metal-organic papers

1-[(2-Chlorophenyl)(4-chlorophenylimino)methyl]ferrocene

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.042
wR factor = 0.107
Data-to-parameter ratio = 13.8

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

The title compound, [Fe(C$_5$H$_5$)(C$_{18}$H$_{12}$Cl$_2$N)], a new Schiff base containing a ferrocenyl (Fc) group, has been synthesized and characterized structurally. The compound is a *trans* isomer. The C$_{Fc}$—C—N—C linkage has a near-planar geometry, indicating conjugation. There are no obvious intermolecular interactions.

Comment

Some Schiff bases bearing a ferrocenyl group, and their complexes, are excellent non-linear optical materials and liquid crystals (Colbert *et al.*, 1995) because of their strong electron donors and electron-flow bridges. In the course of our investigation of the coordination of Schiff bases with transition metal salts, we observed that the title compound, (I), coordinates readily with Ni^{II} and Cu^{II} salts. The crystal structure of (I) is reported here (Fig. 1).

(I)
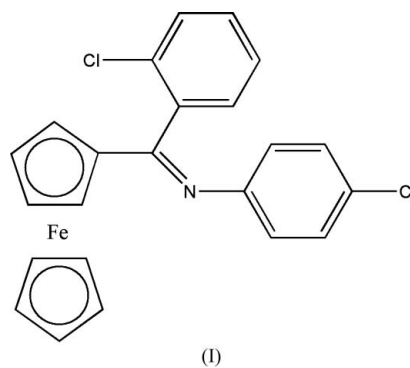
In (I), the N1–C1 bond length [1.259 (4) Å] confirms that it is a C═N double bond. The torsion angle C6—C10—C11—N1 is 175.1 (3)°, indicating that the imino group is nearly in the plane of the substituted cyclopentadienyl ring. There are no obvious intermolecular interactions.

Experimental

[(4-Anilinylimino)2-chlorophenylmethyl]ferrocene (4.14 g, 0.010 mol) was dissolved in anhydrous acetonitrile (100 ml) and added dropwise to a rapidly stirred mixture of tert-butyl nitrite (1.55 g, 0.015 mol) and anhydrous copper(II) chloride (1.36 g, 0.010 mol) in acetonitrile (20 ml). After 10 h, the reaction mixture was poured into 20% aqueous hydrochloric acid (100 ml). The aqueous acetonitrile mixture was extracted twice with 50 ml portions of ether, the combined ether solution was dried over anhydrous magnesium sulfate, and the ether was removed under reduced pressure. The solid residue was crystallized from ethyl acetate and petroleum ether (1:1) (333–363 K). Analysis calculated for C$_{23}$H$_{17}$Cl$_2$FeN: C 63.63, H 3.95, N 3.23%; found: C 63.25, H 4.01, N 3.12%.

Crystal data

[Fe(C₅H₅)(C₁₈H₁₂Cl₂N)]
$M_r = 434.13$
Monoclinic, $P2_1/n$
$a = 8.226 (3)$ Å
$b = 17.288 (6)$ Å
$c = 13.996 (5)$ Å
$\beta = 103.820 (6)°$
$V = 1932.8 (11)$ Å³
$Z = 4$

$D_x = 1.492$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 2747 reflections
$\theta = 2.8–23.1°$
$\mu = 1.06$ mm⁻¹
$T = 294 (2)$ K
Prism, red
$0.22 \times 0.18 \times 0.12$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.720$, $T_{max} = 0.880$
10709 measured reflections

3918 independent reflections
2546 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 26.4°$
$h = -7 \to 10$
$k = -21 \to 21$
$l = -17 \to 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.107$
$S = 1.04$
3918 reflections
284 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 0.4751P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å⁻³
$\Delta\rho_{min} = -0.26$ e Å⁻³

Table 1
Selected geometric parameters (Å, °).

N1—C11	1.259 (4)	C10—C11	1.469 (4)
N1—C12	1.413 (4)	C11—C18	1.510 (4)
C6—C10	1.431 (4)		
C11—N1—C12	123.3 (3)	N1—C11—C18	123.1 (3)
N1—C11—C10	119.2 (3)	C17—C12—N1	120.5 (3)
C12—N1—C11—C10	−174.0 (3)	C11—N1—C12—C13	−99.2 (4)
C12—N1—C11—C18	16.9 (5)	N1—C11—C18—C19	81.1 (4)
C6—C10—C11—N1	175.1 (3)		

The C18–C23 benzene ring is disordered over two sites. The two components were refined as rigid ideal hexagons with C—C = 1.39 Å.
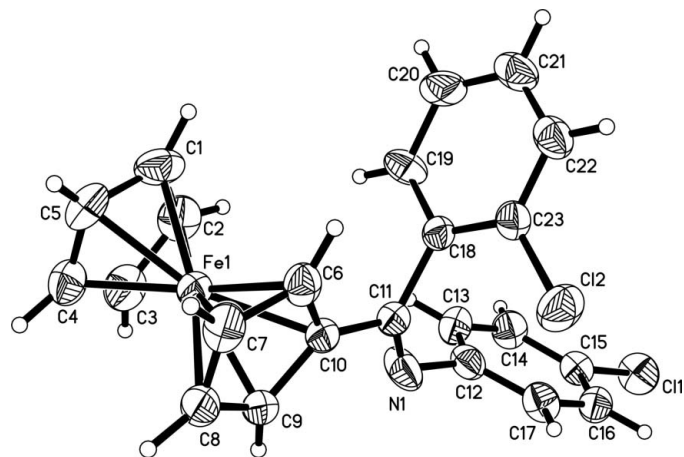

Figure 1
The structure of (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms are shown as small spheres of arbitrary radii. Only one disorder component is shown.

The site occupancy factors are 0.609 (3) and 0.391 (3). All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 or 0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

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*.

References

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