C(43)	0.2532 (4)	0.2201 (2)	1.1448 (3)	0.0791 (13)
Li(1)	0.4079 (4)	0.0508 (2)	1.0092 (4)	0.0389 (11)
Li(2)	0.3685 (5)	0.1192 (2)	0.7879 (4)	0.0458 (13)
O	0.3464 (2)	0.17102 (11)	0.6556 (2)	0.0628 (7)
C(1)	0.3127 (6)	0.1589 (2)	0.5445 (3)	0.126 (2)
C(2)	0.2827 (6)	0.2216 (3)	0.4890 (4)	0.119 (2)
C(3)	0.2777 (5)	0.2673 (2)	0.5770 (5)	0.104 (2)
C(4)	0.3502 (7)	0.2393 (2)	0.6696 (4)	0.125 (2)
C(5)	1.0170 (6)	0.1122 (3)	0.3281 (5)	0.128 (2)
C(6)	0.9882 (6)	0.0817 (3)	0.4301 (6)	0.135 (2)
C(7)	1.0140 (5)	0.0159 (3)	0.4474 (4)	0.110 (2)

Table 2. *Selected geometric parameters (Å, °)*

P(1)—C(10)	1.894 (3)	P(2)—C(40)	1.888 (4)
P(1)—C(20)	1.894 (3)	P(2)—Li(1)	2.493 (5)
P(1)—Li(1)	2.652 (5)	P(2)—Li(1)	2.466 (5)
P(1)—Li(2)	2.574 (5)	Li(2)—O	1.926 (5)
P(1)—Li(1)′	2.564 (5)	Li(1)···Li(2)	3.031 (7)
P(2)—C(30)	1.888 (4)	Li(1)···Li(1′)	3.010 (9)
P(2)—Li(2)—P(1)	108.4 (2)	P(1′)—Li(1)—P(1)	109.5 (2)
O—Li(2)—P(1)	128.1 (2)	P(1′)—Li(1)—P(2)	145.2 (2)
P(2)—Li(1)—P(1)	105.2 (2)		

All H atoms were placed in calculated idealized positions and allowed to ride on their corresponding C atoms with fixed isotropic contributions [U_{iso}(fix) = 0.08 or 0.15 Å^2].

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *SDP-Plus* (Enraf–Nonius, 1988); *CADSHEL* (Kopf & Rübcke, 1993). Program(s) used to solve structure: *SHELXTL-Plus* (Siemens, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: local programs.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1099). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Pentacarbonyl[tris(2-methoxyphenyl)-phosphine-*P*]chromium and its Molybdenum Analogue

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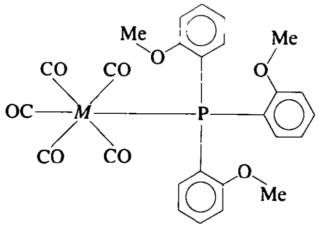
Abstract

In the title compounds, $[Cr(C_{21}H_{21}O_3P)(CO)_5]$ and $[Mo(C_{21}H_{21}O_3P)(CO)_5]$, the *M*—C bond lengths for the *cis*-carbonyl ligands (with respect to the phosphine) are longer than that observed for the *trans*-carbonyl group. The chromium compound has short intermolecular O···O contacts. The two crystal structures are not isomorphous and crystallize in triclinic and monoclinic systems, respectively.

Comment

There have been numerous X-ray structure determinations of phosphine-substituted metal carbonyls. Despite this, we have determined the structures of the complexes $[M(CO)_5(L)]$, where M is Cr [compound (1)] or Mo [compound (2)], and L is tris(2-methoxyphenyl)-phosphine, in order to study the bonding characteristics of the phosphine ligand and the effects of the methoxy substituents on the stereochemistry of the complexes.



(1) M = Cr
(2) M = Co

(I)

The compounds have the expected octahedral geometry in which one carbonyl is *trans* and the remaining four are *cis* with respect to the phosphine ligand. The Cr—P distance of 2.457 (1) Å is longer than those observed in $[Cr(CO)_5(PPh_3)]$, (3), of 2.422 Å,

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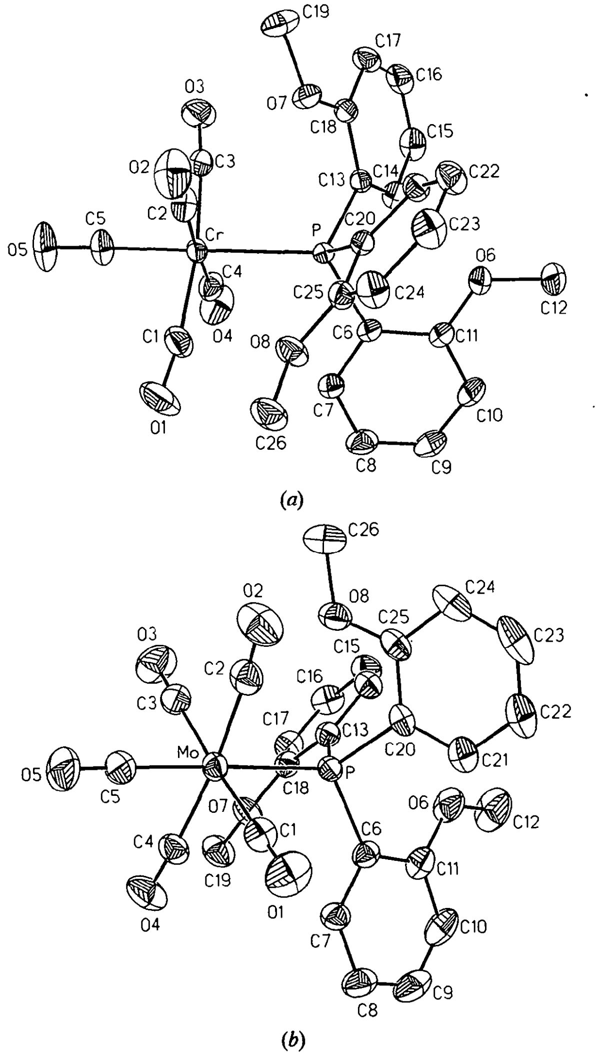
$[Cr(CO)_5\{P(OPh)_3\}]$, (4), of 2.309 Å (Plastas, Stewart & Grim, 1973), and $[Cr(CO)_5\{P(CH_2CH_2CN)_3\}]$, (5), of 2.364 Å (Cotton, Darensbourg & Ilsley, 1981). As the Cr—P bond shortens along the series (1)→(3)→(5)→(4), the carbonyl stretching frequencies, ν(CO) (*E* mode), increase [compound (1) 1936, (3) 1942, (5) 1944 and (4) 1960 cm^{-1}]. The shortest carbonyl Cr—C distance [1.851 (2) Å for Cr—C5] is that *trans* to the P atom, consistent with phosphine having a lower *trans* influence than CO. The Cr—C distances *cis* to the P atom vary from 1.880 (2) to 1.899 (2) Å, with an average value of 1.892 Å. The steric influence of the tris(2-methoxyphenyl)phosphine ligand is evident in the distortion of two of the four *cis* carbonyl ligands. Two of the methoxy groups are oriented towards the Cr-*cis*-(CO) plane, resulting in bending of the Cr—C1—O1 [172.9 (2)°] and Cr—C3—O3 [174.4 (2)°] bonds; the *trans* CO group is not affected and is almost linear [Cr—C5—O5 178.8 (2)°] (Fig. 1).



(*a*)

(*b*)

Fig. 1. Displacement ellipsoid plots (30% probability) of (*a*) complex (1) and (*b*) complex (2), showing both numbering schemes. Atoms C21 of (1) and C14 of (2) are unlabelled for the sake of clarity and can be inferred from the cyclic numbering.

In compound (2), the Mo—P distance is 2.589 (1) Å, longer than that observed in other $Mo(CO)_5$–phosphine complexes {*e.g.* 2.560 Å in $[Mo(CO)_5(PPh_3)]$, (6), and 2.562 Å in $[Mo(CO)_5\{P(tolyl)_3\}]$, (7) (Cotton, Darensbourg & Ilsley, 1981; Alyea, Ferguson & Somogyvari, 1983)}; the observed stretching frequencies of 1942 for compound (6), 1949 for (7) and 1941 cm^{-1} for (2) show a correlation with *M*—P bond lengths similar to that found for the Cr complexes. The carbonyl Mo—C distance *trans* to the P atom [Mo—C5 1.981 (3) Å] is shorter than the *cis* Mo—CO distances and bending of the Mo—C—O bonds away from the methoxy groups is also observed.

In both complexes (1) and (2), intermolecular C—H···O hydrogen bonds are observed: in (1), $C8\cdots O5^{i}$ 3.527 (3) Å and $C8—H8\cdots O5^{i}$ 152 (3)°, $C9\cdots O3^{ii}$ 3.549 (2) Å and $C9—H9\cdots O3^{ii}$ 152 (3)°; in (2), $C8\cdots O3^{iii}$ 3.574 (4) Å and $C8—H8\cdots O3^{iii}$ 131 (2)°, $C16\cdots O4^{iv}$ 3.456 (4) Å and $C16—H16\cdots O4^{iv}$ 126 (2)°, $C21\cdots O5^{v}$ 3.416 (4) Å and $C21—H21\cdots O5^{v}$ 125 (2)°; symmetry codes: (i) $1-x$, $2-y$, $-z$; (ii) $x-1$, $y+1$, z; (iii) $x-\frac{1}{2}$, $\frac{3}{2}-y$, $z-\frac{1}{2}$; (iv) $x+\frac{1}{2}$, $\frac{3}{2}-y$, $z+\frac{1}{2}$; (v) $\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{3}{2}-z$. Notable short intermolecular O···O contacts are observed in compound (1) only: $O1\cdots O4^{i}$ 2.998 (3) and O2···O2(1 − *x*, 1 − *y*, 1 − *z*) 3.051 (3) Å.

Contrary to our expectation, the crystal structures of complexes (1) and (2) are not isomorphous. The orientations of the methoxyphenyl rings of the phosphine ligands differ in the two complexes.

Experimental

Complex (1) was prepared by the reaction of hexacarbonylchromium and tris(2-methoxyphenyl)phosphine in refluxing tetrahydrofuran. Complex (2) was prepared by the reaction of hexacarbonylmolybdenum and tris(2-methoxyphenyl)phosphine in refluxing ethanol in the presence of a catalytic amount of sodium borohydride. Single crystals of both complexes suitable for X-ray analysis were grown from dichloromethane/ethanol solution by the solvent–solvent diffusion method.

Complex (1)

Crystal data

$[Cr(C_{21}H_{21}O_3P)(CO)_5]$
M_r = 544.40
Triclinic
$P\overline{1}$
a = 10.520 (1) Å
b = 10.890 (1) Å
c = 12.854 (1) Å
α = 74.62 (1)°
β = 72.55 (1)°
γ = 64.70 (1)°
V = 1253.9 (2) $Å^3$
Z = 2
D_x = 1.442 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 8–25°
μ = 0.568 mm^{-1}
T = 293 (2) K
Rectangular
0.58 × 0.44 × 0.42 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction:
none
6654 measured reflections
5718 independent reflections
5088 observed reflections
[$I > 2\sigma(I)$]
$R_{\rm int} = 0.0118$

$\theta_{\rm max} = 27.5°$
$h = -1 \rightarrow 13$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 16$
3 standard reflections
monitored every 97
reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R(F) = 0.0335$
$wR(F^2) = 0.1042$
$S = 1.052$
5718 reflections
409 parameters
All H-atom parameters
refined
$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2$
$+ 0.2748P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.36$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.36$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *International Tables*
for Crystallography (1992,
Vol. C, Tables 4.2.6.8 and
6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (1)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	$U_{\rm eq}$
Cr	0.47872 (3)	0.72801 (3)	0.18004 (2)	0.03571 (9)
P	0.23412 (4)	0.73379 (4)	0.26560 (3)	0.02956 (10)
O1	0.4315 (3)	1.0030 (2)	0.2272 (2)	0.1025 (8)
O2	0.5582 (2)	0.5907 (2)	0.4022 (2)	0.0891 (6)
O3	0.5922 (2)	0.4511 (2)	0.1055 (2)	0.0801 (6)
O4	0.4079 (2)	0.8450 (2)	−0.04654 (14)	0.0883 (6)
O5	0.7777 (2)	0.7196 (3)	0.0837 (2)	0.0922 (7)
O6	−0.08448 (13)	0.78906 (13)	0.32409 (11)	0.0462 (3)
O7	0.38133 (14)	0.43832 (12)	0.33473 (11)	0.0462 (3)
O8	0.2246 (2)	0.90792 (14)	0.40895 (11)	0.0528 (3)
C1	0.4402 (2)	0.9007 (2)	0.2122 (2)	0.0590 (5)
C2	0.5254 (2)	0.6420 (2)	0.3201 (2)	0.0529 (4)
C3	0.5439 (2)	0.5534 (2)	0.1371 (2)	0.0499 (4)
C4	0.4308 (2)	0.8032 (2)	0.0401 (2)	0.0522 (4)
C5	0.6644 (2)	0.7216 (2)	0.1198 (2)	0.0559 (5)
C6	0.0848 (2)	0.8872 (2)	0.22032 (13)	0.0362 (3)
C7	0.1138 (2)	0.9982 (2)	0.1507 (2)	0.0468 (4)
C8	0.0052 (3)	1.1146 (2)	0.1109 (2)	0.0583 (5)
C9	−0.1328 (2)	1.1196 (2)	0.1396 (2)	0.0592 (5)
C10	−0.1667 (2)	1.0119 (2)	0.2097 (2)	0.0510 (4)
C11	−0.0587 (2)	0.8967 (2)	0.25216 (14)	0.0393 (3)
C12	−0.2240 (2)	0.7870 (3)	0.3517 (3)	0.0629 (6)
C13	0.2154 (2)	0.59256 (15)	0.22705 (13)	0.0331 (3)
C14	0.1336 (2)	0.6168 (2)	0.15105 (15)	0.0434 (4)
C15	0.1330 (2)	0.5081 (2)	0.1137 (2)	0.0557 (5)
C16	0.2141 (3)	0.3757 (2)	0.1519 (2)	0.0577 (5)
C17	0.2975 (2)	0.3479 (2)	0.2264 (2)	0.0508 (4)
C18	0.2990 (2)	0.4557 (2)	0.26322 (14)	0.0381 (3)
C19	0.4831 (3)	0.3041 (2)	0.3631 (2)	0.0676 (6)
C20	0.1743 (2)	0.7150 (2)	0.41678 (12)	0.0329 (3)
C21	0.1200 (2)	0.6153 (2)	0.47859 (14)	0.0405 (3)
C22	0.0748 (2)	0.6036 (2)	0.5924 (2)	0.0518 (4)
C23	0.0847 (3)	0.6921 (2)	0.6464 (2)	0.0579 (5)
C24	0.1356 (2)	0.7938 (2)	0.5877 (2)	0.0522 (4)
C25	0.1787 (2)	0.8068 (2)	0.47315 (14)	0.0390 (3)
C26	0.2245 (3)	1.0068 (3)	0.4627 (2)	0.0639 (6)

Complex (2)

Crystal data

[$Mo(C_{21}H_{21}O_3P)(CO)_5$]
$M_r = 588.34$

Mo *Kα* radiation
$\lambda = 0.71073$ Å

Monoclinic
$P2_1/n$
$a = 10.889$ (2) Å
$b = 16.395$ (2) Å
$c = 14.808$ (2) Å
$\beta = 92.57$ (1)°
$V = 2640.9$ (7) Å^3
$Z = 4$
$D_x = 1.480$ Mg m^{-3}
D_m not measured

Cell parameters from 25
reflections
$\theta = 8–25°$
$\mu = 0.603$ mm^{-1}
$T = 293$ (2) K
Thick plate
0.54 × 0.28 × 0.18 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction:
none
7456 measured reflections
6039 independent reflections
4100 observed reflections
[$I > 2\sigma(I)$]
$R_{\rm int} = 0.0265$

$\theta_{\rm max} = 27.5°$
$h = -1 \rightarrow 14$
$k = -1 \rightarrow 21$
$l = -19 \rightarrow 19$
3 standard reflections
monitored every 97
reflections
intensity decay: <3%

Refinement

Refinement on F^2
$R(F) = 0.0350$
$wR(F^2) = 0.0849$
$S = 0.899$
6039 reflections
409 parameters
All H-atom parameters
refined
$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.45$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.51$ e Å^{-3}
Extinction correction: none
Atomic scattering factors
from *International Tables*
for Crystallography (1992,
Vol. C, Tables 4.2.6.8 and
6.1.1.4)

Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²) for (2)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	$U_{\rm eq}$
Mo	0.21639 (2)	0.612015 (14)	0.915027 (15)	0.04504 (8)
P	0.26133 (6)	0.76401 (4)	0.95028 (4)	0.04017 (15)
O1	0.2002 (3)	0.6514 (2)	0.7066 (2)	0.0941 (8)
O2	0.4936 (2)	0.5629 (2)	0.8857 (2)	0.0950 (9)
O3	0.2506 (3)	0.5740 (2)	1.1256 (2)	0.1013 (9)
O4	−0.0723 (2)	0.6250 (2)	0.9364 (2)	0.0907 (8)
O5	0.1587 (3)	0.43066 (14)	0.8642 (2)	0.0981 (9)
O6	0.2615 (3)	0.94607 (13)	0.9620 (2)	0.0817 (7)
O7	0.0574 (2)	0.75695 (13)	1.06777 (13)	0.0602 (5)
O8	0.4983 (2)	0.70371 (13)	1.01961 (14)	0.0605 (5)
C1	0.2035 (3)	0.6381 (2)	0.7820 (2)	0.0589 (7)
C2	0.3961 (3)	0.5841 (2)	0.8977 (2)	0.0624 (8)
C3	0.2382 (3)	0.5871 (2)	1.0508 (2)	0.0614 (8)
C4	0.0319 (3)	0.6247 (2)	0.9294 (2)	0.0591 (8)
C5	0.1798 (3)	0.4967 (2)	0.8842 (2)	0.0640 (8)
C6	0.1563 (2)	0.8371 (2)	0.8932 (2)	0.0476 (6)
C7	0.0611 (3)	0.8096 (2)	0.8351 (2)	0.0545 (7)
C8	−0.0229 (3)	0.8618 (3)	0.7928 (2)	0.0712 (9)
C9	−0.0139 (4)	0.9434 (3)	0.8110 (3)	0.0850 (12)
C10	0.0775 (5)	0.9728 (2)	0.8680 (3)	0.0874 (13)
C11	0.1641 (3)	0.9208 (2)	0.9084 (2)	0.0639 (8)
C12	0.2654 (8)	1.0282 (3)	0.9925 (5)	0.115 (2)
C13	0.2615 (2)	0.8006 (2)	1.0677 (2)	0.0426 (6)
C14	0.3614 (3)	0.8387 (2)	1.1109 (2)	0.0537 (7)
C15	0.3518 (3)	0.8711 (2)	1.1975 (2)	0.0629 (8)
C16	0.2445 (3)	0.8661 (2)	1.2399 (2)	0.0680 (9)
C17	0.1446 (3)	0.8282 (2)	1.1990 (2)	0.0604 (8)
C18	0.1523 (3)	0.7953 (2)	1.1132 (2)	0.0477 (6)
C19	−0.0619 (3)	0.7648 (3)	1.1005 (4)	0.0795 (11)

C20	0.4123 (2)	0.7930 (2)	0.9116 (2)	0.0470 (6)
C21	0.4253 (3)	0.8458 (2)	0.8400 (2)	0.0615 (8)
C22	0.5404 (4)	0.8642 (3)	0.8085 (3)	0.0809 (11)
C23	0.6407 (4)	0.8292 (3)	0.8478 (3)	0.0891 (15)
C24	0.6329 (3)	0.7747 (3)	0.9192 (3)	0.0734 (10)
C25	0.5179 (3)	0.7565 (2)	0.9515 (2)	0.0542 (7)
C26	0.6015 (4)	0.6647 (3)	1.0632 (3)	0.0822 (12)

Table 3. *Selected distances (Å) and angles (°) in complexes (1) and (2)*

	(1) *M* = Cr	(2) *M* = Mo
M—P	2.4568 (5)	2.5883 (7)
M—C1	1.880 (2)	2.015 (3)
M—C2	1.898 (2)	2.037 (4)
M—C3	1.899 (2)	2.055 (3)
M—C4	1.890 (2)	2.040 (3)
M—C5	1.851 (2)	1.981 (3)
P—C6	1.840 (2)	1.837 (3)
P—C13	1.836 (2)	1.839 (3)
P—C20	1.838 (2)	1.828 (3)
O1—C1	1.141 (3)	1.137 (3)
O2—C2	1.133 (3)	1.139 (4)
O3—C3	1.138 (2)	1.131 (4)
O4—C4	1.141 (3)	1.144 (4)
O5—C5	1.135 (3)	1.143 (4)
O1—C1—*M*	172.9 (2)	177.5 (3)
O2—C2—*M*	177.5 (2)	174.8 (3)
O3—C3—*M*	174.4 (2)	179.5 (3)
O4—C4—*M*	176.9 (2)	174.3 (3)
O5—C5—*M*	178.8 (2)	178.3 (3)
C1—*M*—P	94.47 (7)	89.85 (9)
C2—*M*—P	87.16 (6)	93.88 (9)
C3—*M*—P	93.61 (6)	88.92 (9)
C4—*M*—P	91.40 (6)	93.36 (9)
C5—*M*—P	178.25 (7)	178.19 (9)
C6—P—*M*	116.77 (6)	115.29 (9)
C13—P—*M*	108.23 (5)	119.78 (9)
C20—P—*M*	119.39 (5)	110.59 (9)
C13—P—C6	103.60 (7)	101.37 (12)
C20—P—C6	102.12 (7)	103.68 (13)
C13—P—C20	105.11 (7)	104.39 (12)

For both compounds, data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structures: *SHELXTL/PC* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXL93*; geometrical calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1238). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dioxo(η^5-pentamethylcyclopentadienyl)(trimethylsilylmethyl)tungsten(VI)

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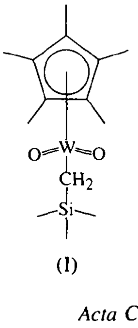
Abstract

(η^5-C_5Me_5)WO_2(CH_2SiMe_3), which has crystallographic mirror symmetry, exhibits the three-legged piano stool geometry typical for complexes of this type. The pentamethylcyclopentadienyl ligand is asymmetrically bound to the metal atom [W—C = 2.321 (8)–2.477 (4) Å]. The W═O and W—C(alkyl) distances are 1.717 (4) and 2.128 (8) Å, respectively.

Comment

The physical properties of (η^5-C_5Me_5)WO_2(CH_2SiMe_3), (I), have been reported elsewhere (Legzdins, Rettig & Sayers, 1994). Further examples of related organometallic oxo chemistry are fully described in the literature (Legzdins, Phillips & Sanchez, 1989; Legzdins, Rettig & Sanchez, 1985). The structure of (η^5-C_5Me_5)WO_2(CH_2SiMe_3) is very similar to that of the cyclopentadienyl derivative (η^5-C_5H_5)WO_2(CH_2SiMe_3) (Legzdins, Rettig & Sanchez, 1985). Corresponding



(I)