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An orthorhombic polymorph of (*p*-nitrophenyl)ferrocene

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.042
wR factor = 0.101
Data-to-parameter ratio = 12.7

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

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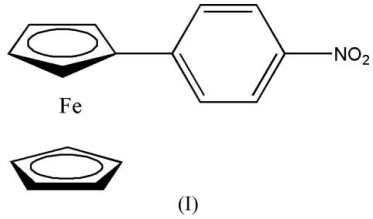
The crystal structure of a new polymorph of (*p*-nitrophenyl)-ferrocene, $[Fe(C_5H_5)(C_{11}H_8NO_2)]$, has been determined at room temperature. The bond lengths and angles in the molecule are normal. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds and van der Waals forces.

Comment

Compounds containing ferrocene building blocks have been widely studied owing to their potential in, for example, catalysis, materials science, molecular devices and hydrometallurgy (Hayashi *et al.*, 1989; Slone *et al.*, 1997). The structure of a monoclinic polymorph (II) of (*p*-nitrophenyl)-ferrocene was originally refined (Roberts *et al.*, 1988) from two-circle diffractometer data without an absorption correction to a rather high $R_{observed}$ value of 0.079. This structure was later redetermined by Gallagher *et al.* (1997) using four-circle diffractometer data, collected at room temperature, giving a significantly more precise structure. In this paper, we report the crystal structure of a new orthorhombic polymorph, (I), of (*p*-nitrophenyl)ferrocene.



(I)

The molecular structure of (I) is shown in Fig. 1. All bond lengths and angles are normal (Allen *et al.*, 1987). Selected torsion angles are given in Table 1. The Fe···*Cg*1 and Fe···*Cg*2 distances are 1.652 (2) and 1.644 (3) Å, respectively, where *Cg*1 and *Cg*2 are the centroids of rings C1–C5 and C6–C10, respectively. The *Cg*1···Fe···*Cg*2 angle is 178.5 (3)°. The dihedral angles formed between the C6–C10 mean plane and planes C1–C5 and C11–C16/N1/O1/O2 are 1.55 (2) and 14.66 (3)°, respectively. In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 2) link molecules into ladders along the [010] direction (Fig. 2).

Experimental

The title compound was synthesized by the reaction of ferrocene (0.01 mol) with a freshly diazotized solution of 4-nitroaniline (0.01 mol) in dilute sulfuric acid (15 ml), followed by chromatography on alumina using dichloromethane and petroleum ether (1:1 *v*/*v*) as

eluent. Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of an ethyl acetate and dichloromethane (1:1 *v*/*v*) solution at room temperature over a period of one week.

Crystal data

$[Fe(C_5H_5)(C_{11}H_8NO_2)]$	$Z = 8$
$M_r = 307.12$	$D_x = 1.549$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo $K\alpha$ radiation
$a = 10.416$ (2) Å	$\mu = 1.14$ mm^{-1}
$b = 7.6525$ (14) Å	$T = 298$ (2) K
$c = 33.053$ (6) Å	Block, red
$V = 2634.6$ (8) Å^3	0.49 × 0.46 × 0.38 mm

Data collection

Bruker SMART CCD area-detector diffractometer	12560 measured reflections
	2314 independent reflections
φ and ω scans	2036 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.031$
	$\theta_{max} = 25.0°$
$T_{min} = 0.586$, $T_{max} = 0.650$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0317P)^2 + 3.1111P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.042$	
$wR(F^2) = 0.101$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.18$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
2314 reflections	$\Delta\rho_{min} = -0.31$ e Å^{-3}
182 parameters	Extinction correction: *SHELXL97*
H-atom parameters constrained	Extinction coefficient: 0.0061 (5)

Table 1
Selected torsion angles (°).

C7—C8—C11—C12	−18.3 (5)	C9—C8—C11—C16	−15.4 (4)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···O2^{i}	0.98	2.54	3.451 (5)	155

Symmetry code: (i) $-x+2, y+\frac{1}{2}, -z+\frac{3}{2}$.

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.98 Å, and refined using a riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1999); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

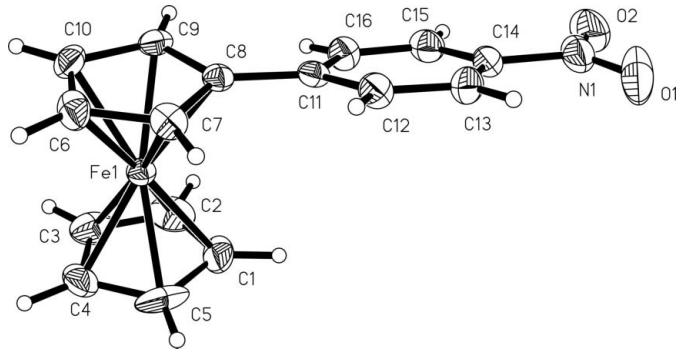


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

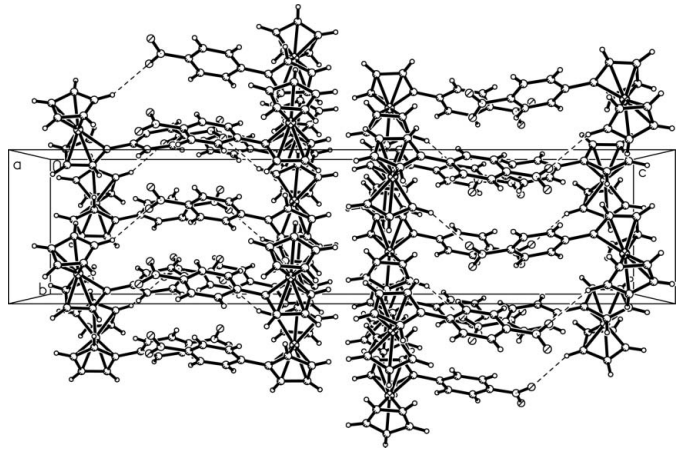


Figure 2
Part of the crystal structure of (I), viewed approximately along the *a* axis. Hydrogen bonds are shown as dashed lines.

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