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1-{(2-Chlorophenyl)[(4-methylphenyl)imino]-methyl}ferrocene

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The title compound, [Fe(C_5H_5)($C_{19}H_{15}$ClN)], a new ketimine-containing ferrocenyl derivative, has been synthesized and characterized structurally. Conjugation between the two benzene rings and the imine group is not observed. In addition, there are no significant intermolecular interactions.

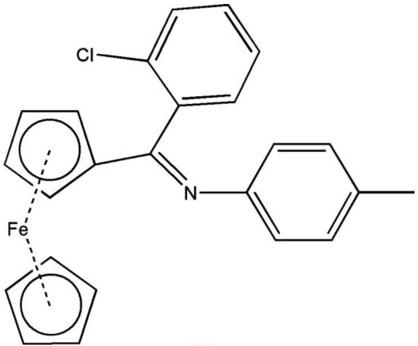
Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.004 Å
R factor = 0.029
wR factor = 0.080
Data-to-parameter ratio = 13.9

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

Comment

The cyclometallation reaction of ferrocenyl derivatives, especially those bearing N-donor ligands, is one of the most advanced research areas of modern organometallic chemistry, because of the wide-ranging application in organic synthesis, such as in the Heck reaction and the Suzuki cross-coupling reaction (Cassol *et al.*, 2005). In the course of our investigation of the cyclometalation of ferrocenyl tertiary amines, we observed that the title compound, (I), was an excellent candidate for ligand orthometallation. Its crystal structure is reported here (Fig. 1).



(I)

In (I), all bond lengths are normal (Allen *et al.*, 1987). The distance between N1 and C11 [1.268 (2) Å] confirms that it corresponds to a C═N double bond. The C2—C1—C11—N1 torsion angle is 175.31 (18)°, which means that the imine group is essentially coplanar with the substituted cyclopentadienyl ring. The relative orientation of the two benzene rings can be ascribed to the steric effect of the *ortho*-chloro substituent [N1—C11—C12—C13 = −78.1 (3)° and C11—N1—C18—C19 = 111.6 (2)°]. There are no significant interactions observed between the molecules.

Experimental

A solution of 2-chlorobenzoylferrocene (1.62 g, 5 mmol), *p*-methylaniline (1.34 g, 12.5 mmol) and *p*-toluenesulfonic acid (50 mg) in toluene (50 ml) was heated under reflux for 10 h with azeotropic removal of water. The mixture was then concentrated on a rotary evaporator and the solid residue was crystallized from ethyl acetate

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and petroleum ether (1:3 *v*/*v*) (333–363 K). A red solid product was obtained. Analysis calculated for $C_{24}H_{20}ClFeN$: C 69.67, H 4.87, N 3.39%; found: C 69.61, H 5.01, N 3.30%.

Crystal data

$[Fe(C_5H_5)(C_{19}H_{15}ClN)]$
$M_r = 413.71$
Triclinic, $P\overline{1}$
$a = 7.624$ (5) Å
$b = 10.436$ (6) Å
$c = 12.791$ (8) Å
$\alpha = 99.096$ (7)°
$\beta = 98.257$ (7)°
$\gamma = 98.981$ (7)°
$V = 977.7$ (10) Å^3
$Z = 2$
$D_x = 1.405$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2965 reflections
$\theta = 2.8$–27.2°
$\mu = 0.92$ mm^{-1}
$T = 293$ (2) K
Block, red
$0.28 \times 0.20 \times 0.14$ mm

Data collection

Bruker APEX-II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.619$, $T_{max} = 0.880$
5343 measured reflections
3397 independent reflections
3037 reflections with $I > 2\sigma(I)$
$R_{int} = 0.012$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 12$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.080$
$S = 1.04$
3397 reflections
244 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0482P)^2 + 0.1948P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C17	1.741 (3)	C1—C11	1.471 (2)
N1—C11	1.268 (2)	C11—C12	1.500 (3)
N1—C18	1.428 (2)		
C11—N1—C18	122.85 (17)	N1—C11—C12	124.55 (16)
N1—C11—C1	118.66 (17)	C1—C11—C12	116.76 (16)
C18—N1—C11—C1	−179.13 (17)	N1—C11—C12—C17	103.3 (2)
C18—N1—C11—C12	−1.0 (3)	N1—C11—C12—C13	−78.1 (3)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and with $U_{iso}(H) = 1.5U_{eq}(C)$, but each group was allowed to rotate freely about its C—C bond. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

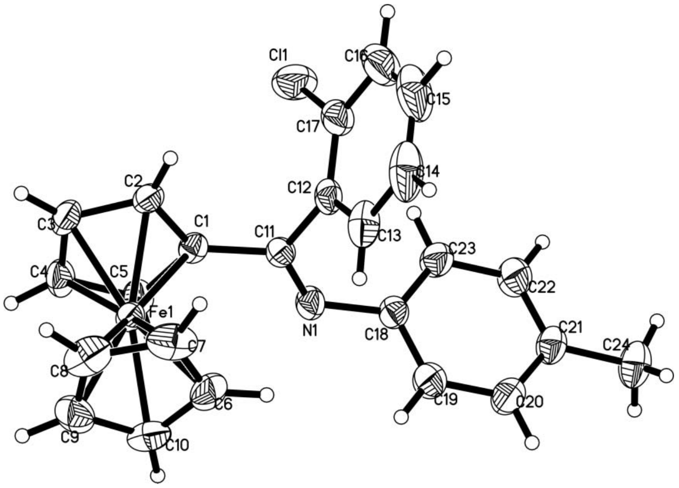

Figure 1
The structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms are shown as small spheres of arbitrary radii.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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, 1997); software used to prepare material for publication: *SHELXTL*.

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