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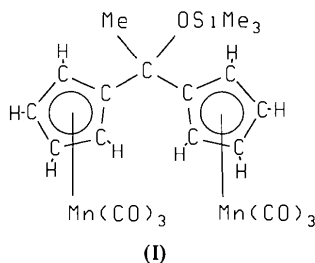

Structure of Mn(CO)₃[C₅H₄C(Me)(OSiMe₃)C₅H₄]Mn(CO)₃

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Abstract. 1,1-Bis(η^5-cyclopentadienyl)-1-(trimethylsiloxy)ethane-bis(tricarbonyl-manganese), $C_{21}H_{20}Mn_2O_7Si$, $M_r = 522.4$, monoclinic, $P2_1/c$, $a = 12.729 (3)$, $b = 8.112 (3)$, $c = 22.432 (6)$ Å, $\beta = 96.51 (2)°$, $V = 2301 (2)$ Å^3, $Z = 4$, $D_x = 1.51$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 11.4$ cm^{-1}, $F(000) = 1064$, $T = 293$ K, $R = 0.044$ for 1637 reflections with $F_o^2 > 3\sigma(F_o^2)$. The dihedral angle between the two planar cyclopentadiene (cp) units is 99·0° and the two Mn(CO)₃ units are pentahapto bonded to these cp rings on the outside of the cp₂C(Me)OSiMe₃ molecule.

Experimental. Title compound (I) obtained by the

(I)

reaction of KH with a THF solution of Mn(CO)₃-C₅H₄C(Me)(OH)C₅H₄Mn(CO)₃ (Kolobova, Valueva, Anisimov & Suleimanov, 1978) followed by the addition of ClSiMe₃. Data crystal obtained by slow evaporation of a pentane–benzene (20:1) solution. Colorless platelet crystal 0·08 × 0·24 × 0·46 mm mounted on a glass fiber with epoxy. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using ω–2θ scans of 4–16° min^{-1} in θ. Unit cell

determined from least-squares analysis of angle data for 25 reflections with $13 < 2\theta < 19°$. Absorption correction based on ψ scans varied from 0·95 to 1·00. Data collected to $(\sin\theta)/\lambda$ of 0·59 Å^{-1}, $0 < h < 15$, $-9 < k < 0$, $-26 < l < 26$. Three standard reflections (008, $1\bar{2}6$, 500) decreased 1·7% over 34·2 h of data collection; a linear correction was applied. 4239 reflections measured, 4044 unique ($R_{int} = 0.03$), 2407 reflections with $I < 3\sigma(I)$ where $\sigma^2(I) = \sigma_{cs}^2(I) + (0.05I)^2$; $\sigma_{cs}(I)$ is standard deviation of I based on counting statistics. Solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w(F_o - F_c)^2$. H atoms were constrained to idealized positions (C—H = 0·95 Å) with fixed isotropic B values of 1·2 times the B value of the attached C atoms; the orientations of the methyl H atoms were determined from difference maps. The non-H atoms were refined anisotropically for a total of 280 parameters. $R = 0.044$, $wR = 0.049$, GOF = 1·1, where non-Poisson $w^{-1} = [\sigma^2(I) + (0.05I)^2]/4F^2$. Final $(\Delta/\sigma)_{max} < 0.1$, $\rho_{max} = 0.33 (7)$ and $\rho_{min} = -0.32 (7)$ e Å^{-3} on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used were those of Enraf–Nonius (1982) *SDP.** Table

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* Tables of H-atom positions, anisotropic temperature factors and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51829 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

$C_{21}H_{20}Mn_2O_7Si$

Table 1. Final fractional coordinates and B_{eq} thermal factors for non-H atoms

E.s.d.'s in parentheses are in the units of the least-significant digit.

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j B_{ij}(\mathbf{a}_i \cdot \mathbf{a}_j).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Mn(1)	0·70532 (9)	0·2072 (2)	0·53269 (5)	2·68 (2)
Mn(2)	0·62182 (9)	0·4470 (2)	0·29437 (5)	2·94 (3)
Si	0·9491 (2)	0·2044 (2)	0·3694 (1)	3·35 (5)
O(1)	0·4772 (4)	0·2301 (9)	0·4968 (3)	6·3 (2)
O(2)	0·6755 (6)	−0·0260 (9)	0·6284 (3)	7·3 (2)
O(3)	0·7278 (5)	−0·0711 (7)	0·4510 (2)	5·1 (2)
O(4)	0·4572 (5)	0·4458 (8)	0·1912 (3)	5·8 (2)
O(5)	0·7432 (6)	0·7175 (9)	0·2479 (3)	7·2 (2)
O(6)	0·7335 (5)	0·1934 (9)	0·2342 (3)	6·9 (2)
O(7)	0·8389 (4)	0·2586 (6)	0·3962 (2)	2·5 (1)
C(1)	0·5668 (6)	0·218 (1)	0·5107 (4)	3·7 (2)
C(2)	0·6868 (7)	0·066 (1)	0·5912 (3)	4·2 (2)
C(3)	0·7180 (6)	0·037 (1)	0·4819 (3)	3·2 (2)
C(4)	0·5218 (6)	0·451 (1)	0·2307 (3)	3·8 (2)
C(5)	0·6956 (7)	0·609 (1)	0·2650 (4)	4·3 (2)
C(6)	0·6914 (7)	0·291 (1)	0·2583 (4)	4·3 (2)
C(7)	0·6111 (6)	0·294 (1)	0·3709 (3)	3·0 (2)
C(8)	0·5114 (6)	0·360 (1)	0·3524 (4)	3·8 (2)
C(9)	0·5197 (6)	0·532 (1)	0·3563 (3)	3·7 (2)
C(10)	0·6246 (6)	0·573 (1)	0·3783 (3)	2·9 (2)
C(11)	0·6829 (5)	0·4241 (9)	0·3869 (3)	2·2 (2)
C(12)	0·7954 (6)	0·4061 (9)	0·4185 (3)	2·4 (2)
C(13)	0·7897 (6)	0·3841 (9)	0·4855 (3)	2·4 (2)
C(14)	0·7213 (6)	0·4685 (9)	0·5202 (3)	2·9 (2)
C(15)	0·7496 (6)	0·427 (1)	0·5813 (3)	3·8 (2)
C(16)	0·8368 (6)	0·321 (1)	0·5852 (4)	3·7 (2)
C(17)	0·8618 (6)	0·294 (1)	0·5259 (3)	3·0 (2)
C(18)	0·8622 (6)	0·558 (1)	0·4088 (3)	3·1 (2)
C(19)	0·9247 (8)	−0·010 (1)	0·3450 (4)	5·7 (3)
C(20)	0·9813 (7)	0·331 (1)	0·3054 (4)	5·8 (3)
C(21)	1·0644 (7)	0·210 (1)	0·4281 (4)	5·4 (2)

Table 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses

Mn(1)—C(1)	1·779 (9)	Si—O(7)	1·647 (5)
Mn(1)—C(2)	1·776 (10)	Si—C(19)	1·835 (10)
Mn(1)—C(3)	1·810 (10)	Si—C(20)	1·849 (10)
Mn(1)—C(13)	2·142 (7)	Si—C(21)	1·859 (10)
Mn(1)—C(14)	2·151 (8)	O(1)—C(1)	1·151 (9)
Mn(1)—C(15)	2·132 (8)	O(2)—C(2)	1·142 (10)
Mn(1)—C(16)	2·142 (8)	O(3)—C(3)	1·133 (9)
Mn(1)—C(17)	2·135 (7)	O(4)—C(4)	1·139 (8)
Mn(2)—C(4)	1·802 (9)	O(5)—C(5)	1·160 (10)
Mn(2)—C(5)	1·783 (11)	O(6)—C(6)	1·130 (10)
Mn(2)—C(6)	1·788 (11)	O(7)—C(12)	1·432 (8)
Mn(2)—C(7)	2·134 (8)	C(11)—C(12)	1·530 (9)
Mn(2)—C(8)	2·143 (9)	C(12)—C(13)	1·525 (9)
Mn(2)—C(9)	2·122 (8)	C(12)—C(18)	1·527 (10)
Mn(2)—C(10)	2·139 (7)		
Mn(2)—C(11)	2·141 (6)		

C(1)—Mn(1)—C(2)	91·6 (4)	Mn(1)—C(1)—O(1)	178·2 (9)
C(1)—Mn(1)—C(3)	91·2 (4)	Mn(1)—C(2)—O(2)	179·2 (9)
C(2)—Mn(1)—C(3)	90·1 (4)	Mn(1)—C(3)—O(3)	178·5 (8)
C(4)—Mn(2)—C(5)	92·9 (4)	Mn(2)—C(4)—O(4)	176·9 (9)
C(4)—Mn(2)—C(6)	89·6 (4)	Mn(2)—C(5)—O(5)	177·5 (8)
C(5)—Mn(2)—C(6)	92·5 (4)	Mn(2)—C(6)—O(6)	178·2 (9)
O(7)—Si—C(19)	103·8 (4)	O(7)—C(12)—C(11)	107·3 (6)
O(7)—Si—C(20)	114·0 (4)	O(7)—C(12)—C(13)	108·1 (6)
O(7)—Si—C(21)	111·8 (3)	O(7)—C(12)—C(18)	112·4 (6)
C(19)—Si—C(20)	109·9 (5)	C(11)—C(12)—C(13)	108·7 (6)
C(19)—Si—C(21)	109·2 (5)	C(11)—C(12)—C(18)	111·3 (6)
C(20)—Si—C(21)	108·0 (5)	C(13)—C(12)—C(18)	108·9 (6)
		Si—O(7)—C(12)	136·8 (5)

Cordes, Durham & Askew (1989). Related diiron (Weaver & Woodward, 1973) and ditungsten (Abriel & Heck, 1986) compounds have been reported and the structure of a monomeric manganese subunit has been reported (Berndt & Marsh, 1963).
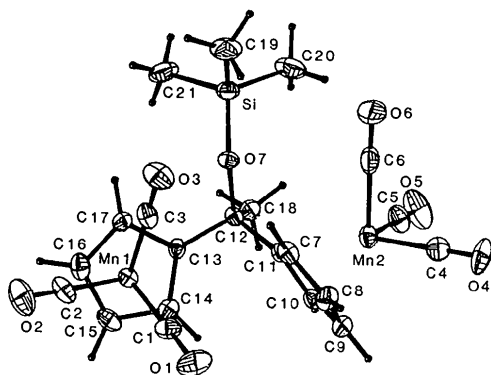
Fig.1. ORTEP (Johnson, 1976) drawing and numbering scheme. The ellipsoids are drawn at the 30% probability surface and H atoms have been given arbitrary radii for figure clarity.

1 gives the atomic coordinates and Table 2 selected bond distances and angles. Fig. 1 shows the molecule with the numbering scheme.

Related literature. Two other dimanganese complexes of this type, with —C(Me)(OH)— and —C(O)-CH=CMe— bridging units, have been reported by

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