

Fig. 1. PLUTO drawing of [(oep)TiMo(CO)₃Cp].

Kadish (1987), Brothers & Collman (1986) and references therein.

The crystal structures containing a hetero metal-metal bond in the metalloporphyrin series are: [(oep)-SnFe(CO)₄] (Barbe, Guilard, Lecomte & Gerardin, 1984), Sn=Fe = 2.491 (1) Å; [(tetraphenylporphinato)Sn{Mn(CO)₄HgMn(CO)₅}]·0.5CH₂Cl₂ (Onaka *et al.*, 1985), Sn-Mn = 2.554 (3) Å; [(oep)InMn(CO)₃] (Guilard, Mitaine, Moise, Lecomte, Boukhris,

Swistak, Tabard, Lacombe, Cornillon & Kadish, 1987), In-Mn = 2.705 (1) Å; [(oep)RhIn(oep)] (Jones, Carroll & Wayland, 1986), Rh-In = 2.584 (2) Å; [(oep)-TiMn(CO)₅] (Guilard *et al.*, 1988), Ti-Mn = 2.6994 (9) Å; [(oep)InMo(CO)₃Cp] (Lecomte, Habbou, Mitaine, Richard & Guilard, 1989), In-Mo = 2.890 (1) Å.

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Acta Cryst. (1989). **C45**, 1226-1228

Structure of Tricarbonyl(η -cyclopentadienyl)[(2,3,7,8,12,13,17,18-octaethylporphinato)indio(III)]molybdenum(0) at 100 (5) K

BY C. LECOMTE* AND A. HABBOU

Laboratoire de Minéralogie-Cristallographie, UA CNRS 809, Université de Nancy I, BP 239, 54506 Vandoeuvre les Nancy CEDEX, France

AND P. MITAINE, P. RICHARD AND R. GUILARD

Laboratoire de Synthèse et d'Electrosynthèse Organométallique, UA CNRS 33, Faculté des Sciences Gabriel, Université de Bourgogne, 21100 Dijon, France

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Abstract. [InMo(C₅H₅)(C₃₆H₄₄N₄)(CO)₃], [(oep)-InMo(CO)₃Cp], *M_r* = 892.67, triclinic, *P* $\bar{1}$, *a* =

12.679 (5), *b* = 13.895 (5), *c* = 15.239 (8) Å, α = 58.81 (4), β = 59.46 (4), γ = 67.85 (4)°, *V* = 1954.5 Å³, *Z* = 2, *D_x* = 1.516 g cm⁻³, $\lambda(\text{Mo K}\alpha^-)$ = 0.71073 Å, μ = 0.17 cm⁻¹, *F*(000) = 912, *T* = 100 K,

* Author to whom correspondence should be addressed.

Table 1. Positional parameters ($\times 10^4$; $\times 10^5$ for In, Mo) and equivalent isotropic temperature factors and their e.s.d.'s for the non-H atoms

	x	y	z	$B_{eq}^*(\text{\AA}^2)$
In	33854 (2)	32633 (2)	21614 (2)	0.68 (1)
Mo	15852 (2)	19302 (2)	28916 (2)	0.85 (1)
N(1)	5265 (2)	2273 (2)	1756 (2)	0.89 (10)
N(4)	3764 (2)	3162 (2)	3465 (2)	0.85 (11)
N(3)	2534 (2)	5009 (2)	2022 (2)	0.91 (11)
N(2)	4029 (2)	4110 (2)	325 (2)	0.83 (11)
C(1)	5789 (3)	1509 (2)	2519 (3)	0.96 (12)
C(2)	6779 (3)	718 (2)	2078 (3)	0.99 (12)
C(3)	6851 (3)	1024 (2)	1042 (3)	0.99 (12)
C(4)	5910 (3)	2011 (2)	842 (3)	0.95 (12)
C(5)	5706 (3)	2624 (2)	-146 (3)	1.04 (13)
C(6)	4836 (3)	3589 (2)	-389 (2)	0.96 (12)
C(7)	4646 (3)	4184 (2)	-1418 (2)	0.98 (12)
C(8)	3722 (3)	5087 (2)	-1318 (3)	1.07 (13)
C(9)	3351 (3)	5034 (2)	-224 (2)	0.89 (12)
C(10)	2448 (3)	5809 (2)	200 (3)	1.03 (13)
C(11)	2067 (3)	5808 (2)	1234 (3)	0.99 (13)
C(12)	1131 (3)	6648 (2)	1633 (3)	1.13 (13)
C(13)	1051 (3)	6352 (2)	2666 (3)	1.01 (12)
C(14)	1929 (3)	5311 (2)	2917 (2)	0.86 (12)
C(15)	2153 (3)	4710 (2)	3885 (3)	1.03 (13)
C(16)	3033 (3)	3751 (2)	4126 (2)	0.89 (12)
C(17)	3347 (3)	3254 (2)	5084 (2)	0.93 (12)
C(18)	4290 (3)	2372 (2)	4971 (3)	1.10 (13)
C(19)	4538 (3)	2312 (2)	3962 (2)	0.93 (12)
C(20)	5437 (3)	1531 (2)	3535 (3)	0.94 (12)
C(25)	7596 (3)	-211 (2)	2650 (3)	1.18 (13)
C(26)	8708 (3)	210 (3)	2403 (4)	2.23 (17)
C(27)	7648 (3)	431 (3)	277 (3)	1.26 (13)
C(28)	6934 (4)	-314 (3)	360 (3)	2.15 (17)
C(29)	5299 (3)	3813 (3)	-2354 (3)	1.37 (13)
C(30)	4772 (4)	2845 (3)	-2127 (3)	2.05 (16)
C(31)	3125 (3)	5939 (3)	-2122 (3)	1.34 (13)
C(32)	1898 (3)	5658 (3)	-1814 (3)	2.00 (16)
C(33)	374 (3)	7594 (3)	1004 (3)	1.39 (14)
C(34)	-557 (3)	7167 (3)	978 (3)	1.88 (16)
C(35)	272 (3)	6958 (3)	3425 (3)	1.29 (13)
C(36)	993 (4)	7637 (3)	3402 (3)	2.04 (17)
C(37)	2755 (3)	3673 (3)	5984 (3)	1.31 (13)
C(38)	3277 (4)	4684 (3)	5669 (3)	2.17 (17)
C(39)	5018 (3)	1622 (3)	5695 (3)	1.32 (13)
C(40)	6260 (3)	2007 (3)	5202 (3)	1.83 (15)
C(41)	2305 (3)	1259 (2)	4009 (3)	1.29 (14)
C(42)	95 (3)	1494 (3)	4265 (3)	1.51 (15)
C(43)	682 (3)	3427 (3)	2942 (3)	1.36 (14)
C(50)	1189 (3)	823 (3)	2375 (3)	1.56 (15)
C(51)	1077 (3)	1963 (3)	1593 (3)	1.68 (16)
C(52)	2248 (3)	2311 (3)	1013 (3)	1.73 (15)
C(53)	3088 (3)	1376 (3)	1431 (3)	1.68 (15)
C(54)	2426 (3)	457 (3)	2272 (3)	1.64 (15)
O(1)	2670 (2)	777 (2)	4713 (2)	2.02 (11)
O(2)	-793 (2)	1232 (2)	5054 (2)	2.16 (11)
O(3)	45 (2)	4250 (2)	3011 (2)	2.24 (12)

* Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as:
 $\langle \ddagger \rangle [a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

$R(F) = 0.0302$, $wR(F) = 0.0249$, $GOF = 1.258$ for 5987 reflections. [(oep)InMo(CO)₃Cp] has two coordinated metal units, which are linked by a single covalent bond; the In—Mo bond distance is 2.890 (1) Å. The average In—N distance is 2.201 (2) ± 0.007 Å and the In atom lies 0.791 (1) Å above the four-N-atom plane towards the Mo atom. The average Mo—CO distance is 1.972 (1) ± 0.01 Å.

Experimental. Crystals were prepared according to Guilard, Mitaine, Moïse, Lecomte, Boukhris, Swistak, Tabard, Lacombe, Cornillon & Kadish (1987). A black

Table 2. Bond distances (Å) and bond angles (°)

In—N(1)	2.206 (2)	C(41)—O(1)	1.158 (4)
In—N(2)	2.208 (2)	C(42)—O(2)	1.151 (4)
In—N(3)	2.195 (2)	C(43)—O(3)	1.146 (4)
In—N(4)	2.196 (2)	C(50)—C(51)	1.413 (4)
In—Mo	2.890 (1)	C(51)—C(52)	1.409 (5)
Mo—C(41)	1.973 (3)	C(52)—C(53)	1.420 (5)
Mo—C(42)	1.962 (3)	C(53)—C(54)	1.414 (5)
Mo—C(43)	1.981 (3)	C(54)—C(50)	1.405 (5)
Mo—Cp*	2.018		
Mo—C(50)	2.341 (3)		
Mo—C(51)	2.349 (3)		
Mo—C(52)	2.347 (3)		
Mo—C(53)	2.353 (3)		
Mo—C(54)	2.356 (3)		
N(1)—C(1)	1.377 (4)	N(2)—C(6)	1.371 (4)
N(1)—C(4)	1.372 (4)	N(2)—C(9)	1.369 (3)
C(1)—C(2)	1.450 (4)	C(6)—C(7)	1.446 (4)
C(2)—C(3)	1.368 (4)	C(7)—C(8)	1.371 (4)
C(3)—C(4)	1.450 (4)	C(8)—C(9)	1.451 (4)
C(4)—C(5)	1.400 (4)	C(9)—C(10)	1.388 (4)
C(5)—C(6)	1.392 (4)	C(10)—C(11)	1.392 (4)
C(2)—C(25)	1.498 (4)	C(7)—C(29)	1.491 (4)
C(3)—C(27)	1.492 (4)	C(8)—C(31)	1.497 (4)
C(25)—C(26)	1.525 (4)	C(29)—C(30)	1.529 (4)
C(27)—C(28)	1.537 (4)	C(31)—C(32)	1.522 (5)
N(3)—C(11)	1.368 (4)	N(4)—C(16)	1.370 (4)
N(3)—C(14)	1.383 (4)	N(4)—C(19)	1.373 (4)
C(11)—C(12)	1.448 (4)	C(16)—C(17)	1.452 (4)
C(12)—C(13)	1.362 (4)	C(17)—C(18)	1.366 (4)
C(13)—C(14)	1.458 (4)	C(18)—C(19)	1.446 (4)
C(14)—C(15)	1.387 (4)	C(19)—C(20)	1.394 (4)
C(15)—C(16)	1.395 (4)	C(20)—C(1)	1.391 (4)
C(12)—C(33)	1.499 (4)	C(17)—C(37)	1.495 (4)
C(13)—C(35)	1.496 (4)	C(18)—C(39)	1.508 (4)
C(33)—C(34)	1.533 (5)	C(37)—C(38)	1.524 (4)
C(35)—C(36)	1.522 (4)	C(39)—C(40)	1.532 (4)
N(1)—In—Mo	110.1 (1)	N(1)—In—N(2)	82.2 (1)
N(2)—In—Mo	105.2 (1)	N(2)—In—N(3)	82.5 (1)
N(3)—In—Mo	111.8 (1)	N(3)—In—N(4)	83.1 (1)
N(4)—In—Mo	116.8 (1)	N(4)—In—N(1)	82.7 (1)
In—Mo—C(41)	70.3 (1)	C(41)—Mo—C(42)	80.3 (1)
In—Mo—C(42)	131.4 (4)	C(41)—Mo—C(43)	103.2 (1)
In—Mo—C(43)	72.1 (1)	C(42)—Mo—C(43)	78.4 (1)
In—Mo—C(50)	141.5 (1)	Mo—C(41)—O(1)	173.5 (2)
In—Mo—C(51)	120.6 (1)	Mo—C(42)—O(2)	177.8 (3)
In—Mo—C(52)	87.7 (1)	Mo—C(43)—O(3)	172.5 (3)
In—Mo—C(53)	83.9 (1)	In—Mo—Cp*	111.1
In—Mo—C(54)	113.8 (1)		
C(1)—N(1)—C(4)	106.9 (2)	C(6)—N(2)—C(9)	106.7 (2)
N(1)—C(4)—C(5)	125.0 (3)	N(2)—C(9)—C(10)	125.0 (3)
N(1)—C(4)—C(3)	109.9 (3)	N(2)—C(9)—C(8)	109.9 (2)
C(3)—C(4)—C(5)	125.1 (3)	C(8)—C(9)—C(10)	125.1 (3)
C(4)—C(3)—C(2)	106.6 (3)	C(9)—C(8)—C(7)	106.7 (3)
C(4)—C(3)—C(27)	124.8 (3)	C(9)—C(8)—C(31)	124.9 (3)
C(2)—C(3)—C(27)	128.4 (3)	C(7)—C(8)—C(31)	128.3 (3)
C(3)—C(2)—C(1)	107.1 (2)	C(8)—C(7)—C(6)	106.5 (3)
C(3)—C(2)—C(25)	127.7 (3)	C(8)—C(7)—C(29)	128.4 (3)
C(1)—C(2)—C(25)	125.1 (3)	C(6)—C(7)—C(29)	125.1 (3)
C(2)—C(1)—N(1)	109.4 (3)	C(7)—C(6)—N(2)	110.3 (2)
C(2)—C(1)—C(20)	126.0 (3)	C(7)—C(6)—C(5)	125.4 (3)
N(1)—C(1)—C(20)	124.5 (3)	N(2)—C(6)—C(5)	124.3 (3)
C(4)—C(5)—C(6)	127.4 (3)	C(9)—C(10)—C(11)	127.6 (3)
C(3)—C(27)—C(28)	111.8 (3)	C(7)—C(29)—C(30)	112.9 (3)
C(2)—C(25)—C(26)	112.2 (2)	C(8)—C(31)—C(32)	112.8 (3)
C(11)—N(3)—C(14)	107.0 (2)	C(16)—N(4)—C(19)	106.9 (2)
N(3)—C(14)—C(15)	124.4 (3)	N(4)—C(19)—C(20)	124.5 (3)
N(3)—C(14)—C(13)	109.2 (2)	N(4)—C(19)—C(18)	109.6 (3)
C(13)—C(14)—C(15)	126.4 (3)	C(18)—C(19)—C(20)	125.9 (3)
C(14)—C(13)—C(12)	106.8 (3)	C(19)—C(18)—C(17)	107.1 (3)
C(14)—C(13)—C(35)	124.0 (3)	C(19)—C(18)—C(39)	124.4 (3)
C(12)—C(13)—C(35)	129.1 (3)	C(17)—C(18)—C(39)	128.3 (3)
C(13)—C(12)—C(11)	107.1 (2)	C(18)—C(17)—C(16)	106.5 (3)
C(13)—C(12)—C(33)	129.4 (3)	C(18)—C(17)—C(37)	128.2 (2)
C(11)—C(12)—C(33)	123.4 (3)	C(16)—C(17)—C(37)	125.4 (3)
C(12)—C(11)—N(3)	109.9 (3)	C(17)—C(16)—N(4)	109.9 (2)
C(12)—C(11)—C(10)	125.5 (3)	C(17)—C(16)—C(15)	125.4 (3)
N(3)—C(11)—C(10)	124.6 (3)	N(4)—C(16)—C(15)	124.7 (3)
C(14)—C(15)—C(16)	127.9 (3)	C(19)—C(20)—C(1)	127.8 (3)
C(13)—C(35)—C(36)	113.1 (3)	C(18)—C(39)—C(40)	112.3 (3)
C(12)—C(33)—C(34)	112.6 (3)	C(17)—C(37)—C(38)	113.5 (3)

* Cp: center of the cyclopentadienyl ring.

crystal, 0.48 × 0.32 × 0.12 mm, of [(oep)InMo(CO)₃Cp] recrystallized from toluene/heptane was mounted on a CAD-4 diffractometer equipped with a Nonius low-temperature device on which a glove box was built to prevent ice formation (Aubry & Lecomte, 1983). Unit-cell dimensions at 100 K were obtained from accurate angle values of 25 reflections with $12 < \theta < 24^\circ$ using monochromatized Mo K α radiation. 7865 reflections were measured up to $(\sin \theta)/\lambda = 0.60 \text{ \AA}^{-1}$ at 100 K ($-15 \leq h \leq 15$, $-17 \leq k \leq 17$, $0 \leq l \leq 18$; 234, 441, 312, 224 standard reflections monitored every 2 h; ω -2 θ scan; scan width $1.4^\circ + 0.35^\circ \tan \theta$; scan speed $v: 0.7 < v < 3.30^\circ \text{ min}^{-1}$. No decay was observed. 5987 reflections [$I \geq 3\sigma(I)$], corrected for Lorentz and polarization effects, were used to solve the structure. The structure was solved by interpretation of the Patterson map; all non-H atoms were refined anisotropically; H-atom coordinates refined from the positions found in difference Fourier synthesis (SHELX76; Sheldrick, 1976). At convergence [$\Delta/\sigma_{\text{max}} = 0.53$ for x of C(28)], a residual Fourier map gave a maximum peak of 0.61 e \AA^{-3} . The weighting scheme used was $w^{-1} = \sigma^2(F) + 0.00018F^2$. Atomic scattering factors from SHELX76 and from *International Tables for X-ray Crystallography* (1974). Final residuals are $R(F) = 0.0302$; $wR(F) = 0.0249$; $\text{GOF} = 1.26$. Fractional coordinates and equivalent isotropic temperature factors of the non-H atoms are given in Table 1;* bond lengths and angles are listed in Table 2; Fig. 1 is the ORTEP (Johnson, 1965) drawing of the molecule.

Related literature. For a review of metal-metal bonding in metalloporphyrin chemistry, see Guillard, Lecomte & Kadish (1987), Brothers & Collman (1986) and references therein.

The crystal structures containing a hetero metal-metal bond in the metalloporphyrin series are: [(oep)SnFe(CO)₄] (Barbe, Guillard, Lecomte & Gerardin, 1984), Sn=Fe = 2.491 (1) Å; [(tetraphenylporphinato)SnMn(CO)₄HgMn(CO)₅}.0.5CH₂Cl₂ (Onaka *et al.*, 1985), Sn-Mn = 2.554 (3) Å; [(oep)InMn(CO)₅] (Guillard, Mitaine, Moïse, Lecomte, Boukhris, Swistak, Tabard, Lacombe, Cornillon & Kadish, 1987), In-Mn = 2.705 (1) Å, [(oep)RhIn(oep)] (Jones, Carrol & Wayland, 1986), Rh-In = 2.584 (2) Å; [(oep)TiMn-

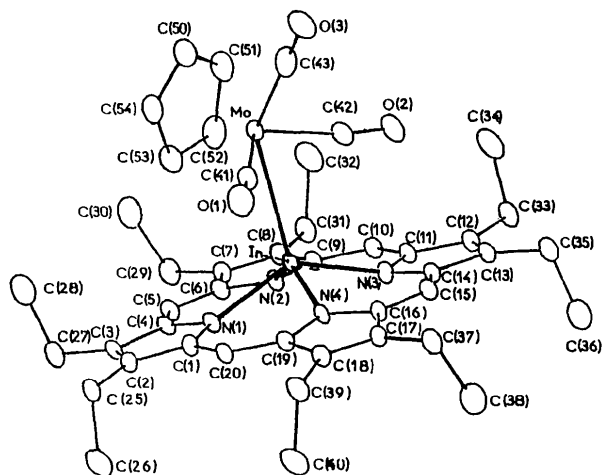


Fig. 1. ORTEP view of [(oep)InMo(CO)₃Cp].

(CO)₅] (Guillard *et al.*, 1988), Ti-Mn = 2.6994 (9) Å; [(oep)TiMo(CO)₃Cp] (Richard, Zrineh, Guillard, Habbou & Lecomte, 1989), Ti-Mo = 2.829 (1) Å. Compared to [(oep)Sn=Fe(CO)₄] (Barbe, Guillard, Lecomte & Gerardin, 1984) and to the above values the In-Mo bond [2.890 (1) Å] corresponds to a single covalent bond.

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* Lists of observed and calculated structure factors, anisotropic thermal parameters, positional and isotropic temperature factors for H atoms and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51796 (101 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.