SHORT-FORMAT PAPERS

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Acta Cryst. (1992). C**48**, 154–155

Hexacarbonylbis[η^5-(methoxycarbonyl)cyclopentadienyl]ditungsten(*W–W*), [$W_2(C_5H_4CO_2CH_3)_2(CO)_6$]

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Abstract. $C_{20}H_{14}O_{10}W_2$, $M_r = 782.0$, monoclinic, $P2_1/n$, $a = 9.825$ (3), $b = 12.101$ (3), $c = 9.980$ (3) Å, $\beta = 110.14$ (2)°, $V = 1114$ (1) Å^3, $Z = 2$, $D_x = 2.331$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 105.9$ cm^{-1}, $F(000) = 724$, $T = 292$ K, $R_F = 0.034$ for 1468 independent reflections and 145 parameters. The molecule lies on a crystallographic inversion center with an *anti* arrangement of the cyclopentadienyl (Cp) rings. The length of the unbridged W—W bond is 3.216 (1) Å. The dihedral angle between the mean planes of a Cp ring and the adjacent C—C(O)—O—C group is 7 (1)°, and the Cp—COOMe bond is ~ 135° from the W—W bond in a projection normal to the Cp plane.

Experimental. Preparation: Avey, Tenhaeff, Weakley & Tyler (1991). Dark red lath, 0.10 × 0.06 × 0.26 mm, from *n*-hexane/thf, attached to a fiber and coated lightly with diluted polystyrene cement; Rigaku AFC6R diffractometer, graphite monochromator; initial cell dimensions and orientation matrix from setting angles for 25 centered reflections in range $11 \leq 2\theta \leq 18$°, improved values by refinement of 20 strong reflections after data collection in shell $25 \leq 2\theta \leq 30$°; ω–2θ scans, 16° min^{-1} on ω, width $(1.10 + 0.30\tan\theta)$°, $2\theta_{\max} = 50$°, h 0 to 11, k 0 to 14, l − 11 to 11, 3 standard reflections measured every 300 (no change); 2078 independent reflections, 1468 observed [$I \geq 3\sigma(I)$]. Heavy-atom method; absorption correction by use of *DIFABS* (Walker & Stuart, 1983) after isotropic refinement (relative correction factors 0.82 to 1.50), subsequently W, O and C atoms anisotropic, H riding [$U(\mathrm{H}) = 1.2\, U_{\mathrm{eq}}(\mathrm{C})$]; full-matrix refinement on F, $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F)$, $\sigma(F)$ from counting statistics, $p = 0.03$; $R_F = 0.034$, $wR_F = 0.041$, $S = 1.30$, max. $\Delta/\sigma = 0.02$ in last cycle, max., min. $\Delta\rho$ 0.77 (near W), -0.93 e Å^{-3}. All calculations by use of *TEXSAN* (Molecular Structure Corporation, 1989), with atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148). 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 with atom labelling is shown in Fig. 1.†

Table 1. *Atomic coordinates* (W × 10^5; O, C × 10^4) *and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	B_{eq}
W(1)	35866 (4)	42445 (3)	47435 (4)	2.98 (2)
O(1)	6120 (9)	3080 (7)	7153 (9)	5.1 (4)
O(2)	4218 (1)	4230 (7)	1885 (9)	5.6 (4)
O(3)	3169 (14)	1755 (8)	3857 (12)	8.6 (6)
O(4)	−307 (10)	2886 (8)	2925 (10)	6.3 (5)
O(5)	−287 (9)	4396 (7)	1618 (9)	5.3 (4)
C(1)	5264 (12)	3541 (10)	6240 (12)	3.8 (5)
C(2)	4042 (12)	4239 (10)	2971 (12)	4.0 (5)
C(3)	3361 (16)	2663 (11)	4198 (15)	6.1 (6)
C(4)	1611 (13)	4066 (10)	5499 (13)	4.5 (6)
C(5)	2485 (12)	4929 (11)	6334 (12)	4.3 (5)
C(6)	2489 (12)	5829 (10)	5429 (13)	4.0 (5)
C(7)	1663 (11)	5535 (9)	4007 (12)	3.6 (5)
C(8)	1119 (11)	4440 (9)	4039 (12)	3.4 (4)
C(9)	117 (12)	3807 (11)	2844 (13)	4.2 (5)
C(10)	−1357 (18)	3894 (14)	337 (16)	7.6 (8)

Related literature. The title compound is an intermediate in the synthesis of the corresponding water-

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† Lists of structure factors, bond angles, intermolecular distances, anisotropic thermal parameters, H-atom coordinates, torsion angles, mean plane information, a packing diagram and a stereo figure have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54450 (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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Table 2. *Bond lengths* (Å) *and selected bond angles* (°)

W(1)—C(1)	1.994 (11)	O(3)—C(3)	1.146 (14)
W(1)—C(2)	1.968 (12)	O(4)—C(9)	1.201 (14)
W(1)—C(3)	1.982 (14)	O(5)—C(9)	1.353 (14)
W(1)—C(4)	2.322 (11)	O(5)—C(10)	1.477 (16)
W(1)—C(5)	2.357 (11)	C(4)—C(5)	1.425 (16)
W(1)—C(6)	2.412 (11)	C(4)—C(8)	1.441 (15)
W(1)—C(7)	2.365 (11)	C(5)—C(6)	1.415 (16)
W(1)—C(8)	2.293 (10)	C(6)—C(7)	1.417 (15)
O(1)—C(1)	1.149 (12)	C(7)—C(8)	1.434 (14)
O(2)—C(2)	1.155 (14)	C(8)—C(9)	1.473 (16)
W(1)—W(1^i)	3.216 (1)		

C(1)—W(1)—C(2)	106.6 (5)	C(6)—C(7)—C(8)	107 (1)
C(1)—W(1)—C(3)	77.2 (5)	C(4)—C(8)—C(7)	108 (1)
C(2)—W(1)—C(3)	77.8 (6)	C(4)—C(8)—C(9)	124 (1)
W(1)—C(1)—O(1)	172 (1)	C(7)—C(8)—C(9)	128 (1)
W(1)—C(2)—O(2)	176 (1)	O(4)—C(9)—O(5)	123 (1)
W(1)—C(3)—O(3)	177 (1)	O(4)—C(9)—C(8)	125 (1)
C(5)—C(4)—C(8)	107 (1)	O(5)—C(9)—C(8)	111 (1)
C(4)—C(5)—C(6)	109 (1)	C(9)—O(5)—C(10)	118 (1)
C(5)—C(6)—C(7)	109 (1)		

Symmetry code: (i) $1 - x$, $1 - y$, $1 - z$.

soluble carboxylic acid [W—W 3.215 (3) Å] (Avey, Tenhaeff, Weakley & Tyler, 1991) and of polyesters with W—W bonds in the backbone (Tenhaeff & Tyler, 1991). The W—W bond lengths in related compounds include: $W_2(C_5H_5)_2(CO)_6$, 3.222 (1) Å (Adams, Collins & Cotton, 1974); $W_2(C_5Me_5)_2(CO)_6$, 3.288 (1) Å (Rheingold & Harper, 1991).

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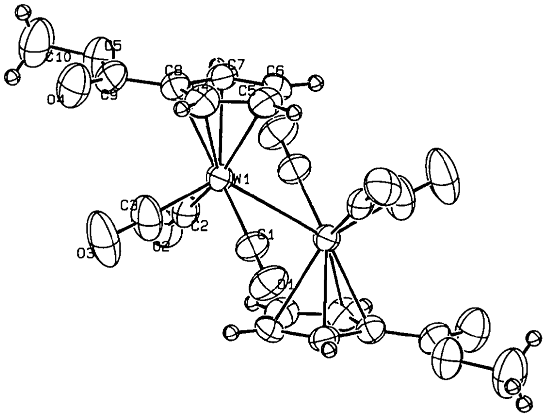

Fig. 1. Molecular structure of $W_2(\eta^5\text{-}C_5H_4COOCH_3)_2(CO)_6$ showing thermal ellipsoids (H atoms, arbitrary radius) and numbering scheme.

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Structure of (μ-Oxalato)-*trans*-bis[*N*,*N*,*N*′,*N*′-tetramethylethylenediamineperfluoro-*tert*-butoxycopper(II)] Benzene Solvate

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Abstract. (μ-Oxalato)-*trans*-bis[*N*,*N*,*N*′,*N*′-tetramethylethylenediamineperfluoro-*tert*-butoxycopper(II)] benzene solvate, $C_{22}H_{32}Cu_2F_{18}N_4O_6.C_6H_6$, $M_r = 995.70$, monoclinic, $C2/m$, $a = 14.581$ (5), $b = 12.484$ (4), $c = 13.239$ (5) Å, $\beta = 123.05$ (2)°, $V = 2019.9$ (12) Å^3, $Z = 2$, $D_x = 1.637$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.18$ mm^{-1}, $F(000) = 1004$, $T = 295$ K, final $R = 0.055$, $wR = 0.067$ for 1629 independent observed reflections. The distorted square-pyramidal five-coordinate Cu^{II} complex and the benzene solvate both have C_{2h} molecular symmetry. The asymmetric unit consists of $\frac{1}{4}$ of the Cu complex and $\frac{1}{4}$ of the solvate molecule. All of the CF_3 groups are poorly defined and the fluorines of one of the crystallographically independent CF_3 groups were treated as a disorder with occupancies of 52 and 48% respectively.

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