

S11—C12—S15	122 (1)	C1F—C1G—C1H	108 (2)
S11—C12—C11	121 (2)	C1G—C1H—C1I	110 (2)
S15—C12—C11	117 (2)	C1H—C1I—C1J	106 (2)
S12—C11—S16	122 (1)	C1F—C1J—C1I	109 (2)
S12—C11—C12	123 (2)		

Table 3. Contact distances (<3.7 Å) between anions

S11...S22	3.65 (1)	S27...S43	3.60 (1)
S11...S26	3.40 (1)	S29...S45	3.62 (1)
S15...S26	3.53 (1)	S30...S46	3.62 (1)
S16...S29	3.66 (1)	S31...S48	3.63 (1)
S18...S24	3.57 (1)	S32...S43	3.66 (1)
S10...S40	3.32 (1)	S32...S47	3.64 (1)
S28...S37	3.64 (1)	S36...S41	3.51 (1)
S29...S30	3.53 (1)	S36...S43	3.49 (1)
S20...S40	3.62 (1)		

Data collection: AFD (Rigaku Corporation, 1985a). Cell refinement: AFD. Data reduction: RCRYSTAN (Rigaku Corporation, 1985b). Program(s) used to solve and refine structure: SDP (Frenz, 1978). Computer: VAX 11/785. Refinement was by the full-matrix least-squares method. H atoms were located and included in the structure-factor calculations but their positions were not refined.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete bond distances and angles, and least-squares-planes data, as well as packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71004 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1014]

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Structure of Tricarbonyltris(diphenylphosphine)molybdenum(0) Hexane Solvate: *fac*-[Mo(CO)₃(PPh₂H)₃]·0.25C₆H₁₄

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Abstract

The two independent molecules in the asymmetric unit confirm a *fac*-octahedral metal geometry with bond distances Mo—P (mean) 2.502 (4) and Mo—C (mean) 1.964 (12) Å.

Comment

Two other *fac*-Mo(CO)₃(triphosphine) structures have been reported: Mo(CO)₃(Ph₂P.NEt.PPh₂.NEt.PPh₂) with Mo—P (mean) 2.476 and Mo—C

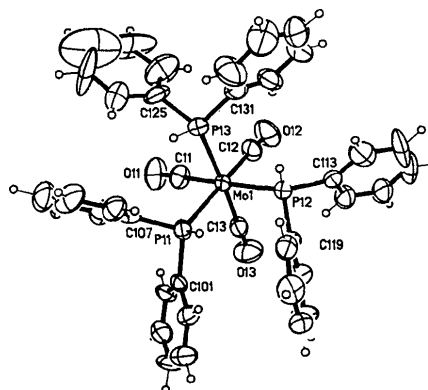


Fig. 1. Perspective view of the title compound (molecule 1) showing the atomic numbering. The H atoms are represented by spheres of arbitrary size. Non-H atoms are represented with 50% probability ellipsoids for thermal motion.

(mean) 1.95 Å (Cheung, Lai & Lam, 1970), and Mo(CO)₃[Ph₂P.(CH₂)₂.PPh.(CH₂)₂.PPh₂] with Mo—P (mean) 2.505 and Mo—C (mean) 1.96 Å (Favas, Kepert, Skelton & White, 1980).

Experimental

Crystal data

[Mo(C₁₂H₁₁P)₃(CO)₃].-

0.25(C₆H₁₄)

M_r = 738.50

Monoclinic

*P*2₁/*n*

a = 18.049 (6) Å

b = 17.661 (8) Å

c = 23.475 (10) Å

β = 92.10 (3)°

V = 7477.94 Å³

Z = 8

D_x = 1.312 Mg m⁻³

Mo *Kα* radiation

λ = 0.71069 Å

Cell parameters from 29 reflections

θ = 2–11°

μ = 0.51 mm⁻¹

T = 290 K

Block

0.37 × 0.34 × 0.12 mm

Colourless

Data collection

Siemens *R3m* diffractometer

ω–2θ scans

Absorption correction:

Gaussian method

T_{min} = 0.826, *T_{max}* =

0.938

10759 measured reflections

9826 independent reflections

5878 observed reflections

[*I*/σ(*I*) ≥ 2.0]

R_{int} = 0.0426

θ_{max} = 45°

h = 0 → 19

k = 0 → 19

l = –25 → 25

3 standard reflections

monitored every 200

reflections

intensity variation: slight decrease

Refinement

Refinement on *F*

Final *R* = 0.062

wR = 0.066

S = 1.1159

5878 reflections

859 parameters

w = 1/[σ²(*F*) + 0.0034*F*²]

(Δ/σ)_{max} = 0.078

Δρ_{max} = 1.3 e Å⁻³

Δρ_{min} = –0.4 e Å⁻³

Atomic scattering factors

from *International Tables*

for *X-ray Crystallography*

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å²)

*U*_{eq} is defined as one third of the trace of the orthogonalized *U*_{ij} tensor.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Mo(1)	0.85730 (5)	0.26429 (6)	0.13626 (4)	0.035 (1)
P(11)	0.98724 (16)	0.28688 (18)	0.10659 (12)	0.038 (1)
P(12)	0.90957 (17)	0.22953 (19)	0.23235 (12)	0.042 (1)
P(13)	0.87953 (19)	0.12973 (19)	0.10764 (14)	0.048 (1)
O(11)	0.7855 (6)	0.3095 (7)	0.0189 (4)	0.090 (5)
O(12)	0.6953 (5)	0.2439 (7)	0.1714 (4)	0.088 (5)
O(13)	0.8328 (6)	0.4331 (6)	0.1681 (5)	0.086 (5)
C(11)	0.8139 (7)	0.2909 (7)	0.0626 (5)	0.053 (5)
C(12)	0.7565 (7)	0.2474 (8)	0.1593 (5)	0.057 (5)
C(13)	0.8448 (7)	0.3694 (8)	0.1571 (5)	0.047 (5)
C(101)	1.0227 (6)	0.3819 (7)	0.1070 (4)	0.036 (4)
C(102)	1.0894 (7)	0.3991 (7)	0.1354 (5)	0.050 (5)
C(103)	1.1186 (7)	0.4717 (8)	0.1336 (6)	0.064 (6)
C(104)	1.0809 (8)	0.5278 (8)	0.1040 (6)	0.065 (6)
C(105)	1.0144 (8)	0.5135 (8)	0.0767 (5)	0.064 (6)
C(106)	0.9862 (6)	0.4395 (7)	0.0784 (5)	0.045 (5)
C(107)	1.0151 (6)	0.2496 (6)	0.0382 (5)	0.044 (4)
C(108)	0.9811 (7)		0.2758 (8)	–0.0125 (5)
C(109)	1.0054 (8)		0.2507 (9)	–0.0653 (6)
C(110)	1.0587 (9)		0.1996 (10)	–0.0676 (7)
C(111)	1.0936 (8)		0.1721 (9)	–0.0195 (8)
C(112)	1.0718 (7)		0.1978 (8)	0.0348 (6)
C(113)	0.8504 (7)		0.2323 (8)	0.2934 (5)
C(114)	0.8470 (8)		0.1720 (9)	0.3317 (6)
C(115)	0.8027 (9)		0.1786 (13)	0.3795 (7)
C(116)	0.7662 (9)		0.2425 (14)	0.3897 (6)
C(117)	0.7685 (8)		0.3013 (10)	0.3537 (6)
C(118)	0.8099 (7)		0.2961 (9)	0.3055 (6)
C(119)	0.9929 (6)		0.2785 (6)	0.2587 (5)
C(120)	0.9904 (7)		0.3532 (8)	0.2710 (5)
C(121)	1.0536 (7)		0.3923 (8)	0.2894 (6)
C(122)	1.1204 (7)		0.3572 (8)	0.2949 (5)
C(123)	1.1240 (7)		0.2830 (9)	0.2813 (6)
C(124)	1.0606 (7)		0.2433 (8)	0.2640 (5)
C(125)	0.8413 (10)		0.0938 (8)	0.0403 (6)
C(126)	0.8892 (12)		0.0788 (11)	–0.0040 (7)
C(127)	0.8547 (20)		0.0528 (19)	–0.0552 (9)
C(128)	0.7869 (35)		0.0414 (35)	–0.0680 (18)
C(129)	0.7469 (23)		0.0515 (21)	–0.0242 (14)
C(130)	0.7711 (12)		0.0815 (11)	0.0319 (8)
C(131)	0.8625 (8)		0.0543 (8)	0.1583 (6)
C(132)	0.8075 (8)		0.0575 (8)	0.1950 (6)
C(133)	0.7938 (10)		0.0015 (10)	0.2340 (7)
C(134)	0.8413 (14)		–0.0612 (13)	0.2364 (8)
C(135)	0.8887 (14)		–0.0677 (11)	0.2000 (9)
C(136)	0.9093 (10)		–0.0083 (9)	0.1607 (8)
Mo(2)	0.34670 (5)		0.19835 (6)	0.13213 (4)
P(21)	0.47897 (16)		0.18361 (18)	0.10568 (12)
P(22)	0.39234 (17)		0.22602 (18)	0.23152 (12)
P(23)	0.35605 (17)		0.33654 (18)	0.10733 (13)
O(21)	0.3338 (5)		0.0293 (5)	0.1658 (4)
O(22)	0.2843 (5)		0.1466 (5)	0.0114 (4)
O(23)	0.1811 (5)		0.2034 (6)	0.1649 (4)
C(21)	0.3397 (6)		0.0897 (8)	0.1533 (5)
C(22)	0.3093 (6)		0.1678 (7)	0.0538 (5)
C(23)	0.2419 (7)		0.2039 (7)	0.1543 (5)
C(201)	0.5176 (6)		0.0879 (7)	0.1077 (5)
C(202)	0.4822 (7)		0.0328 (7)	0.0768 (5)
C(203)	0.5131 (9)		–0.0409 (8)	0.0744 (5)
C(204)	0.5775 (8)		–0.0552 (8)	0.1033 (6)
C(205)	0.6118 (8)		–0.0001 (9)	0.1347 (7)
C(206)	0.5837 (7)		0.0718 (8)	0.1385 (5)
C(207)	0.5115 (6)		0.2146 (7)	0.0359 (5)
C(208)	0.5855 (7)		0.2155 (9)	0.0279 (6)
C(209)	0.6087 (9)		0.2326 (12)	–0.0269 (8)
C(210)	0.5609 (10)		0.2490 (10)	–0.0716 (6)
C(211)	0.4887 (8)		0.2469 (8)	–0.0611 (5)
C(212)	0.4626 (7)		0.2310 (7)	–0.0085 (4)
C(213)	0.4783 (6)		0.1809 (7)	0.2574 (4)
C(214)	0.5423 (7)		0.2239 (8)	0.2659 (5)
C(215)	0.6064 (7)		0.1912 (11)	0.2862 (6)
C(216)	0.6055 (8)		0.1137 (11)	0.2998 (6)
C(217)	0.5429 (8)		0.0712 (10)	0.2921 (6)
C(218)	0.4804 (7)		0.1050 (7)	0.2708 (5)
C(219)	0.3310 (6)		0.2072 (7)	0.2910 (4)
C(220)	0.2888 (6)		0.1449 (8)	0.2889 (5)
C(221)	0.2471 (7)		0.1261 (8)	0.3363 (5)
C(222)	0.2497 (8)		0.1738 (9)	0.3834 (6)
C(223)	0.2920 (8)		0.2380 (10)	0.3847 (6)
C(224)	0.3338 (7)		0.2551 (8)	0.3374 (5)
C(225)	0.2903 (6)		0.3823 (7)	0.0562 (5)
C(226)	0.3022 (7)		0.4545 (8)	0.0375 (6)
C(227)	0.2540 (9)		0.4867 (9)	–0.0016 (7)
C(228)	0.1931 (8)		0.4496 (10)	–0.0218 (6)
C(229)	0.1793 (7)		0.3786 (9)	–0.0021 (6)
C(230)	0.2274 (7)		0.3441 (7)	0.0365 (5)
C(231)	0.3594 (6)		0.4038 (6)	0.1668 (5)
C(232)	0.3026 (7)		0.4081 (7)	0.2056 (6)
C(233)	0.3036 (11)		0.4539 (9)	0.2525 (6)
C(234)	0.3612 (12)		0.4973 (11)	0.2631 (7)
C(235)	0.4191 (11)		0.4968 (10)	0.2267 (8)
C(236)	0.4202 (8)		0.4490 (9)	0.1768 (6)
C(1)	0.5209 (15)		0.5380 (16)	0.0451 (12)
C(2)	0.4844 (16)		0.5556 (17)	0.0087 (13)
C(3)	0.5532 (22)		0.4597 (25)	0.0412 (17)

Table 2. Selected bond lengths (Å) and angles (°)

Mo(1)—P(11)	2.503 (3)	Mo(1)—P(12)	2.490 (3)
Mo(1)—P(13)	2.506 (4)	Mo(1)—C(11)	1.930 (12)
Mo(1)—C(12)	1.940 (13)	Mo(1)—C(13)	1.935 (13)
P(11)—C(101)	1.796 (12)	P(11)—C(107)	1.822 (12)
P(12)—C(113)	1.820 (12)	P(12)—C(119)	1.824 (11)
P(13)—C(125)	1.815 (15)	P(13)—C(131)	1.820 (14)
O(11)—C(11)	1.176 (15)	O(12)—C(12)	1.152 (16)
O(13)—C(13)	1.176 (17)	Mo(2)—P(21)	2.502 (3)
Mo(2)—P(22)	2.494 (3)	Mo(2)—P(23)	2.516 (4)
Mo(2)—C(21)	1.988 (14)	Mo(2)—C(22)	2.009 (12)
Mo(2)—C(23)	1.982 (12)	P(21)—C(201)	1.829 (12)
P(21)—C(207)	1.844 (11)	P(22)—C(213)	1.829 (11)
P(22)—C(219)	1.843 (11)	P(23)—C(225)	1.843 (12)
P(23)—C(231)	1.833 (11)	O(21)—C(21)	1.112 (17)
O(22)—C(22)	1.140 (14)	O(23)—C(23)	1.135 (15)
P(11)—Mo(1)—P(12)	87.9 (1)	P(11)—Mo(1)—P(13)	85.2 (1)
P(12)—Mo(1)—P(13)	87.1 (1)	P(11)—Mo(1)—C(11)	93.8 (4)
P(12)—Mo(1)—C(11)	178.3 (4)	P(13)—Mo(1)—C(11)	93.1 (4)
P(11)—Mo(1)—C(12)	179.7 (4)	P(12)—Mo(1)—C(12)	92.2 (4)
P(13)—Mo(1)—C(12)	95.2 (4)	C(11)—Mo(1)—C(12)	86.2 (5)
P(11)—Mo(1)—C(13)	92.1 (4)	P(12)—Mo(1)—C(13)	93.0 (4)
P(13)—Mo(1)—C(13)	177.3 (4)	C(11)—Mo(1)—C(13)	86.8 (5)
C(12)—Mo(1)—C(13)	87.6 (6)	Mo(1)—P(11)—C(101)	119.0 (4)
Mo(1)—P(11)—C(107)	118.6 (4)	C(101)—P(11)—C(107)	103.5 (5)
Mo(1)—P(12)—C(113)	119.6 (4)	Mo(1)—P(12)—C(119)	118.1 (4)
C(113)—P(12)—C(119)	102.7 (5)	Mo(1)—P(13)—C(125)	120.4 (5)
Mo(1)—P(13)—C(131)	119.1 (5)	C(125)—P(13)—C(131)	104.2 (6)
Mo(1)—C(11)—O(11)	177.0 (11)	Mo(1)—C(12)—O(12)	173.8 (12)
Mo(1)—C(13)—O(13)	175.8 (10)	P(21)—Mo(2)—P(22)	88.1 (1)
P(21)—Mo(2)—P(23)	88.3 (1)	P(22)—Mo(2)—P(23)	90.2 (1)
P(21)—Mo(2)—C(21)	91.8 (3)	P(22)—Mo(2)—C(21)	88.7 (3)
P(23)—Mo(2)—C(21)	178.9 (3)	P(21)—Mo(2)—C(22)	92.1 (3)
P(22)—Mo(2)—C(22)	175.7 (3)	P(23)—Mo(2)—C(22)	94.1 (3)
C(21)—Mo(2)—C(22)	87.0 (5)	P(21)—Mo(2)—C(23)	176.7 (4)
P(22)—Mo(2)—C(23)	91.7 (3)	P(23)—Mo(2)—C(23)	94.9 (4)
C(21)—Mo(2)—C(23)	84.9 (5)	C(22)—Mo(2)—C(23)	87.8 (5)
Mo(2)—P(21)—C(201)	117.1 (4)	Mo(2)—P(21)—C(207)	121.9 (4)
C(201)—P(21)—C(207)	99.5 (5)	Mo(2)—P(22)—C(213)	118.2 (4)
Mo(2)—P(22)—C(219)	119.1 (4)	C(213)—P(22)—C(219)	101.2 (5)
Mo(2)—P(23)—C(225)	122.0 (4)	Mo(2)—P(23)—C(231)	116.9 (4)
C(225)—P(23)—C(231)	102.4 (5)	Mo(2)—C(21)—O(21)	178.0 (10)
Mo(2)—C(22)—O(22)	174.5 (10)	Mo(2)—C(23)—O(23)	175.9 (10)

The title compound was prepared by direct treatment of $C_7H_8Mo(CO)_3$ (C_7H_8 = cycloheptatriene) (Cotton, McCleverty & White, 1967) with three equivalents of PPh_2H in boiling toluene for 12 h, followed by recrystallization from toluene/hexane. A crystal was mounted in a Lindemann capillary. Data were collected with a scan range of $\pm 0.7^\circ$ (ω) around the $K\alpha_1-K\alpha_2$ angles, scan speed $2.5-15^\circ$ (ω) min^{-1} , depending on the intensity of a 2 s pre-scan; backgrounds were measured at each end of the scan for 0.25 of the scan time. The data were processed using profile analysis and then corrected for Lorentz, polarization and absorption effects; crystal bounding faces $\pm(001)$ $d = 0.06$; $\pm(110)$ $d = 0.19$; $\pm(1\bar{1}0)$ $d = 0.17$. Systematic reflection conditions $h0l$, $h + 1 = 2n$, and $0k0$, $k = 2n$, indicate space group $P2_1/n$. The structure was solved by direct methods using *SHELXTL-Plus* (*TREF*) (Sheldrick, 1986) to locate two independent Mo atoms, each with three P atoms. The remaining atoms were then found by *E*-map expansion and successive Fourier syntheses. Anisotropic thermal parameters were used for all non-H atoms. H atoms were given fixed isotropic thermal parameters, $U = 0.08 \text{ \AA}^2$. Those defined by the molecular geometry were inserted at calculated positions and not refined. Fourier syntheses near the end of the refinement showed peaks in appropriate positions for H atoms attached to P atoms. Their coordinates were refined with the bond lengths weakly constrained to 1.3 (1) Å. An area of electron density was attributed to a partly occupied hexane molecule disordered across

a centre of inversion. This was modelled as three atoms each with 0.5 occupancy. Final refinement was by least-squares methods. The largest peaks in the final difference Fourier synthesis were in the region of the disordered solvent. All calculations were performed using *SHELXTL-Plus* (Sheldrick, 1986) on a DEC MicroVAX II.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71000 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1041]

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cis-1-Ferrocenyl-2-(2-nitrophenyl)ethylene

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Abstract

The *cis*-ethylene bond environment in (η^5 -cyclopentadienyl){1-[2-(2-nitrophenyl)vinyl]- η^5 -cyclopentadienyl}-iron is not exactly planar; the corresponding torsion angle around the C=C bond is $8.7 (7)^\circ$. The mean planes of the aromatic ring and the ethylene bond form a dihedral angle of $58.9 (1)^\circ$; the NO_2 -group plane is rotated by $32.8 (2)^\circ$ out of the benzene ring. A rather short contact between the olefin CH group and an NO_2 oxygen atom [$H \cdots O 2.72 (4) \text{ \AA}$] may determine the differences in the reactivity of *cis* and *trans* isomers.

Comment

The *cis/trans* isomers of 1-ferrocenyl-2-(2-nitrophenyl)-ethylene are rather stable and do not interconvert upon-