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Bromopropyldicarbonyl(η^5-pentamethylcyclopentadienyl)iron(II)

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Key indicators

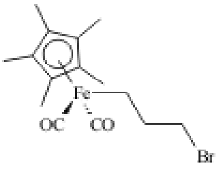
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.120
Data-to-parameter ratio = 28.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In Cp*Fe(CO)$_2$(n-C$_3$H$_6$Br), where Cp* = η^5-C$_5$(CH$_3$)$_5$, the Fe atom is coordinated by a pentamethylcyclopentadienyl ligand, two carbonyl ligands and a bromoalkyl chain in a pseudo-octahedral arrangement. The molecular geometry is similar to that reported for a related structure, with the alkyl chain in an all-*trans* conformation and a Fe—C(alkyl) bond length of 2.057 (3) Å. In the crystal structure, the molecules pack in layers.

Comment

This structure determination was undertaken as part of our ongoing study of halogenoalkyl transition metal compounds (Friedrich *et al.*, 2004). Very few structures of halogenoalkyl complexes have been reported and, to our knowledge, only three structures are known where the halogenoalkyl group has a chain length exceeding one carbon (Friedrich *et al.*, 2001). Furthermore, these are only for compounds of the group 6 metals W and Mo.



(I)

The title compound, (I), is, to our knowledge, the first crystal structure reported for an iron halogenoalkyl molecule. The structure of the related iron alkyl compound, Cp*Fe(CO)$_2$(n-C$_5$H$_{11}$) has been determined (Hill *et al.*, 1999). The shorter chain and the introduction of a bromine atom in the alkyl chain in the title compound has little effect on the molecular geometry, but it does influence the packing of molecules compared with the structure of Cp*Fe(CO)$_2$(n-C$_5$H$_{11}$).

In (I), the Fe atom is coordinated in a pseudo-octahedral fashion by a pentahapto-pentamethylcyclopentadienyl ligand, two carbonyls and a bromoalkyl chain. Fig. 1 illustrates the molecular geometry and the numbering scheme employed. The compound crystallizes with four molecules per unit cell, and the asymmetric unit comprises one molecule. For this molecule the C atoms constituting the pentamethylcyclopentadienyl moiety are coplanar, with an r.m.s. deviation of 0.0504 Å, and the five Fe—C bonds range from 2.100 (3) Å to 2.139 (3) Å.

The halogenoalkyl chain is in the energetically favoured extended, all-*trans* conformation, with a C—Br bond length of 1.971 (3) Å, within the expected bond length range. The C13—

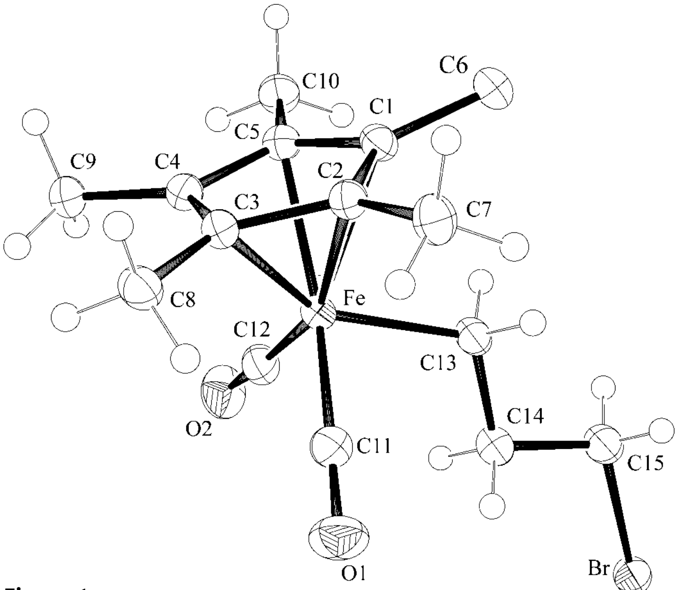


Figure 1
Molecular structure of (I), showing the atomic numbering scheme, with displacement ellipsoids drawn at the 50% probability level (*ORTEP*3, Farrugia, 1997).

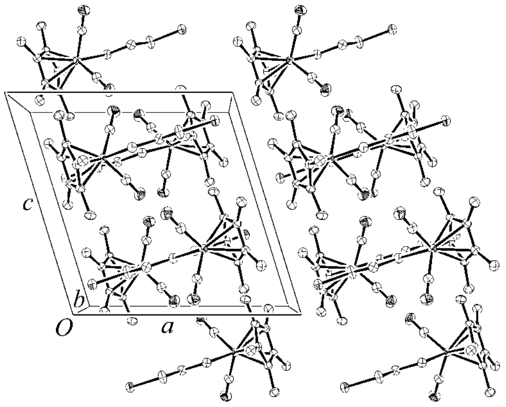


Figure 2
Packing diagram for (I), viewed down the *b* axis, showing the layered packing (H atoms omitted). (*ORTEP*3, Farrugia, 1997).

Fe bond length of 2.057 (3) Å is similar to that reported for Cp*Fe(CO)$_2$(n-C$_5$H$_{11}$) [2.069 (10) Å; Hill *et al.*, 1999]. In the molecule, the atoms constituting the halogenoalkyl chain, the Fe atom and atoms C1 and C6 are coplanar, with an r.m.s. deviation of 0.0091 Å. The plane through these molecules is almost perpendicular to the plane through the ten pentamethylcyclopentadienyl C atoms, with an angle of 89.39 (8)° between the two planes.

The molecular packing in the crystal structure as viewed down the *b* axis is illustrated in Fig. 2 and viewed down the *c* axis in Fig. 3. A layered structure is formed, and the mean plane through the alkyl chain atoms is close to perpendicular to the layer plane defined by the Br atoms, with an angle of 89.15 (7)° between the two planes (Fig. 2). As shown in Fig. 3, the alkyl chains are tilted by 78.4 (1)°, in this plane, relative to the layer plane. Neigbouring molecules along the *b*-axis direction pack with a head-to-tail orientation of successive molecules. In a layer, the pentamethylcyclopentadienyl planes of the molecules are tilted by 19.90 (7)° relative to the layer plane. In the crystal structure of Cp*Fe(CO)$_2$(n-C$_5$H$_{11}$) (Hill *et al.*, 1999), the molecules are tilted at a larger angle relative to the layer plane; however, the value of the angle was not reported.

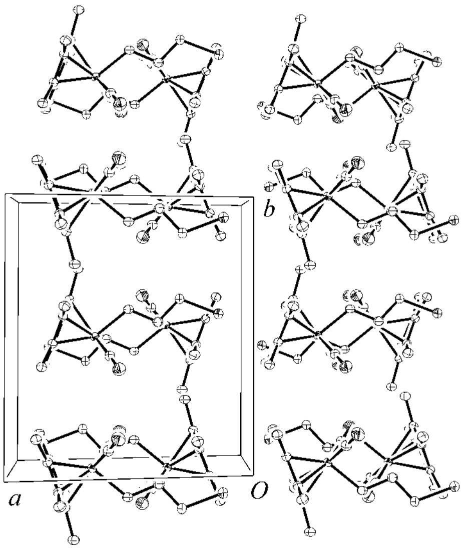


Figure 3
Packing diagram for (I), viewed down the *c* axis (H atoms omitted). (*ORTEP*3, Farrugia, 1997).

Experimental

The compound was prepared according to a reported procedure (Friedrich *et al.*, 1990) and crystals were grown by slow evaporation of a 1:2 mixture of dichloromethane and hexane at 278 K.

Crystal data

[Fe(C$_{10}$H$_{15}$)(C$_3$H$_6$Br)(CO)$_2$]
$M_r = 369.08$
Monoclinic, $P2_1/c$
$a = 11.371$ (9) Å
$b = 12.369$ (11) Å
$c = 11.610$ (8) Å
$\beta = 107.12$ (6)°
$V = 1561$ (2) Å^3
$Z = 4$
$D_x = 1.571$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 636 reflections
$\theta = 2$–32°
$\mu = 3.52$ mm^{-1}
$T = 293$ (2) K
Plate, yellow
0.40 × 0.40 × 0.10 mm

Data collection

Oxford Excalibur2 diffractometer
ω–2θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.262$, $T_{\max} = 0.703$
14319 measured reflections
4989 independent reflections
3757 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.063$
$\theta_{\max} = 31.9$°
$h = -16 \rightarrow 16$
$k = -16 \rightarrow 18$
$l = -16 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.120$
$S = 1.02$
4989 reflections
176 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 1.13$ e Å^{-3}
$\Delta\rho_{\min} = -0.93$ e Å^{-3}

All H atoms were placed in calculated positions, with C—H distances of 0.96–0.97 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ (1.5 for methyl) of the parent atom, and refined using a riding model, with rotation in the

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case of the terminal methyl groups. The maximum electron density peak is 1.615 Å from atom C10 and 1.531 Å from atom H10. The minimum electron density peak is 1.160 Å from atom C15 and 0.689 Å from atom H57*A*.

Data collection: *CrysAlis CCD* (Oxford Diffraction Ltd, 2003); cell refinement: *CrysAlis CCD*; data reduction: *CrysAlis RED* (Oxford Diffraction Ltd, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003) and *WinGX* (Farrugia, 1999).

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