

## [3]Ferrocenophan-1-one

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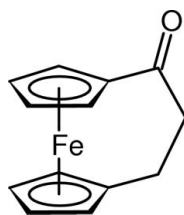
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 Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.039;  $wR$  factor = 0.096; data-to-parameter ratio = 16.3.

The crystal structure of [3]ferrocenophan-1-one,  $[\text{Fe}(\text{C}_{13}\text{H}_{12}\text{O})]$ , has been redetermined at 150 K. The tethered cyclopentadienyl (Cp) rings are tilted by  $9.39$  ( $18^\circ$ ) and assume an eclipsed conformation. The 1-oxopropane-1,3-diyl bridge has a pseudo-envelope conformation with the  $\text{C}=\text{O}$  group deviating by as much as  $22.5$  ( $2^\circ$ ) from coplanarity with its attached Cp ring.

### Related literature

For an overview of the chemistry of ferrocene, see: Štěpnička (2008). For the preparation of the title compound, see: Turbitt & Watts (1972). For its crystal structure at room temperature, see: Jones *et al.* (1965). For an introductory review on the chemistry of ferrocenophanes with carbon bridges, see: Heo & Lee (1999).



### Experimental

#### Crystal data

$[\text{Fe}(\text{C}_{13}\text{H}_{12}\text{O})]$	$V = 966.07$ (4) Å <sup>3</sup>
$M_r = 240.08$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 5.77450$ (10) Å	$\mu = 1.52$ mm <sup>-1</sup>
$b = 7.3303$ (2) Å	$T = 150$ (2) K
$c = 22.8596$ (6) Å	$0.38 \times 0.30 \times 0.28$ mm
$\beta = 93.242$ (2)°	

#### Data collection

Nonius KappaCCD diffractometer	13397 measured reflections
Absorption correction: multi-scan ( <i>SORTAV</i> ; Blessing, 1995)	2223 independent reflections
$T_{\min} = 0.579$ , $T_{\max} = 0.660$	2102 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.032$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$	136 parameters
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 1.31$	$\Delta\rho_{\max} = 0.53$ e Å <sup>-3</sup>
2223 reflections	$\Delta\rho_{\min} = -0.38$ e Å <sup>-3</sup>

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON* and *pubCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2366).

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## supporting information

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**[3]Ferrocenophan-1-one****Petr Štěpnička and Ivana Císařová****S1. Comment**

The chemistry of ferrocene has received considerable attention because of its widespread applications ranging from material science to biomedicine. A particularly successful area is undoubtedly catalysis with ferrocene ligands (Štěpnička, 2008). A number of ferrocene ligands has been prepared and studied as ligands for transition metal-mediated reactions.

Among the numerous ligands reported to date, a specific class is constituted by the donors whose cyclopentadienyl (Cp) rings are interconnected with a linking group (*i.e.*, ferrocenophane-type compounds (Heo & Lee, 1999)).

[3]ferrocenophan-1-one (I) represent a convenient entry to such donors (Štěpnička, 2008). Because the crystal structure of I has been reported already in the middle 1960's (Jones *et al.*, 1965), we have redetermined it at 150 K in order to obtain more precise structural information.

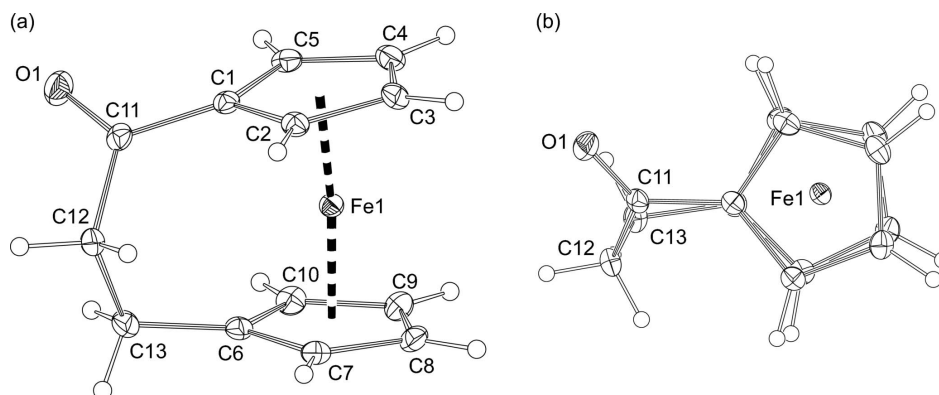
The molecular structure of I (Figure 1) is rather unexceptional as far as interatomic distances and angles concerns. Because of spatial constraints imposed by the 1-oxapropan-1,3-diyl linker, the Cp rings are tilted by 9.39 (18)° and adopt a near-to-eclipsed conformation characterized by the torsion angle C(1)—Cg(1)—Cg(2)—C(6) of -5.6 (2)°, where Cg(1) and Cg(2) are the centroids of the Cp rings C(1–5) and C(6–10), respectively. The iron—Cg distances in I are: Fe1—Cg(1) 1.6399 (14) Å and Fe1—Cg(2) 1.6463 (14) Å. The aliphatic bridge assumes a pseudoenvelope conformation and its C=O bond (C11—O 1.2012 (4) Å) is displaced above its bonding Cp ring, the angle subtended by the C=O vector and the Cp ring being 22.5 (2)°.

**S2. Experimental**

The title compound was synthesized by acylation of ferrocene with acryloyl chloride in the presence of AlCl<sub>3</sub> (Turbitt & Watts, 1972) and characterized by <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} NMR spectra. Orange-red crystals suitable for X-ray diffraction analysis were obtained by liquid-phase diffusion of hexane into a solution of the compound in dichloromethane.

**S3. Refinement**

All H-atoms were included in calculated positions and refined with d(C—H) = 0.93 Å (aromatic) and 0.97 Å (methylene) and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

**Figure 1**

Side (a) and top (b) views of I showing the atom numbering scheme and displacement ellipsoids for the non-H atoms at the 30% probability level.

**[3]ferrocenophan-1-one***Crystal data*[Fe(C<sub>13</sub>H<sub>12</sub>O)] $M_r = 240.08$ Monoclinic,  $P2_1/c$ 

Hall symbol: -P 2ybc

 $a = 5.7745$  (1) Å $b = 7.3303$  (2) Å $c = 22.8596$  (6) Å $\beta = 93.242$  (2)° $V = 966.07$  (4) Å<sup>3</sup> $Z = 4$  $F(000) = 496$  $D_x = 1.651$  Mg m<sup>-3</sup>Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2391 reflections

 $\theta = 0.4$ – $27.5^\circ$  $\mu = 1.52$  mm<sup>-1</sup> $T = 150$  K

Block, orange-red

 $0.38 \times 0.30 \times 0.28$  mm*Data collection*Nonius KappaCCD  
diffractometer

Radiation source: fine-focus sealed tube

Horizontal graphite crystal monochromator

Detector resolution: 9.091 pixels mm<sup>-1</sup> $\omega$  and  $\pi$  scans to fill the Ewald sphere

Absorption correction: multi-scan

(SORTAV; Blessing, 1995)

 $T_{\min} = 0.579$ ,  $T_{\max} = 0.660$ 

13397 measured reflections

2223 independent reflections

2102 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.032$  $\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 1.8^\circ$  $h = -7 \rightarrow 7$  $k = -9 \rightarrow 9$  $l = -29 \rightarrow 29$ *Refinement*Refinement on  $F^2$ 

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.038$  $wR(F^2) = 0.096$  $S = 1.31$ 

2223 reflections

136 parameters

0 restraints

Primary atom site location: structure-invariant  
direct methodsSecondary atom site location: difference Fourier  
mapHydrogen site location: inferred from  
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + 2.5298P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\max} < 0.001$  $\Delta\rho_{\max} = 0.53$  e Å<sup>-3</sup> $\Delta\rho_{\min} = -0.38$  e Å<sup>-3</sup>

*Special details*

**Geometry.** All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement.** Refinement of  $F^2$  against all reflections. The weighted  $R$ -factor  $wR$  and goodness of fit  $S$  are based on  $F^2$ , conventional  $R$ -factors  $R$  are based on  $F$ , with  $F$  set to zero for negative  $F^2$ . The threshold expression of  $F^2 > 2\sigma(F^2)$  is used only for calculating  $R$ -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement.  $R$ -factors based on  $F^2$  are statistically about twice as large as those based on  $F$ , and  $R$ -factors based on all data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.18818 (7)	0.12608 (6)	0.151598 (17)	0.01753 (13)
O1	0.6106 (4)	0.4906 (4)	0.09480 (11)	0.0324 (5)
C1	0.3394 (5)	0.3716 (4)	0.15759 (12)	0.0187 (5)
C2	0.1123 (5)	0.3776 (4)	0.18047 (12)	0.0206 (6)
H2	-0.0117	0.4491	0.1664	0.025*
C3	0.1121 (6)	0.2545 (4)	0.22849 (13)	0.0246 (6)
H3	-0.0127	0.2324	0.2515	0.029*
C4	0.3337 (6)	0.1708 (4)	0.23551 (13)	0.0263 (7)
H4	0.3781	0.0837	0.2635	0.032*
C5	0.4752 (5)	0.2428 (4)	0.19256 (13)	0.0226 (6)
H5	0.6293	0.2122	0.1877	0.027*
C6	0.1582 (5)	0.0986 (4)	0.06275 (12)	0.0212 (6)
C7	-0.0636 (5)	0.0829 (4)	0.08711 (13)	0.0228 (6)
H7	-0.1912	0.1573	0.0782	0.027*
C8	-0.0570 (5)	-0.0656 (4)	0.12733 (14)	0.0242 (6)
H8	-0.1786	-0.1044	0.1493	0.029*
C9	0.1688 (6)	-0.1445 (4)	0.12808 (14)	0.0255 (6)
H9	0.2211	-0.2434	0.1506	0.031*
C10	0.3001 (5)	-0.0440 (4)	0.08799 (13)	0.0240 (6)
H10	0.4530	-0.0675	0.0796	0.029*
C11	0.4118 (5)	0.4452 (4)	0.10113 (13)	0.0209 (6)
C12	0.2345 (6)	0.4393 (4)	0.04970 (13)	0.0238 (6)
H12A	0.2733	0.5291	0.0206	0.029*
H12B	0.0819	0.4680	0.0628	0.029*
C13	0.2334 (6)	0.2466 (4)	0.02244 (13)	0.0250 (6)
H13A	0.1303	0.2467	-0.0126	0.030*
H13B	0.3882	0.2189	0.0107	0.030*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Fe1	0.0204 (2)	0.0150 (2)	0.0171 (2)	0.00020 (17)	-0.00002 (14)	-0.00078 (16)
O1	0.0253 (11)	0.0364 (14)	0.0359 (13)	-0.0055 (10)	0.0037 (9)	0.0061 (11)
C1	0.0220 (13)	0.0145 (12)	0.0194 (13)	-0.0010 (11)	-0.0012 (10)	-0.0025 (11)
C2	0.0256 (14)	0.0172 (13)	0.0189 (13)	0.0012 (12)	0.0003 (11)	-0.0038 (11)
C3	0.0315 (16)	0.0269 (16)	0.0155 (13)	-0.0018 (13)	0.0037 (11)	-0.0037 (12)

C4	0.0352 (17)	0.0240 (15)	0.0189 (14)	-0.0010 (13)	-0.0057 (12)	0.0010 (12)
C5	0.0225 (14)	0.0201 (14)	0.0242 (14)	-0.0009 (12)	-0.0066 (11)	-0.0020 (12)
C6	0.0270 (15)	0.0193 (14)	0.0172 (13)	-0.0014 (12)	-0.0002 (11)	-0.0049 (11)
C7	0.0229 (14)	0.0241 (15)	0.0210 (14)	-0.0026 (12)	-0.0032 (11)	-0.0039 (11)
C8	0.0260 (15)	0.0204 (14)	0.0263 (15)	-0.0068 (12)	0.0016 (12)	-0.0038 (12)
C9	0.0358 (17)	0.0123 (13)	0.0284 (15)	-0.0008 (12)	0.0008 (13)	-0.0013 (12)
C10	0.0268 (15)	0.0198 (14)	0.0258 (15)	0.0018 (12)	0.0051 (12)	-0.0057 (12)
C11	0.0255 (15)	0.0132 (13)	0.0241 (14)	-0.0009 (11)	0.0016 (11)	0.0003 (11)
C12	0.0294 (16)	0.0217 (14)	0.0199 (14)	-0.0035 (12)	-0.0011 (12)	0.0042 (12)
C13	0.0335 (16)	0.0252 (15)	0.0164 (13)	-0.0027 (13)	0.0036 (12)	-0.0028 (12)

*Geometric parameters (Å, °)*

Fe1—C1	2.002 (3)	C4—H4	0.9300
Fe1—C2	2.015 (3)	C5—H5	0.9300
Fe1—C7	2.036 (3)	C6—C10	1.430 (4)
Fe1—C6	2.039 (3)	C6—C7	1.430 (4)
Fe1—C5	2.045 (3)	C6—C13	1.503 (4)
Fe1—C10	2.048 (3)	C7—C8	1.424 (4)
Fe1—C8	2.049 (3)	C7—H7	0.9300
Fe1—C9	2.056 (3)	C8—C9	1.425 (4)
Fe1—C3	2.063 (3)	C8—H8	0.9300
Fe1—C4	2.076 (3)	C9—C10	1.427 (4)
O1—C11	1.212 (4)	C9—H9	0.9300
C1—C2	1.440 (4)	C10—H10	0.9300
C1—C5	1.441 (4)	C11—C12	1.515 (4)
C1—C11	1.480 (4)	C12—C13	1.544 (4)
C2—C3	1.421 (4)	C12—H12A	0.9700
C2—H2	0.9300	C12—H12B	0.9700
C3—C4	1.420 (5)	C13—H13A	0.9700
C3—H3	0.9300	C13—H13B	0.9700
C4—C5	1.415 (4)		
C1—Fe1—C2	42.00 (12)	C4—C3—H3	125.6
C1—Fe1—C7	118.70 (12)	C2—C3—H3	125.6
C2—Fe1—C7	102.70 (12)	Fe1—C3—H3	127.7
C1—Fe1—C6	99.73 (12)	C5—C4—C3	108.2 (3)
C2—Fe1—C6	114.13 (12)	C5—C4—Fe1	68.73 (17)
C7—Fe1—C6	41.09 (12)	C3—C4—Fe1	69.44 (17)
C1—Fe1—C5	41.70 (11)	C5—C4—H4	125.9
C2—Fe1—C5	69.68 (12)	C3—C4—H4	125.9
C7—Fe1—C5	157.51 (13)	Fe1—C4—H4	127.5
C6—Fe1—C5	121.23 (12)	C4—C5—C1	108.2 (3)
C1—Fe1—C10	116.24 (12)	C4—C5—Fe1	71.13 (17)
C2—Fe1—C10	150.81 (12)	C1—C5—Fe1	67.56 (16)
C7—Fe1—C10	68.43 (12)	C4—C5—H5	125.9
C6—Fe1—C10	40.96 (12)	C1—C5—H5	125.9
C5—Fe1—C10	107.44 (13)	Fe1—C5—H5	126.9

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C1—Fe1—C8	158.01 (12)	C10—C6—C7	106.8 (3)
C2—Fe1—C8	123.82 (13)	C10—C6—C13	126.6 (3)
C7—Fe1—C8	40.80 (12)	C7—C6—C13	126.4 (3)
C6—Fe1—C8	69.15 (12)	C10—C6—Fe1	69.86 (16)
C5—Fe1—C8	160.19 (13)	C7—C6—Fe1	69.36 (16)
C10—Fe1—C8	68.35 (13)	C13—C6—Fe1	121.9 (2)
C1—Fe1—C9	154.45 (13)	C8—C7—C6	108.7 (3)
C2—Fe1—C9	163.46 (13)	C8—C7—Fe1	70.09 (17)
C7—Fe1—C9	68.57 (13)	C6—C7—Fe1	69.55 (16)
C6—Fe1—C9	69.15 (12)	C8—C7—H7	125.6
C5—Fe1—C9	123.64 (13)	C6—C7—H7	125.6
C10—Fe1—C9	40.68 (12)	Fe1—C7—H7	126.3
C8—Fe1—C9	40.64 (13)	C7—C8—C9	108.0 (3)
C1—Fe1—C3	69.18 (12)	C7—C8—Fe1	69.11 (17)
C2—Fe1—C3	40.77 (12)	C9—C8—Fe1	69.95 (17)
C7—Fe1—C3	120.66 (13)	C7—C8—H8	126.0
C6—Fe1—C3	152.09 (12)	C9—C8—H8	126.0
C5—Fe1—C3	67.99 (13)	Fe1—C8—H8	126.5
C10—Fe1—C3	166.86 (13)	C8—C9—C10	107.6 (3)
C8—Fe1—C3	111.49 (13)	C8—C9—Fe1	69.41 (17)
C9—Fe1—C3	130.67 (13)	C10—C9—Fe1	69.35 (17)
C1—Fe1—C4	69.06 (12)	C8—C9—H9	126.2
C2—Fe1—C4	68.71 (12)	C10—C9—H9	126.2
C7—Fe1—C4	158.22 (13)	Fe1—C9—H9	126.6
C6—Fe1—C4	160.60 (13)	C9—C10—C6	108.9 (3)
C5—Fe1—C4	40.15 (12)	C9—C10—Fe1	69.97 (17)
C10—Fe1—C4	128.66 (13)	C6—C10—Fe1	69.17 (16)
C8—Fe1—C4	126.57 (13)	C9—C10—H10	125.6
C9—Fe1—C4	114.02 (13)	C6—C10—H10	125.6
C3—Fe1—C4	40.13 (13)	Fe1—C10—H10	126.9
C2—C1—C5	107.3 (3)	O1—C11—C1	121.3 (3)
C2—C1—C11	127.8 (3)	O1—C11—C12	121.2 (3)
C5—C1—C11	123.4 (3)	C1—C11—C12	117.0 (3)
C2—C1—Fe1	69.48 (17)	C11—C12—C13	109.1 (3)
C5—C1—Fe1	70.74 (17)	C11—C12—H12A	109.9
C11—C1—Fe1	114.28 (19)	C13—C12—H12A	109.9
C3—C2—C1	107.6 (3)	C11—C12—H12B	109.9
C3—C2—Fe1	71.43 (18)	C13—C12—H12B	109.9
C1—C2—Fe1	68.51 (17)	H12A—C12—H12B	108.3
C3—C2—H2	126.2	C6—C13—C12	114.1 (2)
C1—C2—H2	126.2	C6—C13—H13A	108.7
Fe1—C2—H2	125.4	C12—C13—H13A	108.7
C4—C3—C2	108.7 (3)	C6—C13—H13B	108.7
C4—C3—Fe1	70.44 (17)	C12—C13—H13B	108.7
C2—C3—Fe1	67.80 (16)	H13A—C13—H13B	107.6

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