3)	0.151 (3)	7.6 (14)
C(3,1)	−0.088 (4)	−0.031 (3)	−0.157 (3)	7.7 (14)
C(3,2)	0.116 (4)	0.045 (3)	−0.176 (3)	7.3 (13)
C(3,3)	−0.085 (4)	0.127 (3)	−0.191 (3)	8.0 (15)
C(4,1)	0.444 (4)	0.190 (3)	0.296 (2)	6.3 (11)
C(4,2)	0.230 (4)	0.266 (3)	0.279 (3)	7.4 (13)
C(4,3)	0.262 (4)	0.104 (3)	0.319 (3)	5.5 (12)
C(5,1)	0.255 (3)	0.236 (2)	−0.046 (2)	4.9 (10)
C(5,2)	0.486 (4)	0.226 (3)	0.074 (3)	7.8 (14)
C(5,3)	0.439 (4)	0.168 (3)	−0.093 (3)	7.9 (15)
C(6,1)	0.526 (4)	−0.096 (3)	0.098 (3)	6.8 (13)
C(6,2)	0.610 (5)	−0.095 (4)	0.277 (3)	10.5 (19)
C(6,3)	0.629 (4)	0.051 (3)	0.198 (3)	8.1 (15)
C(7)	0.580 (3)	0.517 (2)	0.465 (2)	4.4 (9)
C(8)	0.532 (3)	0.413 (3)	0.287 (2)	3.8 (10)
C(9)	0.717 (3)	0.576 (2)	0.329 (2)	3.5 (7)
C(10)	0.894 (3)	0.490 (2)	0.512 (2)	5.1 (8)
C(11)	0.843 (3)	0.404 (2)	0.335 (2)	4.2 (8)
C(12)	0.709 (4)	0.333 (3)	0.468 (3)	5.3 (13)
C(7,1)	0.424 (4)	0.392 (3)	0.478 (3)	8.6 (15)
C(7,2)	0.331 (4)	0.513 (3)	0.374 (3)	6.4 (12)
C(7,3)	0.417 (4)	0.542 (3)	0.550 (3)	6.5 (13)
C(8,1)	0.401 (3)	0.540 (2)	0.179 (2)	4.5 (10)
C(8,2)	0.567 (3)	0.443 (2)	0.137 (2)	4.8 (10)
C(8,3)	0.359 (4)	0.381 (3)	0.135 (3)	6.7 (13)
C(9,1)	0.548 (4)	0.704 (3)	0.328 (2)	6.4 (12)
C(9,2)	0.773 (4)	0.727 (3)	0.309 (3)	7.1 (14)
C(9,3)	0.769 (4)	0.687 (3)	0.465 (3)	6.8 (13)
C(10,1)	0.118 (5)	0.419 (4)	0.515 (3)	9.2 (16)
C(10,2)	0.119 (5)	0.553 (4)	0.603 (3)	9.7 (17)
C(10,3)	0.039 (4)	0.567 (3)	0.430 (3)	7.6 (13)
C(11,1)	0.975 (4)	0.264 (3)	0.382 (3)	6.6 (12)
C(11,2)	0.726 (4)	0.249 (3)	0.273 (3)	6.1 (12)
C(11,3)	0.917 (4)	0.322 (3)	0.217 (3)	7.8 (14)
C(12,1)	0.735 (4)	0.383 (3)	0.635 (3)	7.2 (13)
C(12,2)	0.926 (4)	0.284 (3)	0.602 (3)	7.8 (15)
C(12,3)	0.696 (5)	0.228 (3)	0.590 (3)	8.8 (16)

[a] Figures in parenthese are the estimated standard deviations in the least significant figures.

Table IV. Bond Distances (Å)[a] within Molecules I and II in $W_2[CH_2Si(CH_3)_3]_6$

Molecule I		Molecule II	
W(1)–W(2)	2.255 (2)	W(3)–W(4)	2.254 (2)
W(1)–C(1)	2.16 (4)	W(3)–C(7)	2.11 (3)
−C(2)	2.09 (4)	−C(8)	2.19 (3)
−C(3)	2.20 (3)	−C(9)	2.23 (3)
W(2)–C(4)	2.15 (4)	W(4)–C(10)	2.14 (4)
−C(5)	2.18 (4)	−C(11)	2.10 (3)
−C(6)	2.03 (4)	−C(12)	2.15 (4)
Si(1)–C(1)	1.84 (4)	Si(7)–C(7)	1.88 (3)
−C(1,1)	1.97 (4)	−C(7,1)	1.82 (5)
−C(1,2)	1.93 (4)	−C(7,2)	1.87 (4)
−C(1,3)	1.84 (5)	−C(7,3)	1.94 (4)
Si(2)–C(2)	1.90 (4)	Si(8)–C(8)	1.83 (3)
−C(2,1)	1.90 (4)	−C(8,1)	1.95 (4)
−C(2,2)	1.85 (4)	−C(8,2)	1.82 (3)
−C(2,3)	1.94 (4)	−C(8,3)	1.82 (4)
Si(3)–C(3)	1.92 (3)	Si(9)–C(9)	1.88 (4)
−C(3,1)	1.93 (5)	−C(9,1)	1.93 (4)
−C(3,2)	1.86 (4)	−C(9,2)	1.83 (4)
−C(3,3)	1.80 (5)	−C(9,3)	1.90 (4)
Si(4)–C(4)	1.88 (3)	Si(10)–C(10)	1.82 (4)
−C(4,1)	1.85 (4)	−C(10,1)	2.01 (6)
−C(4,2)	1.94 (5)	−C(10,2)	1.82 (5)
−C(4,3)	1.85 (4)	−C(10,3)	1.89 (5)
Si(5)–C(5)	1.80 (4)	Si(11)–C(11)	1.90 (4)
−C(5,1)	1.85 (4)	−C(11,1)	1.89 (4)
−C(5,2)	1.95 (5)	−C(11,2)	1.96 (4)
−C(5,3)	1.87 (5)	−C(11,3)	1.95 (5)
Si(6)–C(6)	2.04 (4)	Si(12)–C(12)	1.93 (4)
−C(6,1)	2.01 (4)	−C(12,1)	1.89 (5)
−C(6,2)	1.87 (6)	−C(12,2)	1.96 (5)
−C(6,3)	1.96 (5)	−C(12,3)	1.86 (5)

[a] Figures in parentheses are the estimated standard deviations in the least significant figures.

ligands. The methylene carbon atoms directly bonded to tungsten appear at δ_c 68.9 ppm and show coupling to tungsten-183 ($I = 1/2$, natural abundance 15%), $^1J_{183W-13C} = 74$ Hz. The carbyne-like carbon resonances occur at δ 352.6 ppm and also show coupling to tungsten-183, $^1J_{183W-13C} = 73$ Hz. It is interesting to note that the carbyne-like carbon atoms are even more deshielded than the carbyne carbon atoms in XW(CO)₄CPh (X = Cl, Br, I) and the carbene carbons in (CO)₅W(carbene) complexes.[20] All ¹H and ¹³C NMR data for W₂(CSiMe₃)₂(CH₂SiMe₃)₄ and for the related compounds W₂(CH₂SiMe₃)₆ and Ta(CSiMe₃)₂(CH₂SiMe₃)₄ are given in Table I.

We conclude that while W₂(CSiMe₃)₂(CH₂SiMe₃)₄ most likely has a structure similar to that found[12] for the related compounds M₂(CSiMe₃)₂(CH₂SiMe₃)₄, where M = Nb or Ta, some significant difference must arise from the presence of a metal–metal bond. In the Nb and Ta compounds it is not necessary to invoke metal to metal bonding, but for the tungsten compound at least a single bond is required. Further work is being directed toward a full structural characterization of this interesting compound.

Solid-State Structure of W₂(CH₂SiMe₃)₆. The compound is composed of discrete molecules of W₂(CH₂SiMe₃)₆ in the solid state. The crystal on which we collected data belongs to space group $P2_1$ with $Z = 4$, and the asymmetric unit consists of two complete molecules. There is no crystallographically imposed symmetry on either molecule, although with respect to the W₂C₆ skeletons, the molecules have virtual D_{3d} symmetry. A perspective drawing of molecule I illustrating the atom labeling scheme is shown in Figure 2; a similar illustration of molecule II is shown in Figure 3. Each atom in each molecule is uniquely labeled. Each of the methyl carbon atoms has two numbers; the first number is the same as that of the silicon atom to which it is bonded and the second

has a complex m/e distribution which is not readily simulated. This m/e pattern probably arises from the overlapping of the W₂(CH₂SiMe₃)₆⁺ pattern with those of ions derived by loss of one or more hydrogen atoms. It appears that some W₂-(CSiMe₃)₂(CH₂SiMe₃)₄ is formed in the mass spectrometer.

The principal peaks in the infrared spectrum of W₂-(CSiMe₃)₂(CH₂SiMe₃)₄ are listed in the Experimental Section. This spectrum is significantly different from that of W₂(CH₂SiMe₃)₆ but shows many similarities to those of M₂(CSiMe₃)₂(CH₂SiMe₃)₄ (M = Nb, Ta).

In the proton NMR spectrum there are two SiMe₃ resonances in the intensity ratio 2:1. The methylene protons of the CH₂SiMe₃ ligand and to a lesser extent the methyl protons of this ligand are significantly shielded relative to those in M₂(CH₂SiMe₃)₆ (M = Mo, W) and M₂(CSiMe₃)₂-(CH₂SiMe₃)₄ (M ≈ Nb, Ta). This presumably reflects the diamagnetic anisotropy of the W₂C₂ unit as in the case of the disparate chemical shifts for the proximal and distal alkyl groups in W₂(NR₂)₆.[6,7,22]

The carbon-13 NMR spectrum shows two types of methyl carbon atoms readily assignable to the CSiMe₃ and CH₂SiMe₃

Table V. Bond Angles (deg)^a within Molecules I and II in $W_2[CH_2Si(CH_3)_3]_6$

Molecule I		Molecule II	
W(2)-W(1)-C(1)	104.6 (9)	W(4)-W(3)-C(7)	104.9 (9)
-C(2)	101.1 (9)	-C(8)	100.5 (9)
-C(3)	101.1 (9)	-C(9)	97.9 (8)
W(1)-W(2)-C(4)	101.7 (9)	W(3)-W(4)-C(10)	103.2 (10)
-C(5)	103.5 (11)	-C(11)	101.3 (9)
-C(6)	101.6 (11)	-C(12)	98.6 (10)
C(1)-W(1)-C(2)	116 (1)	C(7)-W(3)-C(8)	116 (1)
-C(3)	111 (1)	-C(9)	116 (1)
C(2)-W(1)-C(3)	119 (1)	C(8)-W(3)-C(9)	117 (1)
C(4)-W(2)-C(5)	115 (4)	C(10)-W(4)-C(11)	111 (1)
-C(6)	114 (1)	-C(12)	119 (1)
C(5)-W(2)-C(6)	118 (1)	C(11)-W(4)-C(12)	120 (1)
C(1)-Si(1)-C(1,1)	110 (2)	C(7)-Si(7)-C(7,1)	116 (2)
-C(1,2)	115 (2)	-C(7,2)	110 (2)
-C(1,3)	112 (2)	-C(7,3)	106 (2)
C(1,1)-Si(1)-C(1,2)	110 (2)	C(7,1)-Si(7)-C(7,2)	105 (2)
-C(1,3)	111 (2)	-C(7,3)	110 (2)
C(1,2)-Si(1)-C(1,3)	99 (2)	C(7,2)-Si(7)-C(7,3)	109 (2)
C(2)-Si(2)-C(2,1)	111 (2)	C(8)-Si(8)-C(8,1)	114 (2)
-C(2,2)	110 (2)	-C(8,2)	110 (2)
-C(2,3)	110 (2)	-C(8,3)	109 (2)
C(2,1)-Si(2)-C(2,2)	108 (2)	C(8,1)-Si(8)-C(8,2)	110 (2)
-C(2,3)	114 (2)	-C(8,3)	107 (2)
C(2,2)-Si(2)-C(2,3)	104 (2)	C(8,2)-Si(8)-C(8,3)	106 (2)
C(3)-Si(3)-C(3,1)	110 (2)	C(9)-Si(9)-C(9,1)	116 (2)
-C(3,2)	115 (2)	-C(9,2)	105 (2)
-C(3,3)	107 (2)	-C(9,3)	112 (2)
C(3,1)-Si(3)-C(3,2)	110 (2)	C(9,1)-Si(9)-C(9,2)	109 (2)
-C(3,3)	107 (2)	-C(9,3)	109 (2)
C(3,2)-Si(3)-C(3,3)	107 (2)	C(9,2)-Si(9)-C(9,3)	105 (2)
C(4)-Si(4)-C(4,1)	112 (2)	C(10)-Si(10)-C(10,1)	110 (2)
-C(4,2)	109 (2)	-C(10,2)	114 (2)
-C(4,3)	110 (2)	-C(10,3)	115 (2)
C(4,1)-Si(4)-C(4,2)	106 (2)	C(10,1)-Si(10)-C(10,2)	101 (2)
-C(4,3)	107 (2)	-C(10,3)	108 (2)
C(4,2)-Si(4)-C(4,3)	110 (2)	C(10,2)-Si(10)-C(10,3)	109 (2)
C(5)-Si(5)-C(5,1)	117 (2)	C(11)-Si(11)-C(11,1)	107 (2)
-C(5,2)	108 (2)	-C(11,2)	115 (2)
-C(5,3)	106 (2)	-C(11,3)	105 (2)
C(5,1)-Si(5)-C(5,2)	107 (2)	C(11,1)-Si(11)-C(11,2)	110 (2)
-C(5,3)	111 (2)	-C(11,3)	105 (2)
C(5,2)-Si(5)-C(5,3)	109 (2)	C(11,2)-Si(11)-C(11,3)	113 (2)
C(6)-Si(6)-C(6,1)	111 (2)	C(12)-Si(12)-C(12,1)	110 (2)
-C(6,2)	110 (2)	-C(12,2)	110 (2)
-C(6,3)	109 (2)	-C(12,3)	105 (2)
C(6,1)-Si(6)-C(6,2)	106 (2)	C(12,1)-Si(12)-C(12,2)	118 (2)
-C(6,3)	113 (2)	-C(12,3)	106 (2)
C(6,2)-Si(6)-C(6,3)	109 (2)	C(12,2)-Si(12)-C(12,3)	107 (2)
W(1)-C(1)-Si(1)	127 (2)	W(3)-C(7)-Si(7)	122 (2)
-C(2)-Si(2)	120 (2)	-C(8)-Si(8)	118 (2)
-C(3)-Si(3)	116 (2)	-C(9)-Si(9)	117 (2)
-C(4)-Si(4)	120 (2)	-C(10)-Si(10)	123 (2)
-C(5)-Si(5)	123 (2)	-C(11)-Si(11)	120 (2)
-C(6)-Si(6)	117 (2)	-C(12)-Si(12)	116 (2)

^{a} Figures in parentheses are the estimated standard deviations in the least significant figures.

number differentiates between methyl carbon atoms bonded to the same silicon atom. The final atomic coordinates and anisotropic thermal parameters for the tungsten and silicon atoms are given in Table II. The refined atomic coordinates and isotropic thermal parameters for all carbon atoms are given in Table III.

Values for the bond distances within molecules I and II are given in Table IV. Bond angles within both molecules are reported in Table V.

The most striking structural feature for $W_2(CH_2SiMe_3)_6$ in comparison to the previously reported compounds having tungsten-tungsten triple bonds is the markedly smaller tungsten-tungsten bond distance. The average of the W–W distances in the two independent molecules of $W_2(CH_2SiMe_3)_6$ is 2.255 (2) Å, which is 0.036–0.046 Å shorter than the W–W

distances for compounds having amide groups bonded to the tungsten atoms: $W_2(NMe_2)_6$,[6] 2.293 (1) Å; $W_2(NEt_2)_4Cl_2$,[7] 2.301 (1) Å; $W_2(NEt_2)_4Me_2$,[1] 2.291 (1) Å. This observed reduction in the W–W bond distance in the compound having six alkyl groups bonded to the tungsten atoms might be attributed either to lesser steric interactions in the hexaalkyl compound or to the absence of ligand lone-pair interactions in the tungsten–ligand bonds. The reality of W–N lone-pair interactions has been previously discussed,[1] and while the nitrogen lone-pair interactions would be bonding with respect to the W—N bonds, they might well have an antibonding effect on the W≡W bonds. In $W_2(NEt_2)_4Cl_2$, where there appear to be slightly greater W–N lone-pair interactions as compared to $W_2(NEt_2)_4Me_2$, there is a slight increase in the W≡W distance: 2.301 (1) Å for the dichloro compound
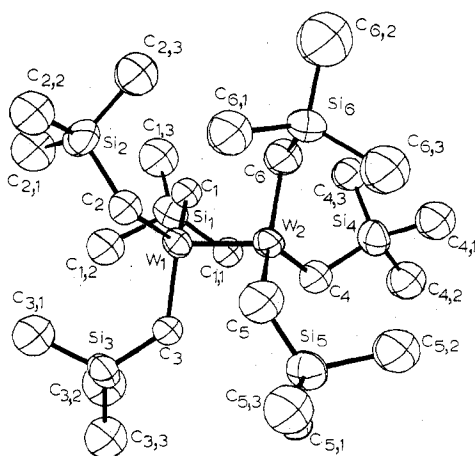
Figure 2. View of molecule I in $W_2(CH_2SiMe_3)_6$ showing 50% probability ellipsoids and the atomic labeling scheme.
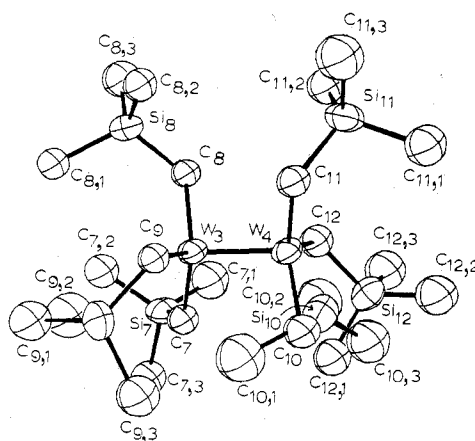

Figure 3. View of molecule II in $W_2(CH_2SiMe_3)_6$ showing 50% probability ellipsoids and the atomic labeling scheme.

compared to 2.291 (1) Å for the dimethyl compound. While steric factors may well play a role in the small variation of $W \equiv W$ distances, we believe that changes in the $N \rightarrow W$ π bonding have the predominant influence.

Because of the many sources of error mentioned in the Experimental Section, the other bond lengths and the various bond angles are relatively inaccurate, as shown both by their individual esd's and by the scatter in values of presumably chemically equivalent parameters. Nevertheless, the averaged values of these parameters are very reasonable. For example, the average of the 12 independent W–C distances is 2.14 Å which is in reasonably close agreement with the value of 2.171 Å in $W_2(NEt_2)_4Me_2$.[1] Similarly, the average of 48 independent Si–C distances is 1.89 Å as compared to the sum of covalent radii, 1.94 Å. The discrepancy is in the direction expected as a result of vibration of the lighter carbon atoms relative to the silicon atoms. The $W \equiv W$ distance we have found in $W_2(CH_2SiMe_3)_6$, 2.255 Å, is 0.088 Å greater than that reported in the analogous molybdenum compound,[9] 2.167

Å. This is in accord with the difference of ~0.08 Å between the $Mo \equiv Mo$[22] and $W \equiv W$[6] distances in the $M_2(NMe_2)_6$ compounds. Thus, the postulated $N \rightarrow M$ π bonding appears to have a similar effect for both W and Mo.

References and Notes

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