

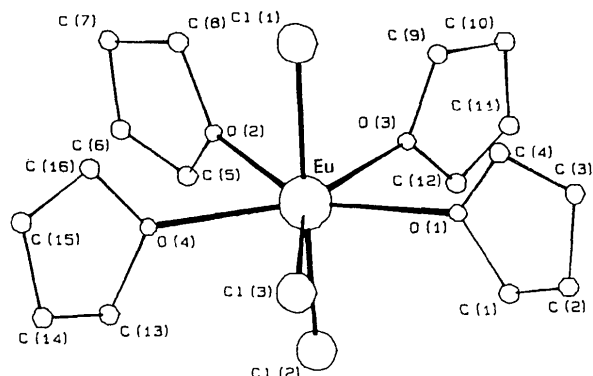
Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} a_i a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
Eu	0.12445 (1)	0.04622 (7)	0.15819 (3)	4.63 (1)
Cl(1)	0.06728 (9)	-0.1826 (4)	0.1026 (2)	7.78 (9)
Cl(2)	0.18124 (9)	0.2750 (4)	0.1903 (2)	7.40 (9)
Cl(3)	0.1238 (1)	0.0797 (6)	0.3070 (2)	11.7 (1)
O(1)	0.0681 (2)	0.238 (1)	0.1329 (4)	7.6 (2)
O(2)	0.1568 (2)	-0.1084 (9)	0.0803 (4)	6.9 (2)
O(3)	0.0957 (2)	0.152 (1)	0.0110 (4)	6.9 (2)
O(4)	0.1771 (2)	-0.141 (1)	0.2509 (4)	7.9 (2)
C(1)	0.0739 (4)	0.339 (2)	0.165 (1)	15.1 (6)
C(2)	0.0381 (4)	0.437 (2)	0.1827 (9)	12.8 (5)
C(3)	0.0065 (4)	0.329 (2)	0.131 (1)	15.1 (6)
C(4)	0.0265 (4)	0.198 (2)	0.113 (1)	16.4 (6)
C(5)	0.1923 (4)	-0.060 (2)	0.0737 (9)	15.6 (5)
C(6)	0.2088 (4)	-0.185 (2)	0.0449 (8)	12.6 (5)
C(7)	0.1779 (4)	-0.326 (2)	0.0218 (8)	13.4 (5)
C(8)	0.1430 (4)	-0.259 (2)	0.0327 (8)	13.3 (5)
C(9)	0.0615 (6)	0.092 (3)	-0.0596 (9)	19.3 (7)
C(10)	0.0484 (5)	0.190 (3)	-0.1312 (8)	14.0 (7)
C(11)	0.0802 (5)	0.315 (2)	-0.1066 (7)	16.7 (6)
C(12)	0.1046 (6)	0.294 (3)	-0.021 (1)	21.8 (8)
C(13)	0.2166 (4)	-0.084 (2)	0.3160 (9)	13.4 (6)
C(14)	0.2269 (5)	-0.193 (3)	0.3826 (8)	14.1 (6)
C(15)	0.1989 (5)	-0.348 (3)	0.3536 (9)	16.9 (7)
C(16)	0.1679 (5)	-0.309 (2)	0.272 (1)	16.0 (7)

Table 2. Interatomic Eu—Cl and Eu—O distances (Å) and bond angles around the Eu atom (°)

Eu—Cl(1)	2.620 (4)	Eu—O(1)	2.430 (8)
Eu—Cl(2)	2.630 (3)	Eu—O(2)	2.486 (8)
Eu—Cl(3)	2.636 (4)	Eu—O(3)	2.481 (6)
Eu—O(4)	2.413 (7)		
Cl(1)—Eu—Cl(2)	171.1 (1)	Cl(3)—Eu—O(1)	75.4 (2)
Cl(1)—Eu—Cl(3)	95.8 (1)	Cl(3)—Eu—O(2)	143.7 (2)
Cl(1)—Eu—O(1)	87.3 (2)	Cl(3)—Eu—O(3)	144.6 (3)
Cl(1)—Eu—O(2)	84.4 (2)	Cl(3)—Eu—O(4)	75.0 (3)
Cl(1)—Eu—O(3)	88.1 (2)	O(1)—Eu—O(2)	140.7 (3)
Cl(1)—Eu—O(4)	92.4 (2)	O(1)—Eu—O(3)	69.7 (2)
Cl(2)—Eu—Cl(3)	93.1 (1)	O(1)—Eu—O(4)	150.2 (3)
Cl(2)—Eu—O(1)	94.2 (2)	O(2)—Eu—O(3)	71.7 (2)
Cl(2)—Eu—O(2)	89.1 (2)	O(2)—Eu—O(4)	68.7 (2)
Cl(2)—Eu—O(3)	84.2 (2)	O(3)—Eu—O(4)	140.2 (3)
Cl(2)—Eu—O(4)	90.8 (3)		

Fig. 1. Molecular diagram of $[\text{EuCl}_3(\text{C}_4\text{H}_8\text{O})_4]$. (H atoms omitted.)

$[\text{TbF}_7]^{3-}$ in the salt $\text{Cs}_3[\text{TbF}_7]$ (Hoppe & Roeder, 1961) have pentagonal bipyramidal geometry. A monocapped octahedron is characteristic of $[\text{Y}(\text{C}_6\text{H}_5\text{COCHCOCH}_3)_3(\text{H}_2\text{O})]$ (Cotton & Legzdins, 1968), while a monocapped trigonal prism is characteristic of $[\text{Y}(\text{CH}_3\text{COCHCOCH}_3)_3(\text{H}_2\text{O})]$ (Cunningham, Sands, Wagner & Richardson, 1969).

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Structure of 2,2,2-Tricarbonyl-1,1-bis(ethylene)- μ -(η^5 : η^5 -fulvalene)-2-methylrhodiumtungsten

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Abstract. $[\text{RhW}(\text{C}_{10}\text{H}_8)(\text{CH}_3)(\text{C}_2\text{H}_4)_2(\text{CO})_3]$, $M_r = 570.10$, monoclinic, $P2_1/c$, $a = 7.3358$ (8), $b = 10.8640$ (15), $c = 21.6310$ (20) Å, $\beta = 90.924$ (8)°, $V = 1723.7$ (3) Å³, $Z = 4$, $D_m = 2.16$, $D_x =$

2.196 g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 77.7$ cm⁻¹, $F(000) = 1080$, $T = 298$ K, $R = 0.021$, $wR = 0.029$ for 4453 observed reflections with $I \geq 3\sigma(I)$. The compound is a bimetallic fulvalene complex with

rhodium and tungsten oriented *trans* to each other across an essentially planar fulvalene dianion.

Experimental. The title compound was prepared by A. Kahn and K.P.C. Vollhardt of this department as part of an investigation of heterobimetallic fulvalene complexes (Kahn, Newman & Vollhardt, 1990). The preparation involved the reaction of the sodium salt of the (fulvalene)tricarbonylmethyltungsten anion with di- μ -chloro-bis[bis(ethylene)rhodium].

Air-stable, golden crystals were prepared by A. Kahn of this department. D_m was measured by flotation in a mixture of tribromomethane and toluene. Sample crystal was a needle of dimensions 0.382 × 0.364 × 0.320 mm. Enraf-Nonius CAD-4 diffractometer; 24 reflections ($21.98 \leq 2\theta \leq 28.92^\circ$) were used to refine cell parameters. Data collection: $3 \leq 2\theta \leq 60^\circ$, index ranges $0 \leq h \leq 10$, $0 \leq k \leq 15$, $-30 \leq l \leq 30$; θ - 2θ scan technique with Mo $K\alpha$ radiation at room temperature. ψ scans: $0 \leq \psi \leq 180^\circ$, 10° step. 5641 total reflections; systematically absent: ($h0l$), $l = 2n + 1$, ($0k0$), $k = 2n + 1$ and redundant: ($0kl$), $l < 0$ were removed; 5017 unique reflections; 4453 reflections with $I \geq 3\sigma(I)$. No indication of crystal decomposition from three standard reflections measured every hour. Empirical absorption correction was performed; minimum relative transmission = 73.19%. Structure solved by Patterson methods and refined *via* standard full-matrix least squares and Fourier techniques with a Digital Equipment Micro-

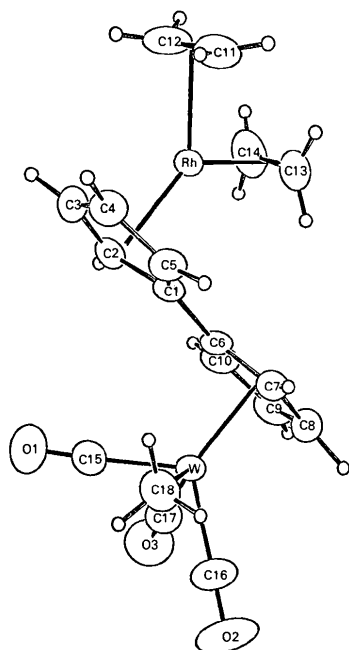


Fig. 1. ORTEP (Johnson, 1976) drawing of the title compound showing the atom-numbering scheme. Ellipsoids are drawn at the 50% probability level.

Table 1. Atomic coordinates and final equivalent isotropic thermal parameters

$$B_{eq} = (1/3) \sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
W	0.26480 (2)	0.23966 (1)	0	2.275 (2)
Rh	0.54641 (3)	0.23955 (2)	0.18359 (1)	2.138 (3)
O1	-0.0265 (4)	0.0650 (3)	0.3589 (1)	5.49 (6)
O2	0.0908 (5)	0.3738 (3)	0.5298 (1)	7.85 (8)
O3	0.2371 (5)	0.0320 (3)	0.5177 (1)	6.44 (8)
C1	0.3735 (4)	0.2397 (2)	0.2672 (1)	2.36 (5)
C2	0.3466 (4)	0.1221 (3)	0.2373 (1)	2.96 (5)
C3	0.2717 (4)	0.1445 (3)	0.1784 (2)	3.52 (6)
C4	0.2503 (4)	0.2742 (3)	0.1709 (2)	3.56 (7)
C5	0.3051 (4)	0.3329 (3)	0.2265 (1)	2.90 (5)
C6	0.4438 (4)	0.2582 (2)	0.3300 (1)	2.47 (5)
C7	0.4547 (4)	0.3728 (3)	0.3621 (1)	2.88 (5)
C8	0.5398 (4)	0.3535 (3)	0.4201 (1)	3.60 (6)
C9	0.5825 (5)	0.2278 (3)	0.4255 (2)	3.81 (7)
C10	0.5242 (4)	0.1683 (3)	0.3699 (1)	3.15 (6)
C11	0.6403 (5)	0.3230 (4)	0.1013 (Z)	4.45 (8)
C12	0.6374 (6)	0.1966 (4)	0.0934 (2)	4.98 (8)
C13	0.7889 (4)	0.3026 (3)	0.2281 (2)	3.52 (6)
C14	0.7995 (4)	0.1775 (3)	0.2203 (2)	4.33 (8)
C15	0.0768 (4)	0.1307 (3)	0.3816 (1)	3.33 (6)
C16	0.1541 (5)	0.3250 (4)	0.4894 (2)	4.48 (7)
C17	0.2471 (5)	0.1076 (3)	0.4808 (1)	3.83 (7)
C18	0.0478 (4)	0.3677 (3)	0.3772 (2)	3.95 (7)

Table 2. Selected interatomic distances (\AA) and angles ($^\circ$)

Rh—C(1)	2.225 (2)	Rh—C(14)	2.118 (3)
Rh—C(2)	2.276 (2)	C(15)—O(1)	1.146 (4)
Rh—C(3)	2.266 (3)	C(16)—O(2)	1.129 (4)
Rh—C(4)	2.217 (3)	C(17)—O(3)	1.148 (3)
Rh—C(5)	2.253 (2)	C(1)—C(2)	1.444 (3)
W—C(6)	2.346 (2)	C(1)—C(5)	1.428 (3)
W—C(7)	2.360 (3)	C(1)—C(6)	1.459 (4)
W—C(8)	2.366 (3)	C(2)—C(3)	1.401 (4)
W—C(9)	2.337 (3)	C(3)—C(4)	1.426 (4)
W—C(10)	2.320 (3)	C(4)—C(5)	1.414 (4)
W—C(15)	1.975 (3)	C(6)—C(7)	1.428 (3)
W—C(16)	1.981 (3)	C(6)—C(10)	1.426 (4)
W—C(17)	1.977 (3)	C(7)—C(8)	1.408 (4)
W—C(18)	2.284 (3)	C(8)—C(9)	1.405 (4)
Rh—C(11)	2.123 (3)	C(9)—C(10)	1.425 (4)
Rh—C(12)	2.124 (3)	C(11)—C(12)	1.384 (6)
Rh—C(13)	2.123 (3)	C(13)—C(14)	1.373 (5)
Cp*(2)—W—C(15)	125.4	C(1)—C(2)—C(3)	107.60 (25)
Cp(2)—W—C(16)	126.4	C(2)—C(3)—C(4)	108.43 (24)
Cp(2)—W—C(17)	118.8	C(3)—C(4)—C(5)	108.63 (25)
Cp(2)—W—C(18)	110.2	C(1)—C(5)—C(4)	107.28 (25)
Cp(1)—Rh—Ct†(1)	132.9	C(1)—C(6)—C(7)	126.15 (22)
Cp(1)—Rh—Ct(2)	132.0	C(6)—C(7)—C(8)	108.9 (3)
W—C(15)—O(1)	177.0 (3)	C(7)—C(8)—C(9)	108.3 (3)
W—C(16)—O(2)	180.0 (5)	C(1)—C(6)—C(10)	127.32 (22)
W—C(17)—O(3)	179.1 (3)	C(7)—C(6)—C(10)	106.45 (23)
C(2)—C(1)—C(5)	107.87 (23)	C(8)—C(9)—C(10)	107.94 (25)
C(2)—C(1)—C(6)	125.64 (22)	C(6)—C(10)—C(9)	108.4 (3)
C(5)—C(1)—C(6)	126.29 (21)		

*Cp corresponds to the centroid of the cyclopentadienyl rings.

†Ct corresponds to the center of the ethylene carbon-carbon double bonds.

VAX computer using locally modified Nonius SDP software (Frenz, 1985). Function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F_o)$, $\sigma(F_o) = [\sigma_o^2(F_o) + p(F_o^2)^2]^{1/2}$; $p = 0.03$. All non-H atoms were refined with anisotropic thermal parameters. H atoms,

except the disordered methyl protons, were located on a difference Fourier map and refined using isotropic thermal parameters. Final number of parameters refined = 273, $R = 0.021$, $wR = 0.029$, $S = 1.446$; secondary-extinction coefficient (Zachariassen, 1963) was included and refined to 2.548×10^{-7} . Maximum shift/e.s.d. on final cycle = 0.05; largest peak on final difference Fourier map = $0.74 \text{ e } \text{Å}^{-3}$. Atomic scattering factors and anomalous corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV).

A drawing of the title compound with the atom-numbering scheme is shown in Fig. 1. Atomic coordinates and final equivalent isotropic thermal parameters of all non-H atoms included in the refinement are given in Table 1.* Selected interatomic distances and angles are listed in Table 2.

* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53442 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monosodium Phosphoenolpyruvate Hydrate: New Diffractometer Data

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Abstract. $\text{Na}^+\cdot\text{C}_3\text{H}_4\text{O}_6\text{P}^-\cdot\text{H}_2\text{O}$, $M_r = 208.04$, monoclinic, Cc , $a = 11.426$ (7), $b = 23.274$ (22), $c = 6.620$ (5) Å, $\beta = 123.66$ (5)°, $V = 1465$ (3) Å³, $Z = 8$, $D_x = 1.886$ (3) g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 4.44$ cm⁻¹, $F(000) = 848$, $T = 303$ (2) K, final $R = 0.034$ for 3357 observed data including both hkl and $\bar{h}\bar{k}\bar{l}$ reflections. The two crystallographically independent anions have slightly different configurations with P—O(ester) bond lengths of 1.606 (3) and 1.618 (3) Å.

Experimental. The structure of sodium phosphoenolpyruvate hydrate was determined by Katti, Hosur & Viswamitra (1981) from 1697 diffractometer data with final $R = 0.063$. A fragment of $0.25 \times 0.2 \times 0.5$ mm was cut from a larger crystal and mounted on a Syntex $P2_1$ diffractometer. Mo $K\alpha$ radiation and graphite monochromator were used for lattice parameters (15 reflections in the range $24 < 2\theta < 27^\circ$) and intensity measurements. 5984 intensities were

The fulvalene ligand is essentially planar with only a 6° twist about the bond connecting C1 and C6. The metals adopt the *trans* orientation found in all fulvalene complexes in which there is no $M-M$ bond, to minimize steric interactions between the respective ligand coordination spheres.

This crystal structure was solved at the X-ray Crystallographic Facility (CHEXRAY) at the Department of Chemistry, University of California, Berkeley, as part of the requirements of a graduate chemistry course on structure analysis by X-ray diffraction. We thank Mr Tim Karpishin, Dr F. Hollander and Professor K. N. Raymond for their guidance.

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 ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

measured, $2\theta \leq 65^\circ$, $h - 10 \rightarrow 17$, $k 0 \rightarrow 35$, $l - 9 \rightarrow 9$, from $\omega/2\theta$ scans. After each group of 50 reflections two standards were measured; variation $\pm 5\%$. Scattering factors for Na⁺, P, C, O and H were from *International Tables for X-ray Crystallography* (1974, Vol. IV); real and imaginary dispersion corrections included for all non-H atoms. No absorption or extinction corrections. The refinement was started with the published coordinates of Katti *et al.* (1981). Merging in point group m yielded 3871 reflections, 3357 with $I > 3\sigma(I)$ (including 1212 Friedel pairs) were used for refinement; $R_{\text{merge}} = 0.017$. Full-matrix refinement on F (Na, P, C, O anisotropic, H isotropic) with $w = 1/\sigma^2(F_o)$ using *SHELX76* (Sheldrick, 1976) gave final $R = 0.0332$ and $wR = 0.0307$ (refinement of the inverted structure gave $R = 0.0337$ and $wR = 0.0311$); max $\Delta/\sigma = 0.05$. The positional parameters of the H atoms were refined with constraints $d(\text{C—H}) = 1.08$ and $d(\text{O—H}) = 0.97$ Å. Residual electron density in final difference map