Journal of Organometallic Chemistry, 282 (1985) 239–246
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

CRYSTAL AND MOLECULAR STRUCTURE OF $W(\equiv CCMe_3)(PMe_3)_3Cl_3$; A SEVEN-COORDINATE TUNGSTEN(VI) ALKYLIDYNE COMPLEX WITH A REGULAR CAPPED OCTAHEDRAL (C_{3v}) STEREOCHEMISTRY

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(Received September 10th, 1984)

Summary

The complex $W(\equiv CCMe_3)(PMe_3)_3Cl_3$ crystallizes in the orthorhombic space group *Pbnm* (No. 62) with a 10.3147(10), b 14.7920(17), c 15.4266(22) Å, V 2353.7(5) Å^3 and D(calcd.) 1.66 g cm^{-3} for mol. wt. 587.6 and $Z = 4$. Diffractometer data were collected for four equivalent forms of reflections with $2\theta = 4.0–45.0°$ (Mo-K_α) and the structure was solved and refined to R_F 2.7% for all 1605 independent data (R_F 2.0% for those 1378 data with $|F_o| > 3.0\sigma(|F_o|)$). The molecule lies on a crystallographic mirror plane, which leads to disorder of the methyl groups of the neopentylidyne ligand. The tungsten atom has a monocapped octahedral coordination geometry (C_{3v} symmetry) with the neopentylidyne ligand in the capping position ($W\equiv C$ 1.793(6) Å), three PMe_3 ligands in the capped face and three Cl ligands in the opposite triangular face ($W-PMe_3$ 2.513(1)–2.519(2), W–Cl 2.542(1)–2.545(2) Å).

Introduction

We have previously determined the crystal structures of a number of alkylidyne complexes of tungsten including $W(\equiv CCMe_3)(=CHCMe_3)(CH_2CMe_3)(dmpe)$ [1,2], $W(\equiv CH \cdot AlMe_{2-x}Cl_{1+x})(PMe_3)_3Cl$ [3,4], $W(\equiv CH)(PMe_3)_4Cl$ [4], $W(\equiv C \cdot Al_2Me_4Cl)(PMe_3)_2(Me)(\eta^2\text{-}C_2H_4)$ [5], $W(\equiv CCMe_3)(PHPh)(PEt_3)_2Cl_2$ [6], $[(C_5Me_4(t\text{-}Bu))W(\equiv CCMe_3)I]_2(\mu\text{-}N_2H_2)$ [7] and $[W_2(\equiv CPMe_3)_2(PMe_3)_4Cl_4{}^{2+}][AlCl_4{}^-]_2$ [8]. We have now turned our attention to the seven-coordinate species $W(\equiv CCMe_3)(PMe_3)_3Cl_3$. The synthesis of this has previously been described by Schrock and coworkers [9]. Of particular interest is the observation that solutions of this complex in dichloromethane are conducting, presumably as a result of ionization (see eq. 1).

$$W(\equiv CCMe_3)(PMe_3)_3Cl_3 \rightleftarrows [W(\equiv CCMe_3)(PMe_3)_3Cl_2{}^+] + Cl^- \qquad (1)$$

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A structural study of the seven-coordinate species seemed necessary to determine its molecular stereochemistry and to see whether or not one chloride ligand was particularly weakly bound to tungsten.

Collection of X-ray diffraction data

A yellow crystal of approximate dimensions 0.27 × 0.23 × 0.17 mm^3 was selected for the structural analysis and was carefully inserted into a thin-walled glass capillary, which was flame sealed, set into an aluminum pin with beeswax, and mounted into a eucentric goniometer on our Syntex $P2_1$ automated four-circle diffractometer. Crystal alignment, determination of the crystal class (orthorhombic), the orientation matrix and accurate cell parameters and data collection (via the θ–2θ scan technique) were carried out as described previously [10]. Details appear in Table 1. The observed systematic absences [$h0l$ for $h + l = 2n + 1$, $0kl$ for $k = 2n + 1$ ($h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; $00l$, $l = 2n + 1$)] are consistent with two possible orthorhombic space groups, the centrosymmetric space group *Pbnm* (No. 62) and the noncentrosymmetric space group $Pbn2_1$ (No. 33). With $Z = 4$ either is possible and the asymmetric unit is one half molecule (sitting on a special position) in *Pbnm* or one entire molecule in $Pbn2_1$. We decided to collect four possible data forms (one half sphere i.e. $+h$, $\pm k$, $\pm l$). The successful solution of the structure indicated that the correct space group was *Pbnm*.

All data were corrected for Lorentz and polarization effects and for the effects of absorption (via interpolation both in 2θ and ϕ between a series of ψ-scans of close-to-axial reflections). The "*R*-factors" for averaging the four equivalent sets of

TABLE 1

EXPERIMENTAL DATA FOR THE X-RAY DIFFRACTION STUDY OF $W(\equiv CCMe_3)(PMe_3)_3Cl_3$

(A) Crystallographic parameters

Cryst. system: Orthorhombic

a 10.3147(10) Å
b 14.7920(17) Å
c 15.4266(22) Å
V 2353.7(5) $Å^3$

Space group: *Pbnm*
[nonstandard D_{2h}^{16}; No. 62]
Formula: $C_{14}H_{36}Cl_3P_3W$
Mol.wt.: 587.6
$Z = 4$
D(calcd) 1.66 g cm^{-3}
T 23°C (296 K)

(B) Data collection

Diffractometer; Syntex $P2_1$
Radiation: Mo-K_α (λ 0.710730 Å)
Monochromator: highly oriented graphite, equatorial geometry, $2\theta_m = 12.2°$, assumed 50% perfect
Reflection measd: $+h$, $\pm k$, $\pm l$ for $2\theta = 4.0$–$45.0°$
Scan type: coupled θ(crystal)–2θ(counter)
Scan width: symmetrical $[2.0 + \Delta(\alpha_2 - \alpha_1)]°$
Scan speed: 2.0 deg/min (in 2θ)
Background: stationary-crystal, stationary-counter at beginning and end of 2θ scan; each for one-half of total scan time
Standards: 3 check reflections remeasured after each 97 reflections; decay was corrected
Reflections collected: 6052 total, merged to 1605 independent reflctns. [File WPCL #173]
μ(Mo-K_α): 57.2 cm^{-1}; empirical correction applied

TABLE 2

ATOMIC COORDINATES FOR $W(\equiv CCMe_3)(PMe_3)_3Cl_3$

Atom	*x*	*y*	*z*	*B* (Å^2)
W	−0.07937(2)	0.21526(1)	0.25000(0)	
P(1)	0.04519(15)	0.23250(9)	0.11097(8)	
P(2)	−0.26961(20)	0.10843(12)	0.25000(0)	
Cl(1)	−0.23775(14)	0.27320(8)	0.13662(8)	
Cl(2)	0.02229(19)	0.37199(10)	0.25000(0)	
C(1)	0.00601(63)	0.10970(40)	0.25000(0)	
C(2)	0.07701(89)	0.01653(47)	0.25000(0)	
C(3)	0.0020(14)	−0.05520(74)	0.30489(89)	
C(4)	0.2107(13)	0.02813(83)	0.29842(86)	
C(5)	0.1010(16)	−0.01963(90)	0.33701(93)	
C(11)	0.00863(62)	0.14948(38)	0.02879(32)	
C(12)	0.21911(61)	0.22943(47)	0.12231(39)	
C(13)	0.02955(73)	0.33515(38)	0.04720(35)	
C(21)	−0.27972(63)	0.03524(36)	0.15599(36)	
C(22)	−0.43251(84)	0.15324(71)	0.25000(0)	
H(11)	0.0131	0.0906	0.0532	6.0
H(12)	0.0696	0.1543	−0.0170	6.0
H(13)	−0.0761	0.1597	0.0069	6.0
H(14)	0.2453	0.2719	0.1650	6.0
H(15)	0.2583	0.2442	0.0684	6.0
H(16)	0.2453	0.1705	0.1394	6.0
H(17)	−0.0594	0.3463	0.0355	6.0
H(18)	0.0751	0.3282	−0.0059	6.0
H(19)	0.0648	0.3845	0.0786	6.0
H(21)	−0.1987	0.0059	0.1473	6.0
H(22)	−0.3004	0.0702	0.1062	6.0
H(23)	−0.3452	−0.0088	0.1650	6.0
H(24)	−0.4929	0.1047	0.2500	6.0
H(25)	−0.4451	0.1892	0.1997	6.0
H(31)	−0.0804	−0.0659	0.2795	6.0
H(32)	−0.0091	−0.0332	0.3623	6.0
H(33)	0.0500	−0.1100	0.3062	6.0
H(41)	0.2689	0.0609	0.2626	6.0
H(42)	0.2461	−0.0297	0.3107	6.0
H(43)	0.1973	0.0601	0.3510	6.0
H(51)	0.0253	−0.0495	0.3573	6.0
H(52)	0.1222	0.0285	0.3752	6.0
H(53)	0.1709	−0.0613	0.3348	6.0

ANISOTROPIC THERMAL PARAMETERS FOR $W(\equiv CCMe_3)(PMe_3)_3Cl_3$ [a]

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
W	2.132(13)	2.071(11)	2.092(12)	−0.0340(94)	0.0000(0)	0.0000(0)
P(1)	3.992(69)	3.963(61)	2.665(52)	−0.602(48)	0.971(52)	−0.151(47)
P(2)	3.188(93)	3.476(77)	3.964(81)	−0.971(71)	0.0000(0)	0.0000(0)
Cl(1)	4.140(67)	4.576(61)	4.348(58)	0.254(52)	−1.601(58)	1.010(50)
Cl(2)	4.430(92)	2.592(68)	3.919(77)	−1.020(64)	0.0000(0)	0.0000(0)
C(1)	2.64(33)	3.10(28)	2.87(29)	−0.15(26)	0.0000(0)	0.0000(0)
C(2)	5.68(48)	3.16(31)	5.11(40)	1.93(33)	0.0000(0)	0.0000(0)
C(3)	6.18(79)	4.37(57)	8.01(80)	0.22(56)	0.15(68)	1.33(58)
C(4)	5.34(72)	6.15(65)	8.93(84)	2.58(56)	0.14(64)	0.05(60)
C(5)	7.3(10)	4.83(62)	8.04(87)	2.20(62)	−0.97(77)	1.65(61)

(continued)

TABLE 2 (continued)

ANISOTROPIC THERMAL PARAMETERS FOR $W(\equiv CCMe_3)(PMe_3)_3Cl_3$ [a]

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
C(11)	6.42(35)	6.38(29)	2.70(21)	−1.05(29)	1.12(22)	−1.03(23)
C(12)	3.60(31)	9.74(44)	6.04(32)	−1.29(29)	2.01(28)	−0.94(30)
C(13)	9.69(47)	5.64(30)	3.74(27)	−1.02(31)	1.47(30)	1.81(25)
C(21)	6.48(39)	5.36(29)	5.11(27)	−2.85(28)	−0.87(26)	−1.03(24)
C(22)	2.61(38)	7.13(53)	13.05(80)	−0.94(40)	0.0000(0)	0.0000(0)

[a] The anisotropic thermal parameters enter the equation for the calculated structure factor in the form: $\exp[-0.25(h^2a^{\star 2}B_{11} + \cdots + 2hka^{\star}b^{\star}B_{12} \cdots)]$.

reflections were $R(I)$ 3.10 and $R_w(I)$ 3.62%. A Wilson plot was used to place the 1605 unique reflections on an approximately absolute scale.

Solution and refinement of the structure

The position of the tungsten atom was quickly and unambiguously located from a Patterson map which indicated that the space group was probably *Pbnm*. All other non-hydrogen atoms were then located with use of difference-Fourier maps and full-matrix least-squares refinement routines. The function $\Sigma w(|F_o| - |F_c|)^2$ was minimized, where $w = \{[\sigma(|F_o|)]^2 + [0.015|F_o|]^2\}^{-1}$. All hydrogen atoms were included in fixed calculated positions on the assumption that d(C–H) 0.95 Å [11]. The analytical atomic scattering factors for neutral W, P, Cl and C were corrected for both the real ($\Delta f'$) and imaginary ($\Delta f''$) components of anomalous dispersion. With use of anisotropic thermal parameters for all non-hydrogen atoms the model converged with R_F 2.7, R_{wF} 2.4% and $GOF = 0.79$ for all 1605 data. The residuals using only those 1378 data with $|F_o| > 3\sigma(|F_o|)$ were R_F 2.0 and R_{wF} 2.3%.

An analysis of the function $\Sigma w(|F_o| - |F_o|)^2$ showed no unusual trends as a function of Miller indices, $|F_o|$, $(\sin\theta)/\lambda$ or sequence number. A final difference-Fourier synthesis was featureless. Final positional parameters and anisotropic thermal parameters are collected in Table 2. Data were corrected for secondary extinction using the simplified Zachariasen method:

$$|F_{o,\mathrm{corr}}| = |F_{o,\mathrm{uncorr}}|\ (1.0 + gI_o)$$

The value determined for g was 0.105×10^{-6}.

Description of the molecular structure

The crystal contains discrete monomeric units of $W(\equiv CCMe_3)(PMe_3)_3Cl_3$ which are mutually separated by normal Van der Waals' distances; there are no abnormally short intermolecular contacts. Each molecule lies on a crystallographic mirror plane; the methyl groups of the neopentylidyne ligand are disordered about the mirror plane. The overall molecular geometry and atomic labeling scheme is depicted in Fig. 1. Interatomic distances and angles are collected in Tables 3 and 4.

The central tungsten atom is in a formal oxidation state of +6 and is linked to three chloride ions, three PMe_3 ligands and a $CCMe_3{}^{3-}$ anion. The tungsten atom lies on a site of crystallographic mirror symmetry (along with atoms Cl(2), C(1),

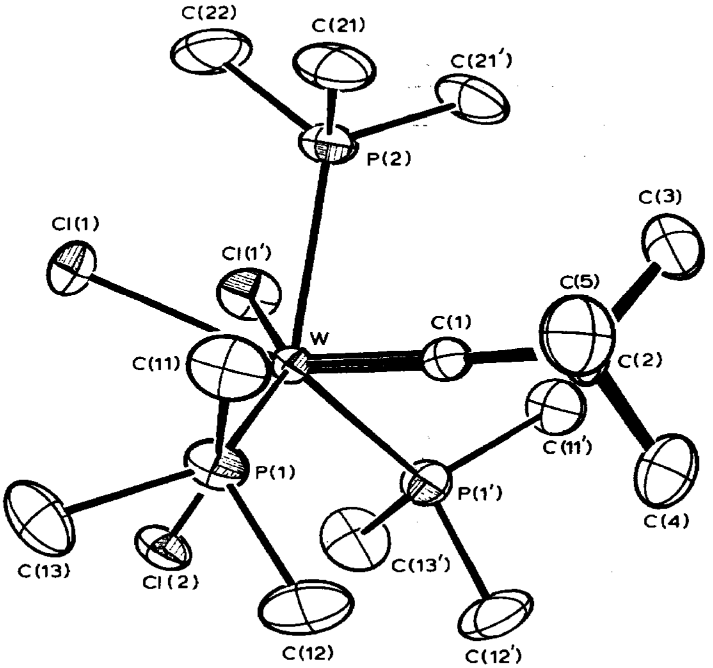

Fig. 1. Overall molecular geometry and labeling of non-hydrogen atoms in the $W(\equiv CCMe_3)(PMe_3)_3Cl_3$ molecule [ORTEP-II diagram]. Note that the neopentylidyne is disordered leading to alternative locations for atoms C(3), C(4) and C(5); these are not shown.

C(2), P(2) and C(22)) and has a capped octahedral coordination geometry of approximate C_{3v} symmetry (see Fig. 2). The neopentylidyne ligand is in the capping position, three PMe_3 ligands occupy positions in the capped octahedral face (C(1)–W–P(1) = C(1)–W–P(1') = 80.63(17)°, C(1)–W–P(2) 80.58(17)°) and the three chloride ligands occupy the triangular octahedral face opposite to the capped face (C(1)–W–Cl(1) = C(1)–W–Cl(1') = 127.55(17)°, C(1)–W–Cl(2) 126.24(17)°). The three PMe_3 ligands are clearly displaced from regular octahedral sites as is evidenced by the obtuse P–W–P angles (i.e., P(1)–W–P(1') 117.17(4), P(1)–W–P(2) =

TABLE 3

INTERATOMIC DISTANCES (Å) FOR $W(\equiv CCMe_3)(PMe_3)_3Cl_3$

Atoms	Dist.	Atoms	Dist.
(A) Distances from the tungsten atom			
W–P(1)	2.513(1)	W–Cl(1)	2.542(1)
W–P(1')	2.513(1)	W–Cl(1')	2.542(1)
W–P(2)	2.519(2)	W–Cl(2)	2.545(2)
W–C(1)	1.793(6)		
(B) Distances from the phosphorus atoms			
P(1)–C(11)	1.805(5)	P(2)–C(21)	1.813(6)
P(1)–C(12)	1.803(6)	P(2)–C(21')	1.813(6)
P(1)–C(13)	1.816(6)	P(2)–C(22)	1.806(9)
(C) Distances within the neopentylidyne ligand			
C(1)–C(2)	1.561(10)	C(2)–C(4)	1.577(15)
C(2)–C(3)	1.563(14)	C(2)–C(5)	1.466(15)

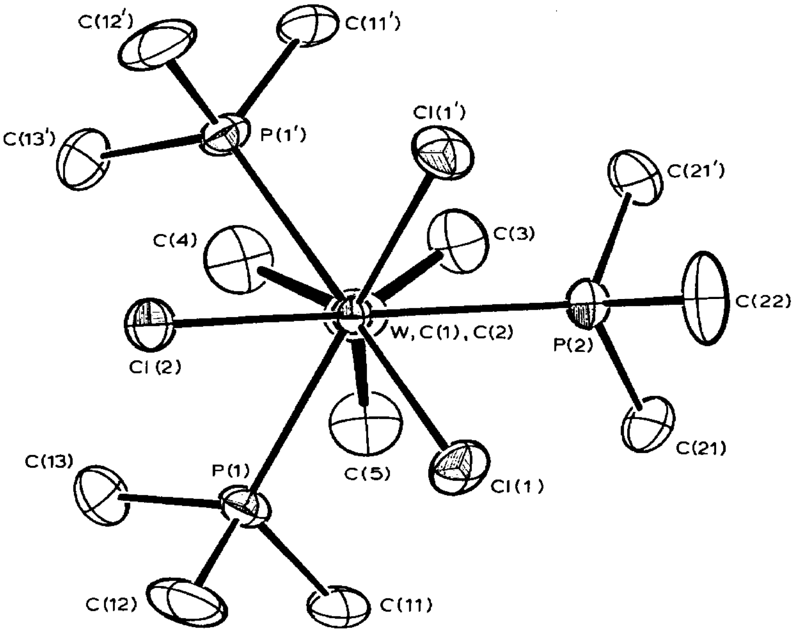

Fig. 2. The $W(\equiv CCMe_3)(PMe_3)_3Cl_3$ molecule viewed down its C_3 axis. The crystallographic mirror plane is horizontal. Atoms C(3), C(4) and C(5) are of half-occupancy and are subject to disorder by reflection about the crystallographic mirror plane. (The resulting C(3'), C(4') and C(5') are omitted for clarity.)

TABLE 4

INTERATOMIC ANGLES (deg.) FOR $W(\equiv CCMe_3)(PMe_3)_3Cl_3$

Atoms	Angle	Atoms	Angle
(A) Angles about the tungsten atom			
P(1)-W-P(1')	117.17(4)	Cl(1)-W-Cl(1')	86.95(4)
P(1')-W-P(2)	117.51(5)	Cl(1')-W-Cl(2)	87.57(4)
P(2)-W-P(1)	117.51(5)	Cl(2)-W-Cl(1)	87.57(4)
C(1)-W-P(1)	80.63(17)	C(1)-W-Cl(1)	127.55(17)
C(1)-W-P(1')	80.63(17)	C(1)-W-Cl(1')	127.55(17)
C(1)-W-P(2)	80.58(17)	C(1)-W-Cl(2)	126.24(17)
(B) Angles within the neopentylidyne ligand			
W-C(1)-C(2)	178.6(4)	C(1)-C(2)-C(5)	113.7(7)
C(1)-C(2)-C(3)	111.5(7)	C(1)-C(2)-C(5')	113.7(7)
C(1)-C(2)-C(3')	111.5(7)	C(3)-C(2)-C(5)	109.4(9)
C(1)-C(2)-C(4)	108.3(6)	C(5)-C(2)-C(4)	109.0(9)
C(1)-C(2)-C(4')	108.3(6)	C(4)-C(2)-C(3)	104.5(8)
(C) Angles about the phosphorus atoms			
W-P(1)-C(11)	115.1(2)	W-P(2)-C(21)	114.8(2)
W-P(1)-C(12)	115.0(2)	W-P(2)-C(22)	119.6(3)
W-P(1)-C(13)	120.1(2)	W-P(2)-C(21')	114.8(2)
C(11)-P(1)-C(12)	105.0(3)	C(21)-P(2)-C(22)	99.5(3)
C(12)-P(1)-C(13)	99.3(3)	C(22)-P(2)-C(21')	99.5(3)
C(13)-P(1)-C(11)	99.8(3)	C(21')-P(2)-C(21)	106.3(3)

P(1′)–W–P(2) = 117.51(5)°) as compared to the normal octahedral values for the Cl–W–Cl angles (Cl(1)–W–Cl(1′) 86.95(4), Cl(1)–W–Cl(2) = Cl(1′)–W–Cl(2) = 87.57(4)°). The three W–P distances are equivalent (W–P(1) = W–P(1′) = 2.513(1) Å, W–P(2) 2.519(2) Å) as are the W–Cl bond lengths (W–Cl(1) = W–Cl(1′) = 2.542(1) Å, W–Cl(2) 2.545(2) Å).

The W–P bond lengths are similar to those found in related complexes [3–8]. The W–Cl bond lengths are towards the long end of the range normally associated with such linkages, cf., W–Cl 2.456(2) Å (*trans* to PHPh) and 2.578(2) Å (*trans* to ≡$CCMe_3$) in the complex $W(\equiv CCMe_3)(PHPh)(PEt_3)_2Cl_2$ [6] and W–Cl 2.545(8) Å (*trans* to ≡$CPMe_3$) in the $[W_2(\equiv CPMe_3)_2(PMe_3)_4Cl_4{}^{2-}]$ dianion [8].

A large number of 7-coordinate structures are now known (for reviews see [12,13,14]). There are four common idealized geometries for heptacoordination:

(1) the pentagonal bipyramid (D_{5h}),
(2) the capped octahedron (C_{3v}),
(3) the capped trigonal prism (C_{2v}) and
(4) the tetragonal base-trigonal base or 4 : 3 geometry (C_s).

It is often difficult to determine which of these idealized cases most closely describes the observed molecular stereochemistry [15–17]. A very recent structural study of $[Et_4N^+][(Ph_3Sn)_3Cr(CO)_4]$ [18] exemplifies this problem. Assuming a capped octahedral structure, the $(OC)_{cap}$–Cr–Sn angles are 74.8(2)–80.3(2)° and $(OC)_{cap}$–Cr–(CO) angles are 113.3(3)–152.2(2)°; despite the fact that visual inspection indicates an irregularly capped octahedral structure, the authors note that "neither qualitative nor quantitative considerations allow a clear choice between capped octahedral, capped trigonal prismatic or 4 : 3 piano stool geometries".

Our present structure is unusual, then, insofar as it presents an example of an almost idealized capped octahedral (C_{3v}) coordination geometry. The central tungsten atom is associated formally with 18 outer valence electrons, i.e., the molecule obeys the "noble gas rule".

We now turn our attention to the neopentylidyne ligand; here the W≡C(1) triple bond is 1.793(6) Å in length and the W≡C(1)–C(2) angle is 178.6(4)°. Despite being in an unusual coordination site (i.e., the unique "capping" position in the capped octahedral geometry) the tungsten–alkylidyne bond length agrees well with other such distances, see Table 5. The present W≡C(1) triple bond distance of 1.793(6) Å may be compared to the W–CH_2PMe_3 single bond distance of 2.305(7) Å in the

TABLE 5

TUNGSTEN–CARBON TRIPLE BOND LENGTHS (Å)

Molecule	W≡C bond length	Ref.
$[W(\equiv CCMe_3)(\eta^5\text{-}C_5Me_4(\text{t-Bu}))I]_2(\mu\text{-}N_2H_2)$	1.769(8)	7
$W(\equiv CCMe_3)(=CHCMe_3)(CH_2CMe_3)(\text{dmpe})$	1.785(8)	2
$W(\equiv CCMe_3)(PMe_3)_3Cl_3$	1.793(6)	This work
$W(\equiv CH \cdot AlMe_{2-x}Cl_{1+x})(Cl)(PMe_3)_3$	1.807(6)	4
$W(\equiv CCMe_3)(PHPh)(PEt_3)_2Cl_2$	1.808(6)	6
$W(\equiv C \cdot Al_2Me_4Cl)(PMe_3)_2(CH_3)(\eta^2\text{-}C_2H_4)$	1.813(5)	5
$W(\equiv C(p\text{-tol}))(\eta^5\text{-}C_5H_5)(CO)_2$	1.82(2)	19
$[\{W(\equiv CPMe_3)(PMe_3)_2Cl_2\}_2{}^{2+}]$	1.833(30)	8

heptacoordinate [$W(CH_2PMe_3)(CO)_2Cl(PMe_3)_3{}^+$] cation [20]. Here, too, the ligand of interest occupies a "capping" position; in this latter case, however, the coordination geometry is that of a capped trigonal prism.

Finally we note that the molecule has almost perfect C_{3v} symmetry and that the three chloride ligands are equivalent.

The X-ray diffraction study indicates that dissociation of the chloride ligand does not arise as a result of a unique weak W–Cl linkage in the ground state geometry.

Additional material

A table of observed and calculated structure factor amplitudes is available upon request (M.R.C.).

Acknowledgments

This work was supported, in part, by NSF grant CHE80-23448 (to M.R.C.). We thank Professor R.R. Schrock for providing crystals of the material studied.

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