Weights $w_i = [\sigma_{oi}^2(F) + 0.00026|F_o|^2]^{-1}$, where $\sigma_{oi}^2(F)$ is the variance based on counting statistics, were assigned to observed reflections. The refinement was conducted until convergence. Final unweighted and weighted R factors were 0.055 and 0.051, and goodness-of-fit = 2.21. Maximum and minimum heights in final difference Fourier synthesis were 0.45 and -0.29 e Å^{-3} respectively. Maximum ratio of least-squares shift to e.s.d. in final refinement cycle was < 0.01.

Anisotropic temperature factors were associated with all non-H atoms. The H-atom positions were calculated and an isotropic temperature factor of 6.00 Å^2 was assigned to them. Complex neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974, Vol. IV). Final atomic parameters are listed in Table 1.* One discrete molecule and its numbering scheme are shown in Fig. 1. Relevant interatomic distances and angles are given in Table 2.

Related literature. A discussion about the chemistry of this kind of compound has been published (Santos, Stein & Vichi, 1985). When binding to Fe, the ligand is usually bidentate (Fontaine, Jacobsen, Shaw & Thornton-Pett, 1987; Jacobsen, Shaw & Thornton-Pett, 1987); however, there are cases where it is monodentate, for instance with Os (Cartwright, Clucas, Dawson, Foster, Harding & Smith, 1986). In the case of the title compound the closest contact between Fe and P2 is intramolecular with a distance of 3.907 (3) Å.

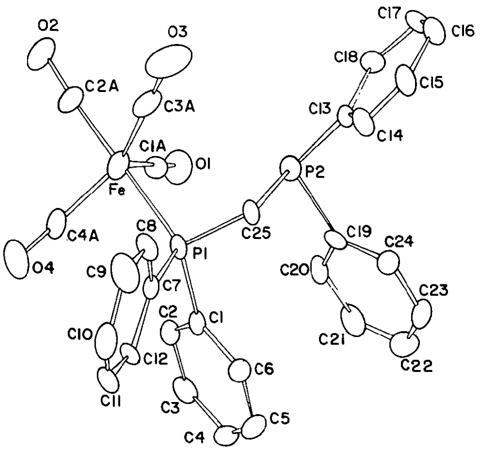

Fig. 1. *ORTEP* (Johnson, 1965) representation of the title compound.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and interatomic distances and angles in the phenyl rings have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54480 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Dicarbonyl(η^5-cyclopentadienyl)[(α,1,2-η)-*p*-methylbenzyl]tungsten

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Abstract. [W(CO)$_2$(C$_5$H$_5$)(C$_8$H$_9$)], $M_r = 410.12$, monoclinic, $P2_1/n$, $a = 6.339$ (1), $b = 17.141$ (2), $c = 12.526$ (1) Å, $\beta = 101.86$ (1)°, $V = 1332.0$ Å^3, $Z = 4$, $D_x = 2.05$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 82.92$ cm^{-1}, $F(000) = 776$, ambient temperature, $R = 0.022$ for 2446 unique observed reflections [$F_o >$

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$2\sigma(F_o)$]. The title compound has a *p*-methylbenzyl ligand with unusual (α,1,2-η)-coordination to the dicarbonyl(η^5-cyclopentadienyl)tungsten moiety.

Experimental. A deep-orange crystal grown from a petroleum ether (boiling range 303–333 K) solution at 253 K was sealed in a Lindemann capillary and used for the data collection. Space group and approximate cell constants were determined from precession and Weissenberg photographs. Exact cell constants were derived by a least-squares procedure from 57 reflections of a Guinier powder diffractogram. Intensity data were collected on a Syntex *R*3 four-circle diffractometer by the $\omega/2\theta$-scan method with graphite-monochromated radiation; $3 \le 2\theta \le 56°$, $0 \le h \le 8$, $0 \le k \le 22$, $-16 \le l \le 16$; 2670 reflections measured, 2451 unique, 2446 observed [$F_o > 2\sigma(F_o)$]. The intensities of three check reflections (002, $\bar{1}01$, 111) were monitored every 100 reflections and showed a slow decay of about 2%. Corrections were made for Lorentz–polarization and absorption effects (empirical). The position of the W atom was taken from a Patterson synthesis and all other atoms except the methyl H atoms were located from subsequent difference Fourier syntheses. The methyl group was treated as a rigid group with H atoms in calculated positions. Refinement on *F* was carried out by blocked-matrix least squares with anisotropic thermal parameters for all non-H atoms (213 parameters). Final residuals were $R = 0.023$, $wR = 0.028$, $w = 1.0/[\sigma^2(F_o) + 0.0002F_o^2]$; $(\Delta/\sigma)_{\max} = 0.1$. A final difference Fourier synthesis showed no features larger than 0.49 e Å^{-3}. All calculations were performed with the *SHELX76* program (Sheldrick, 1976) on an IBM 3090 computer. Atomic scattering factors were taken from Cromer & Mann (1968). Atomic parameters and $U_{\rm eq}$ values are given in Table 1, important bond distances and angles in Table 2.* A drawing of the molecule is shown in Fig. 1.

Related literature. The structure of the Mo analog has been reported by Cotton & LaPrade (1968). Derivatives with 9-borabicyclo[3.3.1]nonan-9-yl (Carriedo, Elliott, Howard, Lewis & Stone, 1984), diethylboryl (Wadepohl & Pritzkow, 1991) and phenyl substituents (Jeffery, Raterman & Stone, 1985) on the α-carbon atom have also been structurally investigated.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{\rm eq}$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U_{\rm eq}$
W	0.08179 (3)	0.221310 (11)	0.035569 (11)	0.038
C1	0.4037 (9)	0.2494 (4)	0.1411 (5)	0.038
C2	0.2713 (8)	0.3149 (3)	0.1559 (4)	0.046
C3	0.0880 (9)	0.3011 (3)	0.2004 (4)	0.052
C4	−0.0552 (9)	0.3632 (3)	0.2105 (4)	0.049
C5	−0.0213 (9)	0.4358 (3)	0.1778 (4)	0.054
C6	0.1604 (10)	0.4499 (3)	0.1294 (5)	0.064
C7	0.2982 (9)	0.3928 (3)	0.1170 (4)	0.047
C8	−0.1724 (12)	0.5028 (4)	0.1900 (8)	0.085
O1	0.3093 (7)	0.2843 (3)	−0.1429 (3)	0.071
O2	−0.2185 (7)	0.3530 (2)	−0.0822 (4)	0.065
C9	0.2267 (8)	0.2600 (3)	−0.0757 (4)	0.047
C10	−0.1088 (8)	0.3044 (3)	−0.0352 (4)	0.045
C11	−0.0684 (12)	0.1125 (4)	−0.0548 (5)	0.097
C12	0.1271 (14)	0.0913 (4)	0.0103 (8)	0.075
C13	0.1069 (17)	0.0981 (4)	0.1175 (8)	0.111
C14	−0.100 (2)	0.1242 (4)	0.1174 (8)	0.157
C15	−0.2046 (11)	0.1331 (3)	0.0111 (7)	0.061

Table 2. *Bond lengths* (Å) *and angles* (°)

W—C1	2.244 (5)	C(1)—C(2)	1.436 (8)
W—C2	2.355 (5)	C(2)—C(3)	1.407 (8)
W—C3	2.470 (5)	C(2)—C(7)	1.443 (7)
W—C9	1.938 (5)	C(3)—C(4)	1.422 (8)
W—C10	1.958 (5)	C(4)—C(5)	1.342 (8)
W—C11	2.286 (6)	C(5)—C(6)	1.428 (9)
W—C12	2.277 (7)	C(5)—C(8)	1.522 (9)
W—C13	2.340 (7)	C(6)—C(7)	1.342 (8)
W—C14	2.373 (10)	O(1)—C(9)	1.156 (7)
W—C15	2.334 (6)	O(2)—C(10)	1.164 (6)
C3—C2—C1	118.1 (4)	C8—C5—C4	121.7 (5)
C7—C2—C1	124.5 (5)	C8—C5—C6	119.7 (5)
C7—C2—C3	117.0 (4)	C7—C6—C5	121.9 (5)
C4—C3—C2	120.3 (4)	C6—C7—C2	120.7 (5)
C5—C4—C3	121.5 (5)	O1—C9—W	178.4 (4)
C6—C5—C4	118.6 (5)	O2—C10—W	176.5 (4)

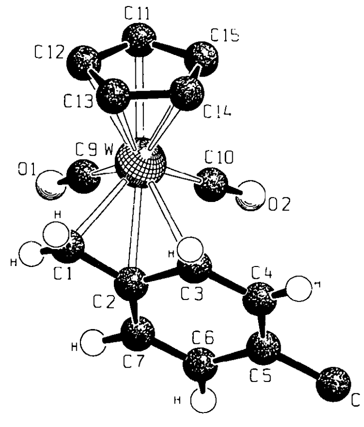

Fig. 1. *SCHAKAL* (Keller, 1988) drawing of the molecule with methyl and cyclopentadienyl H atoms omitted for clarity.

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Di-μ-carbonyl-dicarbonylbis[(2-hydroxyethyl)-η^5-cyclopentadienyl]diiron(*Fe–Fe*)

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Abstract. $C_{18}H_{18}Fe_2O_6$, $M_r = 442.03$, trigonal, $R\bar{3}$, $a = 16.577$ (3), $c = 16.957$ (3) Å, $V = 4035$ (3) Å^3, $Z = 9$, $D_x = 1.637$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 1.65$ mm^{-1}, $F(000) = 2034$, $T = 295$ K, $R_F = 0.039$ for 1173 independent reflections [$I \geq 3\sigma(I)$] and 118 parameters. The compound is the *trans* isomer. The molecules lie on crystallographic inversion centers. Bond lengths include: Fe—Fe 2.534 (1), Fe—CO 1.759 (5), Fe—CO(μ) 1.897, 1.933 (5) Å. Hydrogen bonds between OH groups [all O···O 2.656 (4) Å] link the molecules into nets parallel to the *ab* plane.

Experimental. Preparation: Tenhaeff & Tyler (1991); red-black prism from CH_2Cl_2–hexane, 0.10 × 0.12 × 0.42 mm, mounted on a fiber; Rigaku AFC6R diffractometer, graphite monochromator; orientation matrix and cell dimensions from 20 centered reflections in range $22 \leq 2\theta \leq 26°$; Laue symmetry $\bar{3}$; ω–2θ scans, 8° min^{-1} in ω, width $(1.15 + 0.30\tan\theta)°$; $2\theta_{\max} = 50°$; h −16 to 16, k 0 to 19, l 0 to 20; three standard reflections every 300, no change; 1584 independent reflections, 1173 with $I \geq 3\sigma(I)$, $R_{\text{int}} = 0.051$ (on F^2); no systematic absences other than for R lattice; relative transmission factors 0.94–1.00 (ψ scans), absorption correction not applied.

Fe position from *MITHRIL E* map (Gilmore, 1984), O and C atoms from *DIRDIF* (Beurskens *et al.*, 1984); Fe, O, C atoms anisotropic; hydroxyl-H atom included at position from difference map without refinement, other H atoms at calculated riding positions, $B(H) = 1.2B_{eq}(C)$; full-matrix refinement on F, 118 parameters, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F)$, $\sigma(F)$ from counting statistics + pF^2, $p = 0.03$; $R_F = 0.039$, $wR_F = 0.047$, $S = 1.57$, max. $\Delta/\sigma = 0.02$ in last cycle, max., min. $\Delta\rho$ + 0.56 (near origin), −0.37 e Å^{-3}. All calculations from *TEXSAN* (Molecular Structure Corporation, 1989), with atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, and bond lengths and selected bond angles in Table 2. The molecular structure is shown in Fig. 1 and the hydrogen-bond network in Fig. 2.†

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Fe(1)	0·47818 (4)	0·43548 (4)	0·54996 (4)	2·67 (3)
O(1)	0·6433 (2)	0·6122 (2)	0·5870 (2)	4·3 (1)
O(2)	0·3543 (3)	0·4810 (3)	0·6342 (2)	5·8 (2)
O(3)	0·1716 (3)	0·1409 (3)	0·5115 (3)	6·5 (2)
C(1)	0·5790 (3)	0·5631 (3)	0·5466 (3)	3·2 (2)
C(2)	0·4039 (3)	0·4633 (3)	0·6025 (3)	3·7 (2)
C(3)	0·5140 (3)	0·3387 (3)	0·5024 (3)	3·5 (2)
C(4)	0·5836 (3)	0·3990 (3)	0·5562 (3)	4·0 (2)
C(5)	0·5441 (4)	0·3889 (3)	0·6303 (3)	4·2 (2)
C(6)	0·4491 (3)	0·3221 (3)	0·6231 (3)	3·8 (2)
C(7)	0·4297 (3)	0·2890 (3)	0·5442 (3)	3·4 (2)
C(8)	0·3395 (3)	0·2141 (3)	0·5107 (3)	4·2 (2)
C(9)	0·2547 (4)	0·2035 (4)	0·5515 (4)	5·1 (2)

Related literature. The title compound is an intermediate in the synthesis of polymers with Fe—Fe bonds in the backbone (Tenhaeff & Tyler, 1991). The Fe—Co—Fe bridge is slightly asymmetric compared with that in the parent *trans*-$Fe_2(CO)_4(C_5H_5)_2$ (Bryan & Greene, 1970), but other dimensions are not significantly affected. Intermolecular hydrogen bonds are also present in $Mo_2(CO)_6(C_5H_4CH_2CH_2OH)_2$ (Tenhaeff, Tyler & Weakley, 1991).

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† Lists of structure factors, full bond angles, anisotropic thermal parameters, H-atom coordinates, intermolecular distances, torsion angles, and mean plane information have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54449 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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