

coefficient $g = 2.7 \times 10^{-4}$ was also made. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) and refined with *SHELX76* (Sheldrick, 1976). Final $R = 0.037$, $wR = 0.034$ and $S = 2.04$. The weighting scheme was $2.03/\sigma^2(F)$ and at the end of the refinement $(\Delta/\sigma)_{\max}$ was 0.234. The H atoms of the phenyl and oxazole rings were located by geometrical calculations ($C-H = 1.08 \text{ \AA}$) with fixed isotropic thermal parameters (0.063 \AA^2). The C atoms C13, C14 and C15 were refined with a site occupation factor of 65% (*A*) and 35% (*B*) for C13*A*, C14*A* and C15*A*. The thermal parameters of the atoms of this radical which are rather large were refined isotropically. In the final difference Fourier map $(\Delta\rho)_{\min}$, $(\Delta\rho)_{\max}$ were between -0.61 and 0.64 e \AA^{-3} . Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed on a VAX 8530 and an IBM 3090 computer.

Discussion. The atomic coordinates are listed in Table 1* and bond lengths and angles in Table 2. A projection of the molecule and of the packing of the molecules in the cell are shown in Figs. 1 and 2, respectively.

The coordination around the Zn atom is a distorted tetrahedron. The Zn—Cl and Zn—N(8) bond lengths, respectively 2.264 (1) and 2.031 (4) Å, are

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and a diagram of the disordered β -methylallyl group have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54840 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0556]

close to those reported for ZnCl₂(imidazole)₂ (Lundberg, 1966) and ZnCl₂(pyridine)₂ (Steffen & Palenik, 1976). The oxazole ring is planar as shown by the torsion angles N(8)—C(7)—O(11)—C(10) = $-0.4 (6)$ and O(11)—C(7)—N(8)—C(9) = $1.5 (6)^\circ$. In this heterocycle the N(8)—C(7) bond of 1.332 (7) Å is longer than a double bond and the C(9)—N(8) bond of 1.386 (7) Å is shorter than a single bond. On the other hand, the C—O bonds of average length 1.36 Å have double-bond character. These values could be related to the aromaticity of the oxazole ring. The bond lengths in the phenyl ring are in the range 1.358 (9)–1.407 (9) Å. The torsion angles C(2)—C(1)—C(7)—N(8) = $26.1 (9)$ and C(6)—C(1)—C(7)—O(11) = $25.5 (8)^\circ$ show that the oxazole ring is twisted about $25.8 (9)^\circ$ out of the phenyl-ring plane. The low value of this twist angle and the short distance C(7)—C(1) = 1.448 Å could explain the conjugation between the two rings. The β -methylallyl is disordered.

One of the authors (AK) thanks the Deutscher Akademischer Austauschdienst for financial support and H. Fuess for helpful discussions. Support of the Fonds der Chemischen Industrie is gratefully acknowledged.

References

- HAJJEM, B. & BACCAR, B. (1991). *Synth. Commun.* Submitted.
 LUNDBERG, B. K. S. (1966). *Acta Cryst.* **21**, 901–909.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
 STEFFEN, W. L. & PALENIK, G. J. (1976). *Acta Cryst.* **B32**, 298–300.

Acta Cryst. (1992). **C48**, 1004–1007

The Synthesis and Structure of the Pyridine-Substituted Derivative of Tricarbonyl(η^5 -methylcyclopentadienyl)manganese(I)

BY BARRY CROCOCK AND CONOR LONG*

School of Chemical Sciences, Dublin City University, Dublin 9, Ireland

AND R. ALAN HOWIE

Department of Chemistry, University of Aberdeen, Meston Walk, Aberdeen, Scotland

(Received 13 September 1991; accepted 4 November 1991)

Abstract. Dicarbonyl(η^5 -methylcyclopentadienyl)-(pyridine)manganese(I), C₁₃H₁₂MnNO₂, $M_r =$

269.18, monoclinic, $P2_1/c$, $a = 7.761 (5)$, $b = 20.578 (10)$, $c = 15.634 (11) \text{ \AA}$, $\beta = 103.10 (6)^\circ$, $V = 2432 (3) \text{ \AA}^3$, $Z = 8$ (two independent and structurally distinct molecules per asymmetric unit), $D_m =$

* To whom correspondence should be addressed.

1.45 (5), $D_x = 1.47 \text{ g cm}^{-3}$, Mo $K\alpha$ radiation, $\lambda = 0.710692 \text{ \AA}$, $\mu = 10.3 \text{ cm}^{-1}$, $F(000) = 1104$, room temperature, $R = 0.056$ for 3286 unique reflections. The methyl substituents are *cis* orientated with respect to the pyridine ligand. The Mn—C(cyclopentadienyl) distances are not equivalent; they range from Mn—C(methyl substituted) of 2.209 (5) to 2.111 (5) \AA for the Mn—C(β to the methyl group). The other intramolecular distances are typical for compounds of this type: Mn—C(carbonyl) distances are 1.766 (5) and 1.769 (6) \AA for molecule 1 and 1.778 (5) and 1.761 (5) \AA for molecule 2. The Mn—N distances are 2.023 (4) and 2.038 (4) \AA .

Introduction. Compounds of the type $[(\eta^5\text{-C}_5\text{H}_5)\text{-Mn}(\text{CO})_2\text{L}]$ have been the subject of numerous structural investigations. However, structural data on the ring-substituted derivatives such as $[(\eta^5\text{-C}_5\text{H}_4\text{CH}_3)\text{Mn}(\text{CO})_2\text{L}]$ are less common. These data are of interest because the position of the methyl substituent relative to the ligand *L* may be informative as to the electronic nature of *L* (Wrighton, Abrahamson & Morse, 1976). Also structural information on complexes containing the $(\eta^5\text{-C}_5\text{H}_4\text{X})\text{Mn}(\text{CO})_2$ unit bound to good Lewis bases may provide information on the effect of an electron-rich centre on the metal— $\text{C}_5\text{H}_4\text{X}$ interaction (Westcott, Kakkar, Stringer, Taylor & Marder, 1990). To date there are no structural data on $[(\eta^5\text{-C}_5\text{H}_4\text{CH}_3)\text{Mn}(\text{CO})_2(\text{pyridine})]$ or its derivatives. Therefore we have synthesized and structurally characterized $[(\eta^5\text{-C}_5\text{H}_4\text{CH}_3)\text{Mn}(\text{CO})_2(\text{pyridine})]$ principally to determine the orientation of the methyl substituent with respect to the pyridine ligand in the solid state and also to determine if the metal to cyclopentadienyl-carbon bond vectors have equivalent lengths.

Experimental. The title compound was synthesized *via* the photochemical elimination of CO from commercially available tricarbonyl(methylcyclopentadienyl)manganese(I) in tetrahydrofuran, and reaction of the resultant tetrahydrofuran adduct with pyridine. Suitable crystals for diffraction analysis were grown from degassed ethanol solution at 252 K. A crystal of the dark red compound was exposed to air thus producing a layer of manganese oxide on the crystal surface which protected the bulk of the crystal from further oxidation.

A rectangular prismatic crystal of dimensions $0.3 \times 0.3 \times 0.5 \text{ mm}$ of the title compound was selected and mounted on a Nicolet *P3* four-circle diffractometer. The density of the crystal was measured by a gradient-tube technique (*International Tables for X-ray Crystallography*, 1985, Vol III). Cell dimensions were determined by fitting 14 reflections in the range $22.0 < 2\theta < 23.5^\circ$ and centred on the diffrac-

tometer using graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71069 \text{ \AA}$). A total of 6196 reflections were measured in the range $h - 10 \rightarrow 10$, $k 0 \rightarrow 23$, $l 0 \rightarrow 20$ out to $2\theta = 55^\circ$ using a $\theta/2\theta$ scan technique. Of the 5628 unique reflections, 3286 reflections with $I > 2\sigma(I)$ were considered observed ($R_{\text{int}} = 0.0548$). No absorption correction was applied to these data ($\mu = 10.3 \text{ cm}^{-1}$). Standard reflections 402 and 0,12,0 were measured at an interval of 50 measurements and their intensity decreased by 25% during the data collection. The data were corrected by a linear interpolation between the standard intensity values.

The structure was solved by the application of the automatic Patterson interpretation to find the heavy atoms, followed by partial structure expansion to find all non-H atoms using *SHELXS86* (Sheldrick, 1990). The atom coordinates were refined with full-matrix least-squares refinement using *SHELX76* (Sheldrick, 1976). Anisotropic displacement parameters were used for all non-H atoms. A total of 315 parameters were refined. The H atoms were located in their calculated positions and refined riding on the non-H atom to which they are attached with a different common U_{iso} of 0.05 and 0.13 \AA^2 for the aromatic and methyl H atoms, respectively. In the final stages of refinement the CH_3 unit was treated as a rigid body. This resulted in discrepancy indices of $R = 0.056$ and $wR = 0.055$ $\{w = 1.7056/[\sigma^2(F) + 0.001F^2]\}$. The maximum ratio of the least-squares shift to e.s.d. in the final cycle of refinement was 0.163 for non-H atoms. No corrections were made for extinction. The maximum and minimum features in the final $\Delta\rho$ map were 0.529 and $-0.370 \text{ e \AA}^{-3}$, respectively. Atomic scattering factors for manganese were taken from *International Tables for X-ray Crystallography* (1974, Vol IV). Other scattering factors required were contained within the refinement program. Final atomic parameters are given in Table 1* for all non-H atoms, while a view of the molecular structure of one asymmetric unit along with the atom-numbering scheme is given in Fig. 1. Fig. 2 represents the packing diagram for the unit-cell contents. Table 2 contains a listing of important bond lengths and angles. Other crystallographic programs used on a VAX 6230 computer included *SCHAKAL88* (Keller, 1988).

Discussion. Fig. 1 shows the molecular structure of the two molecules in the asymmetric unit. The principal differences between the structures of the two

* Lists of structure factors, anisotropic displacement parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54829 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0251]

Table 1. Atomic coordinates ($\times 10^4$) and U_{eq} values ($\text{\AA}^2 \times 10^3$) for non-H atoms with *e.s.d.*'s in parentheses

	x	y	z	U_{eq}
Mn(1)	343 (1)	2045 (1)	1302 (1)	39 (1)
N(1)	-664 (5)	1713 (2)	2305 (2)	37 (1)
C(1)	-20 (7)	1174 (3)	2763 (3)	50 (1)
C(2)	-504 (8)	976 (3)	3517 (4)	60 (2)
C(3)	-1737 (8)	1335 (4)	3816 (4)	68 (2)
C(4)	-2456 (8)	1883 (3)	3345 (4)	61 (2)
C(5)	-1908 (7)	2045 (3)	2596 (3)	49 (1)
C(6)	3103 (7)	2234 (3)	1878 (4)	54 (2)
C(7)	2685 (8)	2387 (3)	973 (4)	63 (2)
C(8)	1413 (9)	2885 (3)	835 (4)	71 (2)
C(9)	1050 (8)	3045 (3)	1674 (4)	63 (2)
C(10)	2106 (7)	2634 (3)	2310 (4)	50 (1)
C(11)	2255 (10)	2644 (4)	3282 (4)	76 (2)
C(12)	595 (7)	1284 (3)	825 (3)	48 (1)
C(13)	-1769 (8)	2158 (3)	600 (3)	49 (1)
O(1)	833 (6)	799 (2)	487 (3)	77 (1)
O(2)	-3130 (6)	2233 (2)	119 (3)	73 (1)
Mn(1A)	5350 (1)	338 (1)	6681 (1)	34 (1)
N(1A)	4392 (5)	63 (2)	7736 (2)	40 (1)
C(1A)	3196 (7)	444 (3)	8004 (4)	59 (2)
C(2A)	2546 (8)	293 (4)	8731 (4)	77 (2)
C(3A)	3155 (9)	-259 (4)	9203 (4)	74 (2)
C(4A)	4344 (10)	-634 (3)	8942 (4)	75 (2)
C(5A)	4941 (8)	-460 (3)	8209 (3)	54 (2)
C(6A)	5975 (7)	1345 (2)	6926 (3)	47 (1)
C(7A)	6189 (8)	1163 (3)	6082 (3)	50 (1)
C(8A)	7502 (7)	690 (3)	6188 (3)	51 (1)
C(9A)	8138 (6)	576 (3)	7109 (3)	46 (1)
C(10A)	7203 (7)	995 (2)	7556 (3)	42 (1)
C(11A)	7485 (9)	1056 (3)	8539 (4)	67 (2)
C(12A)	3206 (7)	391 (3)	5977 (3)	43 (1)
C(13A)	5630 (7)	-460 (2)	6330 (3)	42 (1)
O(1A)	1826 (5)	430 (2)	5511 (2)	61 (1)
O(2A)	5850 (5)	-974 (2)	6068 (3)	59 (1)

molecules are (i) the dihedral angle C(10)—Cp—Mn(1)—N(1), where Cp is the centroid of the cyclopentadienyl ring, is $1.6(2)^\circ$ in molecule 1 and $6.2(2)^\circ$ in molecule 2, (ii) the angle between the mean planes of the cyclopentadienyl and pyridine rings is $24.7(2)^\circ$ for molecule 1 while the equivalent angle in molecule 2 is $33.9(2)^\circ$. The Mn—N distances are $2.023(4) \text{ \AA}$ (molecule 1) and $2.038(4) \text{ \AA}$ (molecule 2). The two molecules in the asymmetric unit are independent with no significant intermolecular interactions. The orientation of the methyl group on the cyclopentadienyl ring is *cis* to the pyridine ligand. This structural feature appears to be common to all compounds containing the $(\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{X})\text{Mn}(\text{CO})_2$ fragment bound to a nitrogen donor ligand (Weidenhammer, Herrmann & Ziegler, 1979; Wang, Lee, Wen & Liu, 1991). The preferred orientation in compounds where the ligand *L* is $(\text{C}_6\text{H}_5)_3\text{P}$ (Zaworotko, Shakir, Atwood, Sriyungwat, Reynolds & Albright, 1982), $[(\text{CH}_3)_2(\text{C}_2\text{H}_5)\text{S}]^+$ (Adams & Chodosh, 1978), and anthronylketene (Herrmann, Plank, Ziegler & Weidenhammer, 1979) is *trans* to a carbonyl group. The orientation in the title compound causes the mean plane of the cyclopentadienyl ring to tilt slightly from orthogonality to the Mn to ring-centroid vector [$92.6(3)^\circ$ molecule 1; $93.2(2)^\circ$

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

Cp = ring centroid of the cyclopentadienyl ring.			
Molecule 1			
N(1)—Mn(1)	2.023 (4)	C(6)—Mn(1)	2.162 (5)
C(7)—Mn(1)	2.118 (6)	C(8)—Mn(1)	2.117 (6)
C(9)—Mn(1)	2.173 (6)	C(10)—Mn(1)	2.201 (5)
C(12)—Mn(1)	1.766 (6)	C(13)—Mn(1)	1.769 (6)
Mn(1)—Cp	1.786 (1)		
Molecule 2			
N(1A)—Mn(1A)	2.038 (4)	C(6A)—Mn(1A)	2.143 (5)
C(7A)—Mn(1A)	2.111 (5)	C(8A)—Mn(1A)	2.119 (5)
C(9A)—Mn(1A)	2.171 (5)	C(10A)—Mn(1A)	2.209 (5)
C(12A)—Mn(1A)	1.778 (5)	C(13A)—Mn(1A)	1.761 (5)
Mn(1A)—Cp(A)	1.785 (1)		
Molecule 1			
C(12)—Mn(1)—Cp	122.7 (2)	C(10)—Cp—Mn(1)	92.6 (3)
N(1)—Mn(1)—Cp	118.5 (1)	C(13)—Mn(1)—Cp	125.8 (2)
C(6)—Mn(1)—N(1)	105.0 (2)	C(7)—Mn(1)—N(1)	143.5 (2)
C(7)—Mn(1)—C(6)	38.5 (2)	C(8)—Mn(1)—N(1)	142.6 (2)
C(8)—Mn(1)—C(6)	64.4 (2)	C(8)—Mn(1)—C(7)	38.7 (2)
C(9)—Mn(1)—N(1)	103.4 (2)	C(9)—Mn(1)—C(6)	63.4 (2)
C(9)—Mn(1)—C(7)	64.7 (2)	C(9)—Mn(1)—C(8)	39.2 (2)
C(10)—Mn(1)—N(1)	85.3 (2)	C(10)—Mn(1)—C(6)	37.5 (2)
C(10)—Mn(1)—C(7)	64.1 (2)	C(10)—Mn(1)—C(8)	64.5 (2)
C(10)—Mn(1)—C(9)	37.8 (2)	C(12)—Mn(1)—N(1)	97.3 (2)
C(12)—Mn(1)—C(6)	98.1 (2)	C(12)—Mn(1)—C(7)	90.5 (2)
C(12)—Mn(1)—C(8)	119.2 (3)	C(12)—Mn(1)—C(9)	155.2 (3)
C(12)—Mn(1)—C(10)	132.9 (2)	C(13)—Mn(1)—N(1)	93.4 (2)
C(13)—Mn(1)—C(6)	157.8 (2)	C(13)—Mn(1)—C(7)	122.1 (2)
C(13)—Mn(1)—C(8)	93.4 (3)	C(13)—Mn(1)—C(9)	100.7 (2)
C(13)—Mn(1)—C(10)	135.3 (2)	C(13)—Mn(1)—C(12)	91.7 (2)
C(1)—N(1)—Mn(1)	121.8 (3)	C(5)—N(1)—Mn(1)	121.6 (3)
Molecule 2			
C(12A)—Mn(1A)—Cp(A)	124.0 (2)	C(10A)—Cp(A)—Mn(1A)	93.2 (2)
N(1A)—Mn(1A)—Cp(A)	121.3 (1)	C(13A)—Mn(1A)—Cp(A)	123.4 (2)
C(6A)—Mn(1A)—N(1A)	103.5 (2)	C(7A)—Mn(1A)—N(1A)	142.0 (2)
C(7A)—Mn(1A)—C(6A)	38.9 (2)	C(8A)—N(1A)—N(1A)	148.4 (2)
C(8A)—Mn(1A)—C(6A)	64.5 (2)	C(8A)—Mn(1A)—C(7A)	38.4 (2)
C(9A)—Mn(1A)—N(1A)	109.5 (2)	C(9A)—Mn(1A)—C(6A)	63.7 (2)
C(9A)—Mn(1A)—C(7A)	64.5 (2)	C(9A)—Mn(1A)—C(8A)	39.9 (2)
C(10A)—Mn(1A)—N(1A)	88.5 (2)	C(10A)—Mn(1A)—C(6A)	37.6 (2)
C(10A)—Mn(1A)—C(7A)	63.9 (2)	C(10A)—Mn(1A)—C(8A)	63.9 (2)
C(10A)—Mn(1A)—C(9A)	37.6 (2)	C(12A)—Mn(1A)—N(1A)	93.2 (2)
C(12A)—Mn(1A)—C(6A)	101.2 (2)	C(12A)—Mn(1A)—C(7A)	90.9 (2)
C(12A)—Mn(1A)—C(8A)	117.3 (2)	C(12A)—Mn(1A)—C(9A)	154.8 (2)
C(12A)—Mn(1A)—C(10A)	137.1 (2)	C(13A)—Mn(1A)—N(1A)	95.0 (2)
C(13A)—Mn(1A)—C(6A)	156.8 (2)	C(13A)—Mn(1A)—C(7A)	122.6 (2)
C(13A)—Mn(1A)—C(8A)	92.4 (2)	C(13A)—Mn(1A)—C(9A)	97.0 (2)
C(13A)—Mn(1A)—C(10A)	130.9 (2)	C(13A)—Mn(1A)—C(12A)	91.6 (2)
C(1A)—N(1A)—Mn(1A)	119.5 (4)	C(5A)—N(1A)—Mn(1A)	123.3 (4)

molecule 2]. Thus the Mn to C(π) distances are not equivalent, ranging in molecule 1 from the longest [Mn(1)—C(10)] at $2.201(5) \text{ \AA}$ to the shortest [Mn(1)—C(8)] at $2.117(6) \text{ \AA}$, the Mn(1) to ring-centroid distance is $1.786(1) \text{ \AA}$ [the equivalent distances in molecule 2 are $2.209(5)$, $2.119(6)$ and $1.785(1) \text{ \AA}$ respectively]; the shortest Mn—C(π) distance in molecule 2 is $2.111(5) \text{ \AA}$ for Mn(1A)—C(7A). This observation contrasts with similar compounds in which all the Mn to C(π) distances are the same within error: $2.132(9)$ – $2.162(8) \text{ \AA}$ for $L = (\text{C}_6\text{H}_5)_3\text{P}$; $2.14(2)$ – $2.16(2) \text{ \AA}$, $L = \text{methylmethoxycarbene}$ (Fontana, Schubert & Fischer, 1978); $2.17(2)$ – $2.19(2) \text{ \AA}$, $L = \text{tricarbonyl}[1-3-\eta-(3\text{-oxo-1-phenylpropane})\text{jiron}$ (Andrianov, Struchkov, Kolobova, Antonova & Obezyuk, 1976); $2.159(8)$ – $2.18(1) \text{ \AA}$, $L = \text{bicyclo}[2.2.1]\text{hepta-2,5-diene}$ (Grannoff & Jacobson, 1968); and $2.07(2)$ – $2.11(1) \text{ \AA}$, $L =$

sulfur dioxide (Barbeau & Dubey, 1973). It would seem likely that the variation in the Mn—C(π) distances in the title compound are not solely the result of steric factors and may be the result of the increased electron density on the metal atom because of the presence of the good σ -donor but a poor

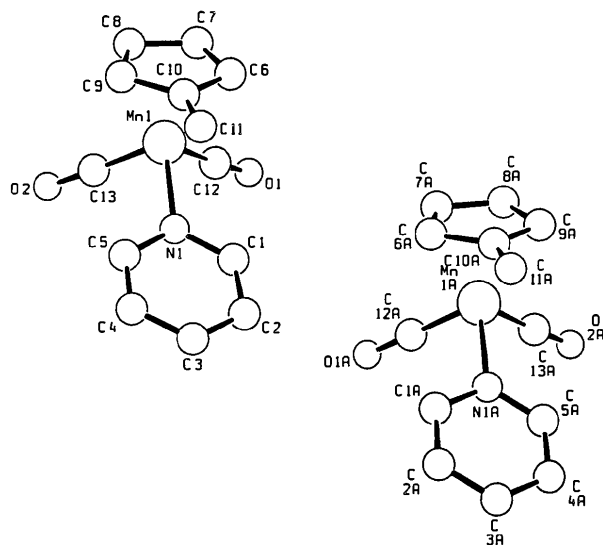


Fig. 1. A SCHAKAL drawing of the molecular structures of the two molecules in the asymmetric unit, indicating the atom-numbering system used. H atoms have been omitted for clarity as have bonds between the Mn atoms and the cyclopentadienyl C atoms.

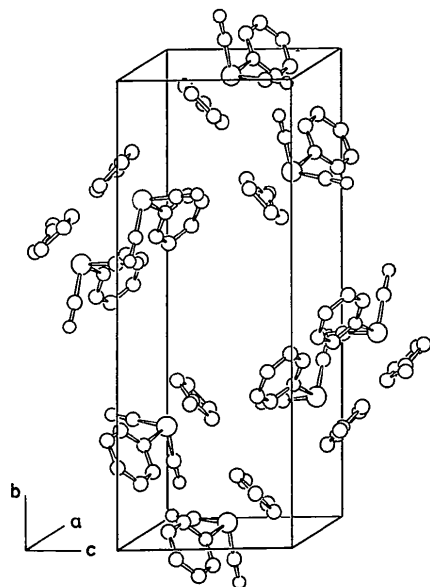


Fig. 2. A SCHAKAL drawing of the unit-cell contents; the H atoms and the bonds between the Mn atoms and the cyclopentadienyl C atoms have been omitted for clarity.

π -acceptor pyridine ligand. The five cyclopentadienyl C atoms in molecule 1 are essentially coplanar, the maximum deviation from the mean plane being 0.002 (5) Å for C(8); however, in molecule 2 the deviation from the mean plane is greater with C(10) 0.011 (5) Å out of the plane. As expected, the methyl C is displaced by 0.075 (5) Å [0.057 (6) Å for molecule 2] from the mean plane of the cyclopentadienyl ring (Chinn & Hall, 1983).

The Mn(1)—(CO) and C—O distances and the Mn—C—O angles are within the ranges expected for this class of compound. The angles within the Mn(CO)₂L tripod are 91.7 (2)° for C(13)—Mn(1)—C(12), 93.4 (2)° for C(13)—Mn(1)—N(1) and 97.3 (2)° for C(12)—Mn(1)—N(1) [91.6 (2), 95.0 (2) and 93.2 (2)° for molecule 2]. The angles around the Mn involving Mn—X [X = C(carbonyl) or N] and the Mn—Cp where Cp is the centroid of the cyclopentadienyl ring (Table 2) clearly show the distortion of the tetrahedral geometry of the Mn coordination such that the X—Mn—X and X—Mn—Cp angles are less and greater, respectively, than the ideal values.

The authors acknowledge the financial support of the European Community for this research.

References

- ADAMS, R. D. & CHODOSH, D. F. (1978). *J. Am. Chem. Soc.* **100**, 812–817.
- ANDRIANOV, V. G., STRUCHKOV, YU. T., KOLOBOVA, N. E., ANTONOVA, A. B. & OBEZYUK, N. S. (1976). *J. Organomet. Chem.* **122**, C33–C36.
- BARBEAU, C. & DUBEY, R. J. (1973). *Can. J. Chem.* **51**, 3684–3689.
- CHINN, J. W. & HALL, M. B. (1983). *J. Am. Chem. Soc.* **105**, 4930–4941.
- FONTANA, S., SCHUBERT, U. & FISCHER, E. O. (1978). *J. Organomet. Chem.* **146**, 39–44.
- GRANOFF, B. & JACOBSON, R. A. (1968). *Inorg. Chem.* **7**, 2328–2333.
- HERRMANN, W. A., PLANK, J., ZIEGLER, M. L. & WEIDENHAMMER, K. (1979). *J. Am. Chem. Soc.* **101**, 3133–3135.
- KELLER, E. (1988). SCHAKAL88. Program for the Graphical Representation of Molecular and Crystallographic Models. Univ. of Freiburg, Germany.
- SHELDRIK, G. M. (1976). SHELX76. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- WANG, T.-F., LEE, Y.-S., WEN, Y.-S. & LIU, L.-K. (1991). *J. Organomet. Chem.* **403**, 353–358.
- WEIDENHAMMER, K., HERRMANN, W. A. & ZIEGLER, M. L. (1979). *Z. Anorg. Allg. Chem.* **457**, 183–188.
- WESTCOTT, S. A., KAKKAR, A. K., STRINGER, G., TAYLOR, N. J. & MARDER, T. B. (1990). *J. Organomet. Chem.* **394**, 777–794.
- WRIGHTON, M. S., ABRAHAMSON, H. B. & MORSE, D. L. (1976). *J. Am. Chem. Soc.* **98**, 4105–4109.
- ZAWOROTKO, M. J., SHAKIR, R., ATWOOD, J. L., SRIYUNYONGWAT, V., REYNOLDS, S. D. & ALBRIGHT, T. A. (1982). *Acta Cryst.* **B38**, 1572–1574.