

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 [$U_{\text{iso}}(\text{fix}) = 0.08$ 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); *CADSHL* (Kopf & Rübcke, 1993). Program(s) used to solve structure: *SHELXTL-Plus* (Siemens, 1990). Program(s) used to refine structure: *SHELXL93* (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.

References

- Bartlett, R. A., Olmstead, M. M. & Power, P. P. (1986). *Inorg. Chem.* **25**, 1243.
- Enraf–Nonius (1988). *SDP-Plus Structure Determination Package*. Version 4.0. Enraf–Nonius, Delft, The Netherlands.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Hey, E., Hitchcock, P. B., Lappert, M. F. & Rai, A. K. (1987). *J. Organomet. Chem.* **325**, 1–12.
- Jones, R. A., Stuart, A. L. & Wright, T. C. (1983). *J. Am. Chem. Soc.* **105**, 7459–7460.
- Kopf, J. & Rübcke, J. (1993). *CADHKL4 (CADSHL.FOR)*, version 3.10). University of Hamburg, Germany.
- Rabe, G. W., Riede, J. & Schier, A. (1996). *Inorg. Chem.* **35**, 40–45.
- Sheldrick, G. M. (1990). *SHELXTL-Plus*. Release 4.0 for Siemens R3 Crystallographic Research System. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

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

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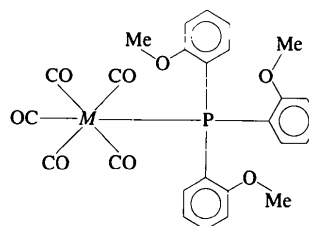
(Received 6 December 1995; accepted 1 February 1996)

Abstract

In the title compounds, [Cr(C₂H₂₁O₃P)(CO)₅] and [Mo(C₂H₂₁O₃P)(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 = Mo

(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(PPh₃)], (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(CH₂CH₂CN)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, $\nu(\text{CO})$ (*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).

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(PPh₃)], (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...O5ⁱ 3.527 (3) Å and C8—H8...O5ⁱ 152 (3)°, C9...O3ⁱⁱ 3.549 (2) Å and C9—H9...O3ⁱⁱ 152 (3)°; in (2), C8...O3ⁱⁱⁱ 3.574 (4) Å and C8—H8...O3ⁱⁱⁱ 131 (2)°, C16...O4^{iv} 3.456 (4) Å and C16—H16...O4^{iv} 126 (2)°, C21...O5^v 3.416 (4) Å and C21—H21...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...O4ⁱ 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(C₂₁H₂₁O₃P)(CO)₅]

M_r = 544.40

Triclinic

P $\bar{1}$

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) Å³

Z = 2

D_x = 1.442 Mg m⁻³

D_m not measured

Mo *K*α 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

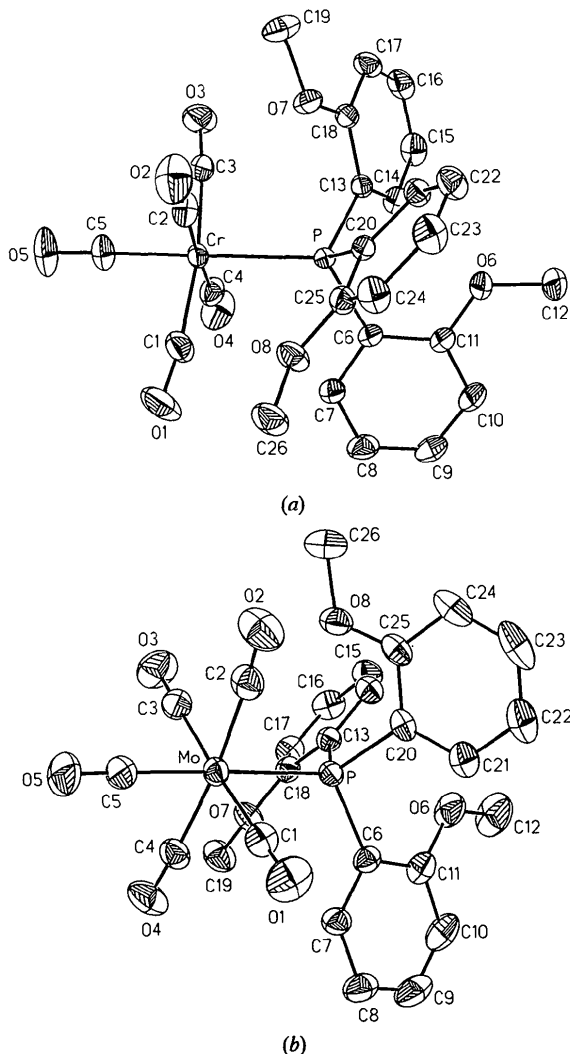


Fig. 1. Displacement ellipsoid plots (30% probability) of (a) complex (1) and (b) complex (2), showing both numbering schemes. Atoms C21 of (1) and C14 of (2) are unlabelled for the sake of clarity and can be inferred from the cyclic numbering.

Data collection

Siemens P4 diffractometer $\theta_{\max} = 27.5^\circ$
 $\theta/2\theta$ scans $h = -1 \rightarrow 13$
 Absorption correction: $k = -13 \rightarrow 13$
 none $l = -16 \rightarrow 16$
 6654 measured reflections 3 standard reflections
 5718 independent reflections monitored every 97
 5088 observed reflections reflections
 $[I > 2\sigma(I)]$ intensity decay: $<3\%$
 $R_{\text{int}} = 0.0118$

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\max} = 0.001$
 $R(F) = 0.0335$ $\Delta\rho_{\max} = 0.36 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.1042$ $\Delta\rho_{\min} = -0.36 \text{ e } \text{\AA}^{-3}$
 $S = 1.052$ Extinction correction: none
 5718 reflections Atomic scattering factors
 409 parameters from *International Tables*
 All H-atom parameters for *Crystallography* (1992,
 refined Vol. C, Tables 4.2.6.8 and
 $w = 1/[\sigma^2(F_o^2) + (0.0612P)^2$
 $+ 0.2748P]$ 6.1.1.4)
 where $P = (F_o^2 + 2F_c^2)/3$

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for (1)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{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(C₂₁H₂₁O₃P)(CO)₅]
 $M_r = 588.34$ Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$

Monoclinic

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

Cell parameters from 25

reflections
 $\theta = 8-25^\circ$
 $\mu = 0.603 \text{ mm}^{-1}$
 $T = 293 (2) \text{ K}$
 Thick plate
 $0.54 \times 0.28 \times 0.18 \text{ mm}$
 Colourless

Data collection

Siemens P4 diffractometer $\theta_{\max} = 27.5^\circ$
 $\theta/2\theta$ scans $h = -1 \rightarrow 14$
 Absorption correction: $k = -1 \rightarrow 21$
 none $l = -19 \rightarrow 19$
 7456 measured reflections 3 standard reflections
 6039 independent reflections monitored every 97
 4100 observed reflections reflections
 $[I > 2\sigma(I)]$ intensity decay: $<3\%$
 $R_{\text{int}} = 0.0265$

Refinement

Refinement on F^2 $(\Delta/\sigma)_{\max} = 0.001$
 $R(F) = 0.0350$ $\Delta\rho_{\max} = 0.45 \text{ e } \text{\AA}^{-3}$
 $wR(F^2) = 0.0849$ $\Delta\rho_{\min} = -0.51 \text{ e } \text{\AA}^{-3}$
 $S = 0.899$ Extinction correction: none
 6039 reflections Atomic scattering factors
 409 parameters from *International Tables*
 All H-atom parameters for *Crystallography* (1992,
 refined Vol. C, Tables 4.2.6.8 and
 $w = 1/[\sigma^2(F_o^2) + (0.0389P)^2]$ 6.1.1.4)
 where $P = (F_o^2 + 2F_c^2)/3$

Table 2. Fractional atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2$) for (2)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j.$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{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) <i>M</i> = Cr	(2) <i>M</i> = Mo
<i>M</i> —P	2.4568 (5)	2.5883 (7)
<i>M</i> —C1	1.880 (2)	2.015 (3)
<i>M</i> —C2	1.898 (2)	2.037 (4)
<i>M</i> —C3	1.899 (2)	2.055 (3)
<i>M</i> —C4	1.890 (2)	2.040 (3)
<i>M</i> —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— <i>M</i>	172.9 (2)	177.5 (3)
O2—C2— <i>M</i>	177.5 (2)	174.8 (3)
O3—C3— <i>M</i>	174.4 (2)	179.5 (3)
O4—C4— <i>M</i>	176.9 (2)	174.3 (3)
O5—C5— <i>M</i>	178.8 (2)	178.3 (3)
C1— <i>M</i> —P	94.47 (7)	89.85 (9)
C2— <i>M</i> —P	87.16 (6)	93.88 (9)
C3— <i>M</i> —P	93.61 (6)	88.92 (9)
C4— <i>M</i> —P	91.40 (6)	93.36 (9)
C5— <i>M</i> —P	178.25 (7)	178.19 (9)
C6—P— <i>M</i>	116.77 (6)	115.29 (9)
C13—P— <i>M</i>	108.23 (5)	119.78 (9)
C20—P— <i>M</i>	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.

References

- Alyea, E. C., Ferguson, G. & Somogyvari, A. (1983). *Organometallics*, **2**, 668–674.
 Cotton, F. A., Darensbourg, D. J. & Ilsley, W. H. (1981). *Inorg. Chem.* **20**, 578–583.
 Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

- Plastas, J., Stewart, J. M. & Grim, S. O. (1973). *Inorg. Chem.* **12**, 265–272.
 Sheldrick, G. M. (1990). *SHELXTL/PC. Structure Determination Software Programs*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
 Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System. Version 2.1*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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Dioxo(η^5 -pentamethylcyclopentadienyl)(tri-methylsilylmethyl)tungsten(VI)

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Abstract

(η^5 -C₅Me₅)WO₂(CH₂SiMe₃), 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 -C₅Me₅)WO₂(CH₂SiMe₃), (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 -C₅Me₅)WO₂(CH₂SiMe₃) is very similar to that of the cyclopentadienyl derivative (η^5 -C₅H₅)WO₂(CH₂SiMe₃) (Legzdins, Rettig & Sanchez, 1985). Corresponding

