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 (11σ) 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-C_5H_5)-$(CO)_2(PPh_3)$].

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

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Abstract. $C_{14}H_{10}IMoNO_2$, $[Mo(C_5H_5)(C_7H_5N)(CO)_2I]$, orthorhombic, *Pbca*, $a = 12·035$ (4), $b = 17·248$ (5), $c = 14·510$ (5) Å, $U = 3012$ Å^3, $D_m = 1·95$, $Z = 8$, $D_c = 1·972$ Mg m^{-3}, $F(000) = 1696$, μ(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. $(\eta^5\text{-}C_5H_5)Mo(CO)_2(CNPh)I$ was prepared by treatment of $(\eta^5\text{-}C_5H_5)Mo(CO)_3I$ 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 $\theta < 27°$ were measured by the ω–2θ 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 satisfied 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

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Table 1. *Atomic coordinates* (×10^5 *for* Mo *and* I, ×10^4 *for* C, N *and* O)

	x	*y*	*z*
Mo	64036 (6)	18578 (4)	39509 (5)
I	87306 (5)	15570 (4)	37895 (5)
C(1)	5325 (8)	2712 (5)	3608 (6)
N(2)	4737 (8)	3209 (5)	3396 (7)
C(3)	4022 (8)	3832 (5)	3234 (7)
C(4)	3230 (9)	3760 (6)	2541 (8)
C(5)	2518 (10)	4388 (8)	2399 (10)
C(6)	2561 (11)	5023 (8)	2931 (10)
C(7)	3385 (14)	5115 (6)	3588 (9)
C(8)	4110 (10)	4493 (7)	3768 (7)
C(9)	5530 (10)	631 (6)	3907 (8)
C(10)	6376 (12)	575 (7)	4557 (9)
C(11)	6191 (10)	1120 (7)	5267 (8)
C(12)	5148 (11)	1492 (6)	5056 (8)
C(13)	4774 (9)	1209 (6)	4209 (9)
C(14)	7052 (8)	2794 (6)	4518 (6)
O(15)	7367 (7)	3360 (5)	4847 (6)
C(16)	6678 (8)	1989 (5)	2612 (7)
O(17)	6788 (7)	2077 (5)	1827 (5)

Table 2. *Bond lengths* (Å)

Mo–I	2·858 (1)	N(2)–C(3)	1·397 (13)
Mo–C(9)	2·363 (11)	C(3)–C(4)	1·392 (15)
Mo–C(10)	2·382 (12)	C(4)–C(5)	1·395 (17)
Mo–C(11)	2·309 (11)	C(5)–C(6)	1·342 (20)
Mo–C(12)	2·293 (12)	C(6)–C(7)	1·385 (20)
Mo–C(13)	2·289 (11)	C(7)–C(8)	1·409 (17)
Mo–C(14)	1·973 (10)	C(8)–C(3)	1·381 (15)
C(14)–O(15)	1·150 (12)	C(9)–C(10)	1·390 (18)
Mo–C(16)	1·983 (10)	C(10)–C(11)	1·412 (17)
C(16)–O(17)	1·158 (11)	C(11)–C(12)	1·443 (17)
Mo–C(1)	2·025 (10)	C(12)–C(13)	1·396 (17)
C(1)–N(2)	1·153 (13)	C(13)–C(9)	1·418 (15)

Table 3. *Valency angles* (°)

I–Mo–C(1)	137·7 (3)	C(8)–C(3)–C(4)	122·1 (9)
I–Mo–C(14)	78·2 (3)	C(3)–C(4)–C(5)	117·2 (10)
I–Mo–C(16)	77·1 (3)	C(4)–C(5)–C(6)	121·7 (12)
C(1)–Mo–C(14)	76·2 (4)	C(5)–C(6)–C(7)	121·2 (12)
C(1)–Mo–C(16)	77·5 (4)	C(6)–C(7)–C(8)	118·9 (11)
C(14)–Mo–C(16)	104·4 (4)	C(7)–C(8)–C(3)	118·5 (10)
Mo–C(14)–O(15)	175·9 (9)	C(13)–C(9)–C(10)	108·0 (10)
Mo–C(16)–O(17)	176·8 (9)	C(9)–C(10)–C(11)	109·4 (11)
Mo–C(1)–N(2)	177·8 (9)	C(10)–C(11)–C(12)	106·2 (10)
C(1)–N(2)–C(3)	174·2 (11)	C(11)–C(12)–C(13)	108·1 (10)
N(2)–C(3)–C(4)	118·3 (9)	C(12)–C(13)–C(9)	108·1 (10)
N(2)–C(3)–C(8)	119·6 (9)		

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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.

Table 4. *Intermolecular contacts* (≤3·70 Å)

C(13)···O(15)^{I}	3·29 (2)	C(6)···O(17)^{III}	3·65 (2)
C(12)···O(15)^{I}	3·36 (2)	C(6)···C(11)^{I}	3·67 (2)
C(14)···O(17)^{II}	3·37 (2)	C(7)···C(16)^{III}	3·67 (2)
C(7)···O(17)^{III}	3·44 (2)	C(6)···C(9)^{III}	3·67 (2)
C(4)···O(17)^{IV}	3·51 (2)	C(4)···O(15)^{IV}	3·68 (2)
C(4)···C(16)^{IV}	3·59 (2)	C(4)···C(14)^{IV}	3·70 (2)
C(7)···O(15)^{V}	3·59 (2)	C(5)···C(9)^{III}	3·70 (2)
C(6)···C(16)^{III}	3·60 (2)		

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

(I)	$-\frac{1}{2}+x,\ \frac{1}{2}-y,\ 1-z$	(IV)	$-\frac{1}{2}+x,\ y,\ \frac{1}{2}-z$
(II)	$x,\ \frac{1}{2}-y,\ \frac{1}{2}+z$	(V)	$1-x,\ 1-y,\ 1-z.$
(III)	$-\frac{1}{2}+x,\ y,\ \frac{1}{2}-z$		

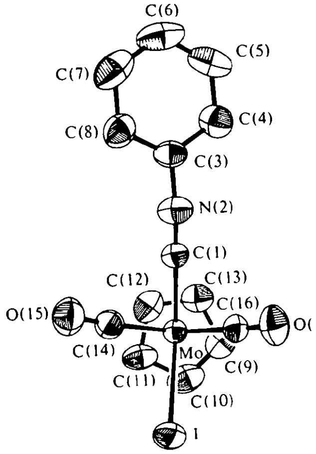

Fig. 1. The (η^5-C_5H_5)Mo(CO)$_2$(CNPh)I molecule, viewed along the perpendicular to the C_5H_5 ring. The ellipsoids are shown at the 50% probability level.

The *trans* I–Mo–CNPh and OC–Mo–CO angles of 138 and 104° closely resemble I–Mo–P and OC–Mo–CO angles of 141 and 105° in *trans*-(η^5-C_5H_5)Mo(CO)$_2$(PPh$_3$)I (Bush, Hardy, Manojlović-Muir & Sim, 1971), 136 and 109° in *trans*-(η^5-C_5H_5)Mo(CO)$_2$[P(OMe)$_3$]I (Hardy & Sim, 1972) and 138 and 107° in *trans*-(η^5-C_5H_4.CH$_3$)Mo(CO)$_2$[P(OMe)$_3$]I (Hardy & Sim, 1972).

The Mo–I separation of 2·858 (1) Å may be compared with values of 2·836 (4), 2·850 (3) and 2·858 (3) Å in (η^5-C_5H_5)Mo(CO)$_2$[P(OMe)$_3$]I, (η^5-C_5H_4.CH$_3$)Mo(CO)$_2$[P(OMe)$_3$]I and (η^5-C_5H_5)Mo(CO)$_2$(PPh$_3$)I, respectively. These distances exceed the Mo–Br separation of 2·671 (3) Å in (η^5-C_5H_5)Mo(CO)$_2$(PPh$_3$)Br (Sim, Sime, Woodhouse & Knox, 1979) by 0·17–0·19 Å, in excellent agreement with the difference of 0·19 Å between the covalent radii of I (1·33 Å) and Br (1·14 Å) atoms.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34581 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

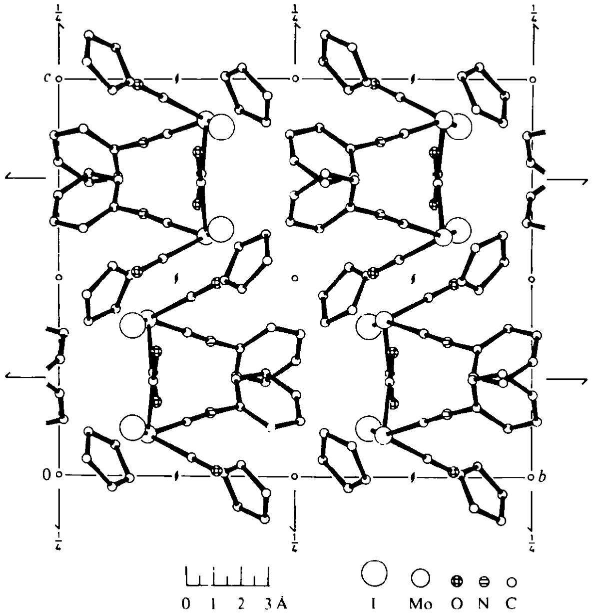

Fig. 2. The crystal structure viewed along **a**.

The Mo—CO lengths, 1·973 (10) and 1·983 (10) Å, do not differ significantly and the mean, 1·978 (7) Å, may be compared with the 1·96 Å in (η^5-C_5H_5)-$Mo(CO)_2(PPh_3)(COMe)$ (Churchill & Fennessey, 1968), 1·98 Å in (η^5-C_5H_5)$Mo(CO)_2(PPh_3)I$, 2·00 Å in (η^5-$C_5H_4.CH_3$)$Mo(CO)_2[P(OMe)_3]I$ and 2·02 Å in (η^5-C_5H_5)$Mo(CO)_2[P(OMe)_3]I$. The Mo—CNPh length of 2·025 (10) Å is only slightly greater than the mean Mo—CO distance, indicating that the bond has significant double-bond character (Sim, Sime, Woodhouse & Knox, 1974).

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The Structure of Bis(*N*-methylethylenediamine)copper(II) Adipate Dihydrate

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Abstract. $C_6H_{20}CuN_4^{2+}.C_6H_8O_4^{2-}.2H_2O$, $[Cu(C_3H_{10}N_2)_2](C_6H_8O_4).2H_2O$, M_r = 391·95, triclinic, $P\bar{1}$, a = 7·241 (2), b = 8·271 (3), c = 8·304 (3) Å, α = 89·92 (3), β = 114·87 (2), γ = 90·45 (2)°, V = 451·2 Å^3, Z = 1, D_m (flotation) = 1·43, D_x = 1·443 Mg m^{-3}, μ(Mo $K\alpha$) = 1·29 mm^{-1}. The structure was solved by direct methods and refined by the block-diagonal least-squares technique, with anisotropic temperature parameters for nonhydrogen atoms and isotropic parameters for hydrogens, to an R value of 0·035. The analysis was based on 1931 reflections with $I > 2\sigma(I)$. The four N atoms from the diamine molecules form the coordination plane around the Cu^{II} atom; the two water O atoms lying in axial positions complete the elongated octahedral coordination sphere. The adipate group is hydrogen bonded to the N and water O atoms of the adjacent complex cations.

Introduction. The structures of some bis(*N*-methylethylenediamine)copper(II) carboxylates have been investigated previously in this laboratory (Hämäläinen, 1973; Hämäläinen & Pajunen, 1973, 1974). The carboxylate groups in these bis compounds are joined to the cations by hydrogen bonds, giving rise to net-like structures. We have now synthesized a bis chelate using a longer-chain dicarboxylic acid, adipic acid, as the anion and studied the structure by X-ray analysis.

The title compound was synthesized by allowing 0·045 mol of the finely powdered Cu^{II} salt of adipic acid [prepared by the method of Rajan (1962)] to react

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