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## Key indicators

Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
Disorder in main residue  
 $R$  factor = 0.025  
 $wR$  factor = 0.069  
Data-to-parameter ratio = 14.6For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Dicarbonyl( $\eta^5$ -1-carboxycyclopentadienyl)-  
methyliron(II)

The title carboxyl-functionalized iron complex,  $[\text{Fe}(\text{CH}_3)(\text{C}_6\text{H}_5\text{O}_2)(\text{CO})_2]$ , was prepared by the reaction of  $\text{CO}_2$  with a lithiated parent unsubstituted compound. The  $\text{Fe}(\text{CO})_2\text{Me}$  legs of the tripodal piano stool are characterized by disorder between the methyl group and the carbonyl group, and the two ligands were refined (50% site occupancy) over the two positions. The ligands are alternately located on either side of a local pseudo-mirror plane running through the carboxylic functionality and the central Fe atom.

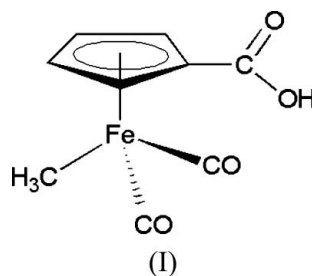
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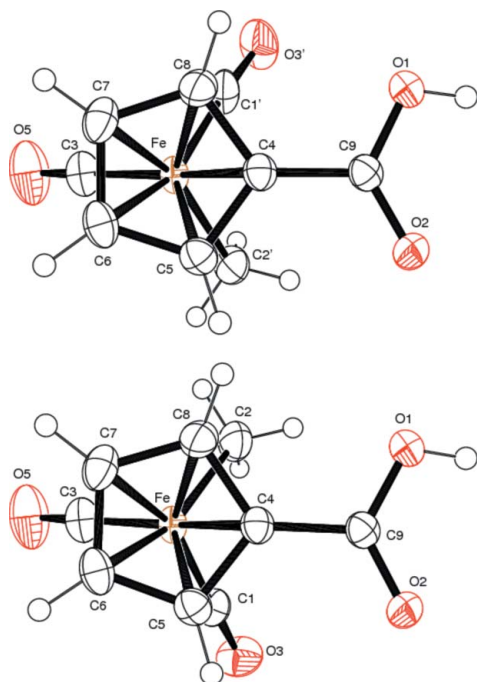
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## Comment

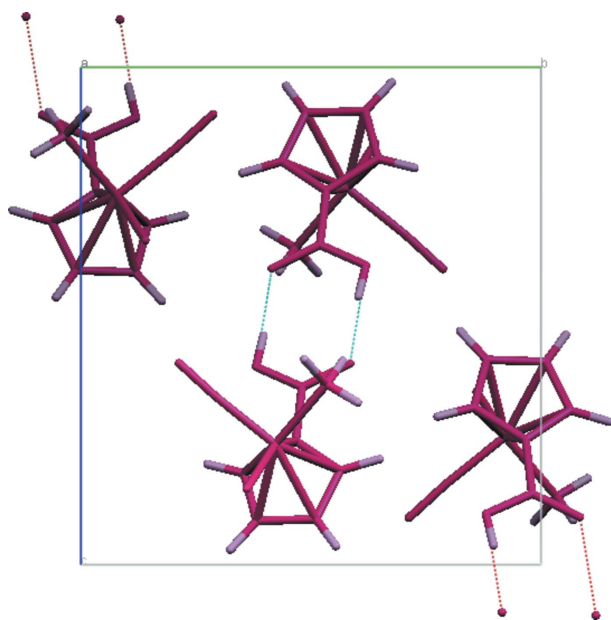
Group six analogues of the title complex have been prepared (El Mouattasim *et al.*, 1994) with the aim of utilizing the functional carboxylic acid group as a labelling agent for amino acids. We prepared the title compound, (I), as part of our interest in solvent-free solid-gas reactions. It was envisaged that the network of hydrogen bonding between neighbouring molecules *via* the carboxylic acid functionality will serve as channels for small gaseous molecules to permeate into the crystal structure (Braga *et al.*, 2002; Fig. 2). Preliminary studies with  $\text{SO}_2$  have indicated otherwise, indicating that intramolecular forces, such as steric interactions between ligands bonded to the central metal atom, may be more crucial in influencing the single-crystal/gas reactivity (Munyaneza *et al.*, 2005).



In the title compound, the  $\text{Fe}(\text{CO})_2\text{Me}$  legs of the tripodal piano stool are characterized by a disordered Me group which was refined over positions C2 and C2'. These are located on opposite sides of a local pseudo-mirror plane running through the carboxylic acid functionality and the central Fe atom, with 50% occupancy of each site (Fig. 1). Mousser *et al.* (1996) have prepared four-legged piano stool tungsten analogues of the title complex, and it is interesting to note that the Me group was similarly disordered between two *trans* positions in *syn* and *anti* configurations relative to the carboxylic acid functional group. In the crystal structure, molecules form the expected centrosymmetric dimers *via*  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds (Table 2 and Fig. 2).



**Figure 1**  
ORTEP3 (Farrugia, 1999) diagrams of the title complex, showing the two conformations with the Me and CO ligands interchanged. Displacement ellipsoids are shown at 50% probability level.



**Figure 2**  
Packing diagram, showing the hydrogen bonding (dashed lines) between neighbouring carboxyl groups.

## Experimental

The title complex was prepared by adapting the method of El Mouattasim *et al.* (1994), except that  $(\eta^5\text{-C}_5\text{H}_5)\text{Fe}(\text{CO})_2\text{Me}$  and butyllithium were substituted for  $(\eta^5\text{-C}_5\text{H}_5)\text{Mo}(\text{CO})_3\text{Me}$  and *sec*-butyllithium. Single crystals suitable for X-ray diffraction were grown from  $\text{CH}_2\text{Cl}_2$ -hexane (70/30). IR  $\nu_{\text{CO}}$  ( $\text{CH}_2\text{Cl}_2$ ,  $\text{cm}^{-1}$ ): 2059 (vs), 1998 (vs);  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , p.p.m.): 5.31 (s, 2H, CpH), 4.86 (s, 2H, CpH), 0.28 (s, 3H, Me).

## Crystal data

$[\text{Fe}(\text{CH}_3)(\text{C}_6\text{H}_5\text{O}_2)(\text{CO})_2]$   
 $M_r = 236.00$   
 Monoclinic,  $P2_1/c$   
 $a = 7.6183$  (15) Å  
 $b = 10.714$  (2) Å  
 $c = 12.039$  (2) Å  
 $\beta = 105.661$  (4)°  
 $V = 946.2$  (3) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.657$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 830 reflections  
 $\theta = 2.6\text{--}30.2^\circ$   
 $\mu = 1.58$  mm<sup>-1</sup>  
 $T = 173$  (2) K  
 Block, brown  
 $0.24 \times 0.19 \times 0.19$  mm

## Data collection

Bruker SMART 1K CCD diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Bruker, 1999)  
 $T_{\text{min}} = 0.704$ ,  $T_{\text{max}} = 0.754$   
 6617 measured reflections

2276 independent reflections  
 1978 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$   
 $\theta_{\text{max}} = 28.0^\circ$   
 $h = -10 \rightarrow 9$   
 $k = -13 \rightarrow 14$   
 $l = -11 \rightarrow 15$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.025$   
 $wR(F^2) = 0.070$   
 $S = 1.08$   
 2276 reflections  
 156 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0402P)^2 + 0.1317P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.034$   
 $\Delta\rho_{\text{max}} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.30$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

C2—Fe	2.031 (8)	C2'—Fe	2.004 (9)
C1—O3	1.164 (7)	C3—O5	1.124 (2)
C1—Fe	1.782 (7)	C3—Fe	1.7670 (19)
C1'—O3'	1.146 (7)	C9—O2	1.2472 (19)
C1'—Fe	1.790 (8)	C9—O1	1.2926 (19)
O3—C1—Fe	178.9 (16)	C3—Fe—C1	88.1 (6)
O3'—C1'—Fe	176.1 (18)	C3—Fe—C2	90.3 (5)
O5—C3—Fe	179.7 (2)	C1—Fe—C2	87.2 (8)
O2—C9—O1	123.71 (15)		

**Table 2**

Hydrogen-bond geometry (Å, °).

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
O1—H1 <sup>i</sup> ⋯O2 <sup>i</sup>	0.84	1.79	2.6240 (19)	174

Symmetry code: (i)  $-x + 2, -y, -z$ .

The H atom attached to the O atom was positioned geometrically and allowed to ride [ $\text{O—H} = 0.84$  Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ ]. The H atoms attached to aromatic C atoms were positioned geometrically and allowed to ride on their parent atoms during refinement, with  $\text{C—H} = 0.95$  Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . The disordered methyl H atoms were accounted for using a two-site model, in which the H atoms were placed geometrically and allowed to ride, with  $\text{C—H} = 0.98$  Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL and ORTEP3 (Farrugia, 1999); software used to prepare material for publication: SHELXTL.

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