Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Bis{2-[2-(4-nitrophenoxy)ethoxy]ethyl} ferrocene-1,1′-dicarboxylate

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The title compound, [Fe($C_{16}H_{16}NO_6$)$_2$], has been synthesized by reacting 1,1′-bis(chlorocarbonyl)ferrocene with 2-[2-(4-nitrophenoxy)ethoxy]ethanol. The metal ion is localized on a twofold axis and the whole molecule is V-shaped, with an angle of 78.63 (2)° between the two cyclopentadienyl arms (non-bonding angle between N atoms of nitro groups and Fe as pivot). The C-1—centroid—centroid—C-1′ pseudo-torsion angle is 45.1 (8)°.

Received 6 April 2006
Accepted 7 May 2006

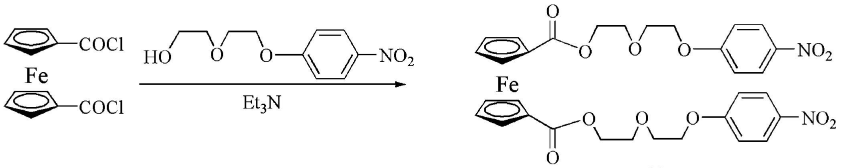
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.082
Data-to-parameter ratio = 13.0

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

Comment

It is well known that ferrocene derivatives bearing crown ether or related macrocyclic units can act as electrochemical probes of the binding of metal cations. However, reports on ferrocene-based receptors having open-chain recognition sites are considerably less common, and only a few examples have been published (Miyaji *et al.*, 2003).



(I)

The title compound, (I), a new open-chain ferrocene receptor, has been prepared (see *Experimental*) and its structure characterized in an X-ray crystallographic study (Fig. 1). The metal centre is located on a twofold axis and the two arms substituting the cyclopentadienyl rings adopt a *cis* conformation ('V' shape). This arrangement is different from the *cis* ('U' shape) and *trans* ('S' and 'Z' shape) conformations previously reported (Khan *et al.*, 1989; Nakamura & Okabe, 2004; Nakamura & Nishikawa, 2005). The two arms in (I) make an angle of 78.63 (2)° [non-bonding angle N1···Fe1···N1^{i}; symmetry code: (i) $-x, y, -z + \frac{1}{2}$]. The C-1—centroid—centroid—C-1′ pseudo-torsion angle is 45.1 (8)°. The dihedral angle between the two benzene ring planes is 29.41 (5)°. Other dimensions are as expected.

Experimental

Dichloromethane was freshly distilled over calcium hydride, and the other solvents were used without further purification. 1,1′-Bis(chlorocarbonyl)ferrocene and 2-[2-(4-nitrophenoxy)ethoxy]ethanol were prepared according to literature methods (Sorensen *et al.*, 2002; Saadioui *et al.*, 1997). Under an inert atmosphere, a solution of 1,1′-bis(chlorocarbonyl)ferrocene (0.10 g, 0.32 mmol) in dichloromethane (10 ml) was added to a solution of 2-[2-(4-nitrophenoxy)ethoxy]ethanol (0.16 g, 0.69 mmol) and triethylamine (0.14 ml, 1 mmol) in dichloromethane (5 ml) with stirring. The resulting

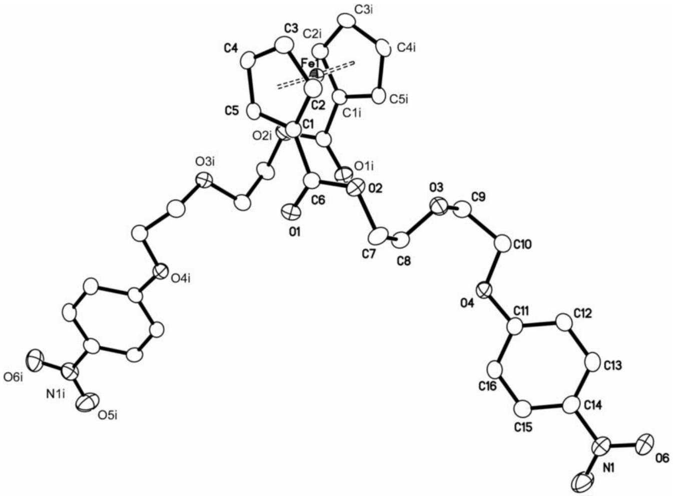


Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. H atoms have been omitted. [Symmetry code: (i) $-x, y, -z + \frac{1}{2}$.]

mixture was refluxed for about 2 d, cooled to 298 K, and washed with brine. The product was purified by thin-layer chromatography on silica gel (THF–CH_2Cl_2 = 1:30) to afford (I) as an orange solid (0.12 g, 53%). Single crystals of (I) were obtained by liquid–liquid diffusion from *n*-hexane and dichloromethane at 298 K.

Crystal data

$[Fe(C_{16}H_{16}NO_6)_2]$
$M_r = 692.45$
Monoclinic, $C2/c$
$a = 17.326$ (3) Å
$b = 8.0669$ (15) Å
$c = 21.673$ (4) Å
$\beta = 92.441$ (2)°
$V = 3026.4$ (10) Å^3
$Z = 4$
$D_x = 1.520$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.57$ mm^{-1}
$T = 298$ (2) K
Block, red
0.32 × 0.28 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.839$, $T_{max} = 0.925$
7611 measured reflections
2775 independent reflections
2537 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.031$
$wR(F^2) = 0.082$
$S = 1.05$
2775 reflections
213 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.8069P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

All H atoms were included in calculated positions and refined as riding on their carrier atoms, with U_{iso}(H) = $1.2U_{eq}$(carrier C). Constrained distances: C—H = 0.93 (aromatic CH) and 0.97 Å (methylene CH_2).

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

We are grateful for financial support from the National Natural Science Foundation of China (No. 20572064) and the State Key Laboratory of Elemento-Organic Chemistry.

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