

References

- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A24, 321–324.
- MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J. P. (1978). *MULTAN. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. (1976). *PLUTO*. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- STEWART, J. M. (1976). The XRAY 76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* 42, 3175–3187.

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Structure of *cis*-Bromodicarbonyl(η -cyclopentadienyl)(triphenylphosphine)molybdenum Dichloromethane Solvate

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Abstract. [MoBr(C₅H₅)(C₁₈H₁₅P)(CO)₂] $\cdot\frac{1}{4}$ CH₂Cl₂, C₂₅H₂₀BrMoO₂P $\cdot\frac{1}{4}$ CH₂Cl₂, triclinic, $P\bar{1}$, $a = 18.927$ (4), $b = 12.335$ (2), $c = 11.003$ (2) Å, $\alpha = 103.61$ (1), $\beta = 105.58$ (1), $\gamma = 99.70$ (1)°, $U = 2330$ Å³, $D_m = 1.66$, $Z = 4$, $D_c = 1.654$ Mg m⁻³, $F(000) = 1154$, $\mu(\text{Mo } K\alpha) = 2.52$ mm⁻¹. The structure was refined to $R = 0.057$ for 5212 diffractometer data. The Br and *trans* CO sites are partially mutually disordered. The mean Mo–P and Mo–Br lengths are 2.532 (2) and 2.671 (3) Å.

Introduction. [MoBr(η -C₅H₅)(CO)₂(PPh₃)] was prepared by heating [MoBr(η -C₅H₅)(CO)₃] with triphenylphosphine under reflux. Red polyhedral crystals were obtained from solution in a petrol–dichloromethane mixture and one 0.20 × 0.20 × 0.25 mm was selected for the X-ray investigation. The intensities of 6944 reflections with $\theta \leq 25^\circ$ were measured by the ω - 2θ step-scan procedure with Zr-filtered Mo $K\alpha$ radiation on a Hilger & Watts four-circle diffractometer. The 5212 observations that satisfied the criterion $I > 3\sigma(I)$ were used in the subsequent analysis.

The coordinates of the two unique Mo atoms were deduced from a Patterson synthesis and the remaining atoms, apart from those of the CO group *trans* to the Br atom in each molecule, were located from successive electron-density distributions. After preliminary least-squares refinement, a difference synthesis in-

dicated that there is disorder involving some interchange between the Br atom and the *trans* CO group in each molecule, the effect being similar to the halogen-carbonyl disorder that has been reported in several Ir complexes, e.g. [Ir(CO)ClO₂(PPh₃)] (La Placa & Ibers, 1965), [Ir(CO)₂Cl(PPh₃)] (Payne & Ibers, 1969), [Ir(CN)(CO)Cl(NCS)(PPh₃)₂] (Ibers, Hamilton & Baddley, 1973) and [IrBr{C₂(CN)₄}(CO)(PPh₃)₂] (Manojlović-Muir, Muir & Ibers, 1969).

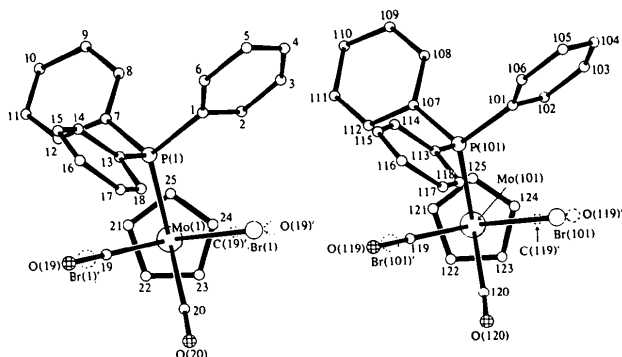
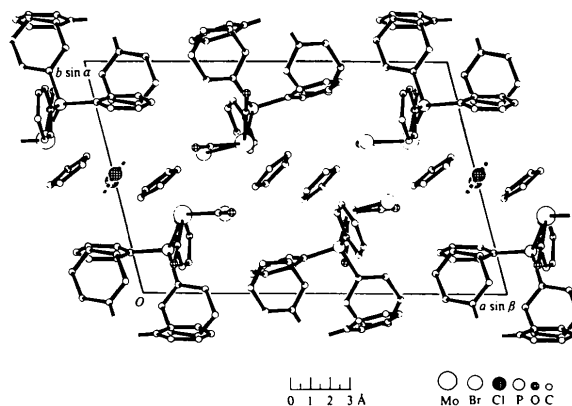
The population parameters of the Br atoms were initially estimated from peak heights in an electron-density distribution and were subsequently adjusted by least squares. The population parameters of the C and O atoms of the disordered CO group were kept equal to the appropriate Br parameters. The C and O positions in the disordered group were held fixed at Mo–C and C–O = 1.96 and 1.14 Å.

A peak of ca 13 e Å⁻³, about 1.5 Å from a centre of symmetry, and the centrosymmetrically related peak were identified as Cl atoms of a CH₂Cl₂ molecule. The C atom of this solvent molecule is necessarily disordered and appeared as two peaks, of height 1.4 e Å⁻³, related by the centre of symmetry.

When least-squares adjustment of the various parameters reached $R = 0.064$, most of the H atoms were apparent in a difference map. The H atoms were included at ideal positions and further refinement converged at $R = 0.057$, $R_w = 0.050$, with weights $w^{-1} = 17 + 0.01(|F_o| - 70)^2 - 30 \sin \theta$. The peaks and

Table 1. Atomic coordinates and population parameters, where different from 1.0

	x	y	z	Population parameter		x	y	z	Population parameter
Mo(1)	0.83611 (4)	0.66078 (6)	-0.04501 (6)		Mo(101)	0.61945 (4)	0.35353 (6)	0.27555 (7)	
Br(1)	0.71875 (6)	0.65433 (11)	-0.04731 (11)	0.792 (4)	Br(101)	0.73785 (7)	0.39556 (12)	0.49201 (12)	0.689 (4)
Br(1)'	0.88706 (29)	0.84269 (48)	-0.12185 (46)	0.208	Br(101)'	0.58646 (26)	0.16768 (35)	0.07178 (38)	0.311
P(1)	0.90319 (11)	0.81259 (16)	0.17641 (18)		P(101)	0.57168 (10)	0.20250 (16)	0.37428 (18)	
C(1)	0.8760 (4)	0.7906 (7)	0.3173 (7)		C(101)	0.5977 (4)	0.2428 (6)	0.5538 (7)	
C(2)	0.8547 (6)	0.6802 (8)	0.3256 (8)		C(102)	0.5915 (5)	0.3493 (7)	0.6221 (8)	
C(3)	0.8343 (6)	0.6625 (9)	0.4312 (9)		C(103)	0.6094 (5)	0.3821 (8)	0.7582 (8)	
C(4)	0.8320 (6)	0.7509 (10)	0.5280 (9)		C(104)	0.6329 (5)	0.3061 (9)	0.8278 (8)	
C(5)	0.8538 (5)	0.8611 (10)	0.5254 (8)		C(105)	0.6388 (5)	0.2018 (8)	0.7616 (8)	
C(6)	0.8755 (5)	0.8819 (8)	0.4194 (8)		C(106)	0.6202 (5)	0.1686 (7)	0.6255 (8)	
C(7)	1.0053 (4)	0.8231 (7)	0.2288 (8)		C(107)	0.4677 (4)	0.1549 (7)	0.3181 (8)	
C(8)	1.0418 (5)	0.7990 (8)	0.3438 (10)		C(108)	0.4280 (5)	0.1562 (8)	0.4076 (9)	
C(9)	1.1172 (6)	0.7980 (9)	0.3751 (12)		C(109)	0.3494 (6)	0.1212 (9)	0.3617 (13)	
C(10)	1.1568 (6)	0.8245 (10)	0.2946 (14)		C(110)	0.3110 (5)	0.0880 (9)	0.2276 (16)	
C(11)	1.1235 (5)	0.8501 (9)	0.1819 (12)		C(111)	0.3509 (6)	0.0867 (9)	0.1413 (12)	
C(12)	1.0479 (5)	0.8496 (8)	0.1508 (9)		C(112)	0.4294 (5)	0.1196 (8)	0.1844 (9)	
C(13)	0.8972 (4)	0.9610 (6)	0.1836 (7)		C(113)	0.6022 (4)	0.0685 (6)	0.3329 (7)	
C(14)	0.9586 (5)	1.0525 (8)	0.2393 (11)		C(114)	0.5517 (5)	-0.0383 (7)	0.2848 (9)	
C(15)	0.9501 (6)	1.1633 (8)	0.2462 (15)		C(115)	0.5773 (7)	-0.1367 (8)	0.2517 (11)	
C(16)	0.8808 (7)	1.1818 (8)	0.1971 (12)		C(116)	0.6519 (8)	-0.1298 (9)	0.2751 (12)	
C(17)	0.8193 (5)	1.0913 (8)	0.1410 (10)		C(117)	0.7021 (6)	-0.0229 (10)	0.3237 (13)	
C(18)	0.8264 (5)	0.9797 (7)	0.1329 (10)		C(118)	0.6782 (5)	0.0761 (8)	0.3527 (11)	
C(19)	0.8733	0.7929	-0.1007	0.80	C(119)	0.5929	0.2160	0.1242	0.69
O(19)	0.8949	0.8697	-0.1331	0.80	O(119)	0.5775	0.1360	0.0362	0.69
C(19)'	0.7502	0.6562	0.0228	0.20	C(119)'	0.7064	0.3844	0.4346	0.31
O(19)'	0.7002	0.6535	0.0622	0.20	O(119)'	0.7571	0.4024	0.5270	0.31
C(20)	0.7429 (6)	0.6531 (7)	-0.2028 (13)		C(120)	0.7153 (7)	0.3639 (8)	0.2136 (9)	
O(20)	0.6994 (4)	0.6468 (6)	-0.2782 (7)		O(120)	0.7561 (4)	0.3677 (7)	0.1854 (7)	
C(21)	0.9387 (5)	0.5893 (8)	-0.0640 (10)		C(121)	0.5084 (5)	0.4004 (9)	0.1809 (12)	
C(22)	0.8765 (5)	0.5338 (8)	-0.1828 (8)		C(122)	0.5687 (7)	0.4634 (10)	0.1504 (10)	
C(23)	0.8202 (5)	0.4686 (7)	-0.1504 (8)		C(123)	0.6187 (6)	0.5401 (8)	0.2715 (11)	
C(24)	0.8478 (6)	0.4779 (8)	-0.0180 (10)		C(124)	0.5886 (7)	0.5252 (8)	0.3716 (10)	
C(25)	0.9191 (6)	0.5526 (8)	0.0377 (9)		C(125)	0.5209 (6)	0.4395 (9)	0.3145 (12)	
					Cl	0.99289 (27)	0.48825 (34)	0.36332 (38)	
					C(50)	0.9596 (19)	0.4381 (28)	0.4816 (34)	0.50

Fig. 1. The two independent molecules of [MoBr(η -C₅H₅)(CO)₂(PPh₃)], each viewed along the normal to the η -C₅H₅ ring.Fig. 2. The crystal structure viewed along *c*. For clarity, only the principal orientation of each molecule of the complex is shown.

trenches in a final difference map all lay between 0.7 and -0.8 e Å⁻³, the major features being near the disordered sites.

Scattering factors and dispersion corrections were taken from *International Tables for X-ray Crystallography* (1962). Calculations were performed with XRAY 70 (Stewart, Kundell & Baldwin, 1970).*

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34602 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. The atomic coordinates, bond lengths, valency angles and intermolecular contacts are given in Tables 1-4. The atom numbering is shown in Fig. 1 and the crystal structure in Fig. 2.

The analysis confirms the spectroscopic assignment of the complex as the *cis* isomer. Both molecules adopt a conformation in which a Mo-CO bond is oriented approximately over the midpoint of a C-C bond of the cyclopentadienyl ring. The mean *trans* P-Mo-CO angle of 128° and the *cis* Br-Mo-CO and Br-Mo-P

Table 2. Bond lengths (Å)

	Molecule A	Molecule B
Mo(1)—Br(1)	2.676 (2)	2.666 (2)
Mo(1)—Br(1)'	2.698 (6)	2.648 (4)
Mo(1)—P(1)	2.538 (2)	2.525 (2)
Mo(1)—C(20)	2.087 (11)	2.095 (14)
C(20)—O(20)	0.977 (13)	0.904 (17)
Mo(1)—C(21)	2.303 (11)	2.313 (10)
Mo(1)—C(22)	2.290 (10)	2.294 (13)
Mo(1)—C(23)	2.310 (8)	2.315 (11)
Mo(1)—C(24)	2.383 (10)	2.374 (11)
Mo(1)—C(25)	2.363 (11)	2.379 (13)
P(1)—C(1)	1.823 (9)	1.822 (8)
P(1)—C(7)	1.834 (8)	1.837 (8)
P(1)—C(13)	1.837 (8)	1.838 (9)
C(1)—C(2)	1.385 (13)	1.392 (11)
C(2)—C(3)	1.371 (16)	1.384 (12)
C(3)—C(4)	1.349 (15)	1.400 (15)
C(4)—C(5)	1.361 (17)	1.360 (14)
C(5)—C(6)	1.400 (15)	1.383 (12)
C(6)—C(1)	1.394 (11)	1.390 (13)
C(7)—C(8)	1.395 (14)	1.389 (15)
C(8)—C(9)	1.378 (14)	1.387 (13)
C(9)—C(10)	1.364 (21)	1.381 (19)
C(10)—C(11)	1.366 (19)	1.360 (21)
C(11)—C(12)	1.376 (14)	1.386 (13)
C(12)—C(7)	1.381 (15)	1.376 (12)
C(13)—C(14)	1.360 (10)	1.379 (11)
C(14)—C(15)	1.389 (15)	1.387 (15)
C(15)—C(16)	1.358 (17)	1.347 (19)
C(16)—C(17)	1.354 (12)	1.377 (15)
C(17)—C(18)	1.390 (14)	1.370 (17)
C(18)—C(13)	1.387 (13)	1.377 (13)
C(21)—C(22)	1.430 (11)	1.430 (17)
C(22)—C(23)	1.399 (14)	1.410 (13)
C(23)—C(24)	1.379 (14)	1.403 (19)
C(24)—C(25)	1.383 (13)	1.394 (14)
C(25)—C(21)	1.414 (16)	1.375 (17)
C(50)—Cl		1.78 (4)
C(50)—Cl*		1.65 (4)

* Cl atom with coordinates 2 - x, 1 - y, 1 - z.

Table 3. Valency angles (°)

	Molecule A	Molecule B
Br(1)—Mo(1)—P(1)	82.1 (1)	79.6 (1)
Br(1)—Mo(1)—C(20)	74.0 (4)	73.9 (3)
P(1)—Mo(1)—C(20)	131.4 (3)	125.2 (3)
Mo(1)—C(20)—O(20)	178.0 (10)	179.0 (8)
Mo(1)—P(1)—C(1)	117.2 (2)	116.9 (2)
Mo(1)—P(1)—C(7)	111.1 (3)	112.7 (3)
Mo(1)—P(1)—C(13)	116.5 (2)	114.6 (3)
C(1)—P(1)—C(7)	103.4 (4)	103.7 (4)
C(1)—P(1)—C(13)	103.2 (4)	103.4 (4)
C(7)—P(1)—C(13)	103.8 (4)	104.0 (3)
P(1)—C(1)—C(2)	120.1 (6)	119.3 (7)
P(1)—C(1)—C(6)	122.3 (7)	122.3 (6)
P(1)—C(7)—C(8)	121.7 (7)	121.2 (6)
P(1)—C(7)—C(12)	120.5 (7)	118.5 (7)
P(1)—C(13)—C(14)	122.9 (7)	122.1 (7)
P(1)—C(13)—C(18)	118.0 (6)	118.4 (6)
C(2)—C(1)—C(6)	117.6 (8)	118.3 (7)
C(1)—C(2)—C(3)	120.6 (8)	121.0 (9)
C(2)—C(3)—C(4)	121.5 (11)	119.4 (9)
C(3)—C(4)—C(5)	120.0 (11)	119.9 (8)
C(4)—C(5)—C(6)	119.8 (9)	120.8 (10)
C(5)—C(6)—C(1)	120.4 (9)	120.7 (8)
C(8)—C(7)—C(12)	117.7 (8)	120.3 (8)
C(7)—C(8)—C(9)	120.5 (11)	119.7 (9)
C(8)—C(9)—C(10)	119.5 (11)	119.9 (13)
C(9)—C(10)—C(11)	121.8 (10)	119.6 (10)
C(10)—C(11)—C(12)	118.2 (12)	121.7 (11)
C(11)—C(12)—C(7)	122.2 (10)	118.8 (10)
C(14)—C(13)—C(18)	119.1 (8)	119.5 (8)
C(13)—C(14)—C(15)	120.3 (9)	119.9 (9)
C(14)—C(15)—C(16)	120.7 (8)	120.7 (9)
C(15)—C(16)—C(17)	119.6 (9)	119.1 (12)
C(16)—C(17)—C(18)	120.8 (9)	121.5 (11)
C(17)—C(18)—C(13)	119.6 (7)	119.3 (8)
C(22)—C(21)—C(25)	106.7 (8)	108.8 (8)
C(21)—C(22)—C(23)	107.6 (9)	106.4 (10)
C(22)—C(23)—C(24)	108.0 (7)	108.0 (10)
C(23)—C(24)—C(25)	109.9 (10)	108.5 (9)
C(24)—C(25)—C(21)	107.7 (8)	108.4 (11)
Cl—C(50)—Cl*		115 (2)

* Cl atom with coordinates 2 - x, 1 - y, 1 - z.

angles of 74–82° are typical of angles in [Mo(η -C₅H₅)(CO)₂XY] complexes (Bush, Hardy, Manojlović-Muir & Sim, 1971; Churchill & Fennessey, 1968; Hardy & Sim, 1972).

The mean Mo—Br length of 2.671 (3) Å is in good agreement with Mo—Br distances of 2.66 Å in the seven-coordinate Mo^{II} complexes [MoBr₂(CO)₃-(Ph₂PCH₂CH₂PPh₂)] (Drew, 1972a) and [MoBr₂(CO)₂(Ph₂AsCH₂AsPh₂)₂] (Drew, 1972b). The PPh₃ ligand is situated *trans* to a CO group and the mean Mo—P length is 2.532 (2) Å. This is slightly longer than the Mo—P distances of 2.47 Å in [Mo(η -C₅H₅)(CO)₂(COMe)(PPh₃)] where the PPh₃ ligand is *trans* to COMe (Churchill & Fennessey, 1968), 2.48 Å in [Mo(η -C₅H₅)(CO)₂I(PPh₃)] where the PPh₃ ligand is *trans* to I (Bush, Hardy, Manojlović-Muir & Sim, 1971), and 2.50 Å in [Mo(η -C₅H₅)(CO)(NCO)(PPh₃)₂] where the PPh₃ ligands are mutually *trans* (McPhail, Knox, Robertson & Sim, 1971). This series

Table 4. Intermolecular contacts ≤ 3.60 Å

The e.s.d.'s of these distances are ca 0.02 Å.

O(119)'...C(22) ^I	3.22	O(19)...C(15) ^{III}	3.53
O(20)...C(110) ^{II}	3.24	O(19)'...C(116) ^{VII}	3.54
O(119)'...C(23) ^I	3.27	Br(1)'...C(4) ^{VI}	3.55
O(19)...C(14) ^{III}	3.37	C(18)...C(117) ^{VII}	3.55
O(20)...C(109) ^{II}	3.40	O(120)...C(16) ^{VIII}	3.55
C(21)...C(25) ^{IV}	3.43	O(120)...C(24)	3.55
O(119)...C(112) ^V	3.45	C(20)...C(110) ^{II}	3.56
O(19)...C(4) ^{VI}	3.45	Cl...C(22) ^{IV}	3.57
O(119)...C(114) ^V	3.50	O(20)...C(103) ^{VI}	3.58
O(19)'...C(123)	3.51	C(5)...C(14) ^{IX}	3.60
O(19)...C(12) ^{III}	3.52	O(19)...C(5) ^{VI}	3.60

The superscripts refer to the following transformations of the atomic coordinates:

(I)	x,	y,	1 + z	(VI)	x,	y,	-1 + z
(II)	1 - x,	1 - y,	-z	(VII)	x,	1 + y,	z
(III)	2 - x,	2 - y,	-z	(VIII)	x,	-1 + y,	z
(IV)	2 - x,	1 - y,	-z	(IX)	2 - x,	2 - y,	1 - z.
(V)	1 - x,	-y,	-z				

may be explicable in terms of the relative π -acceptor capabilities of the groups *trans* to the phosphine.

In both molecules, the CO group *cis* to the Br atom has the C atom *ca* 0.13 Å (11 σ) further away from the Mo atom than expected. Abnormal bond lengths of this type are symptomatic of halogen-carbonyl disorder (Payne & Ibers, 1969) and it is possible that the crystal contains a small amount of *trans*-[MoBr(η -C₅H₅)(CO)₂(PPh₃)].

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References

- BUSH, M. A., HARDY, A. D. U., MANOJLOVIĆ-MUIR, L. J. & SIM, G. A. (1971). *J. Chem. Soc. A*, pp. 1003–1009.
- CHURCHILL, M. R. & FENNESSEY, J. P. (1968). *Inorg. Chem.* **7**, 953–959.
- DREW, M. G. B. (1972*a*). *J. Chem. Soc. Dalton Trans.* pp. 1329–1333.
- DREW, M. G. B. (1972*b*). *J. Chem. Soc. Dalton Trans.* pp. 626–631.
- HARDY, A. D. U. & SIM, G. A. (1972). *J. Chem. Soc. Dalton Trans.* pp. 1900–1903.
- IBERS, J. A., HAMILTON, D. S. & BADDLEY, W. H. (1973). *Inorg. Chem.* **12**, 229–232.
- International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.
- LA PLACA, S. J. & IBERS, J. A. (1965). *J. Am. Chem. Soc.* **87**, 2581–2586.
- MCPHAIL, A. T., KNOX, G. R., ROBERTSON, C. G. & SIM, G. A. (1971). *J. Chem. Soc. A*, pp. 205–214.
- MANOJLOVIĆ-MUIR, L. J., MUIR, K. W. & IBERS, J. A. (1969). *Discuss. Faraday Soc.* **47**, 84–92.
- PAYNE, N. C. & IBERS, J. A. (1969). *Inorg. Chem.* **8**, 2714–2718.
- STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

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Structure of *trans*-Dicarbonyl(η -cyclopentadienyl)iido(phenyl isocyanide)molybdenum

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Abstract. C₁₄H₁₀IMoNO₂, [Mo(C₅H₅)(C₇H₅N)(CO)₂I], orthorhombic, *Pbca*, $a = 12.035$ (4), $b = 17.248$ (5), $c = 14.510$ (5) Å, $U = 3012$ Å³, $D_m = 1.95$, $Z = 8$, $D_c = 1.972$ Mg m⁻³, $F(000) = 1696$, $\mu(\text{Mo } K\alpha) = 2.93$ mm⁻¹. The structure was refined to $R = 0.040$ for 1471 diffractometer data. The Mo–I, Mo–C(isocyanide) and mean Mo–C(carbonyl) lengths are 2.858 (1), 2.025 (10) and 1.978 (7) Å, respectively.

Introduction. (η^5 -C₅H₅)Mo(CO)₂(CNPh)I was prepared by treatment of (η^5 -C₅H₅)Mo(CO)₃I with phenyl isocyanide (Joshi, Pauson & Stubbs, 1963/64). The crystal used for the X-ray study was selected from a mixture of the *cis* and *trans* isomers. Mo $K\alpha$ radiation was employed and X-ray intensities for reflections with $\theta < 27^\circ$ were measured by the ω -2 θ step-scan procedure on a Hilger & Watts four-circle diffractometer fitted with a graphite monochromator. The 1564 reflections that were above background on a preliminary rapid scan were recorded. Of these, 1471 satis-

fied the criterion $I > 3\sigma(I)$ and were used in the subsequent analysis.

The coordinates of the Mo and I atoms were deduced from a Patterson synthesis and the remaining atoms located in an electron-density distribution. The atomic parameters were then adjusted by least squares and when R had been reduced to 0.043 a difference synthesis revealed the H atoms. These were included at the positions taken from the difference map and further refinement converged at $R = 0.040$, $R_w = 0.056$. The weighting scheme was

$$w = [1 - \exp(-p_1 \sin^2 \theta / \lambda^2)] / [1 + p_2 |F_o| + p_3 |F_o|^2]$$

and in the final stages p_1 , p_2 and p_3 were assigned the values 90.0, 0.54 and 0.01, respectively. Scattering factors, with dispersion corrections for Mo and I, were taken from *International Tables for X-ray Crystallography* (1962). The calculations were performed with programs developed for the Glasgow University KDF9