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Key indicators

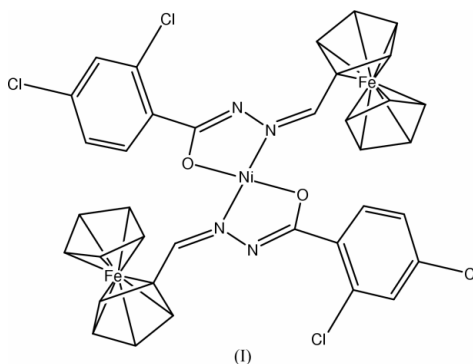
Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.036
 wR factor = 0.094
Data-to-parameter ratio = 13.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis[1-(2,4-dichlorobenzoylhydrazonomethyl)-
ferrocene(1-)]nickel(II)

The title complex, $[\text{Ni}(\text{C}_{18}\text{H}_{13}\text{Cl}_2\text{FeN}_2\text{O})_2]$, results from the reaction of $\text{Ni}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (Ac is acetyl) and 2,4-dichlorobenzoylhydrazine in anhydrous ethanol. The complex molecule is centrosymmetric, with the enolizable O atom and the azomethine N atom of the ligand coordinating to the nickel ion to form a five-membered chelate ring. The N_2O_2 coordinating atoms and the central Ni ion are coplanar.

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Comment

Schiff bases from acylhydrazine and their complexes have strong antitumour and antiviral activities (Ali & Bose, 1984), while the ferrocenyl group can improve these properties (Ali *et al.*, 1973). Some ferrocene derivatives are excellent non-linear optical materials (Long, 1995), because they can act as strong electron donors and contain electron-flow bridges. Due to the possible wide-ranging uses, the structures of these compounds are of interest. In the present work, we report a new crystal structure of an Ni complex of a ferrocene derivative, (I).



In (I), the Ni atom is located at a center of symmetry (Fig. 1). The sum of the interior angles in the chelate ring is $540.0(3)^\circ$, so the five atoms involved are coplanar. The sum of the three bond angles around C12 is $359.8(2)^\circ$, which shows that atom C12 has essentially sp^2 hybridization. The Ni—O and Ni—N bond distances are normal (Table 1). As expected, the C12—O1 bond length [$1.304(3)\text{ \AA}$] lies between those of a C—O single bond and a C=O double bond. The bond lengths N1—C11 [$1.299(3)\text{ \AA}$] and N2—C12 [$1.305(3)\text{ \AA}$] are identical and close to that of typical of C=N (1.30 \AA). These results show that the $-\text{CH}=\text{N}-\text{N}=\text{C}-\text{O}$ fragment of the ligand remains as a conjugated system even after the loss of an H atom from its enolized carbonyl O atom. There are intramolecular non-classical hydrogen bonds (Table 2). There are also intermolecular close contacts between Cl and O atoms; $\text{O1} \cdots \text{Cl2}(x, \frac{1}{2} - y, -\frac{1}{2} + z)$ $3.212(2)\text{ \AA}$ (Fig. 2).

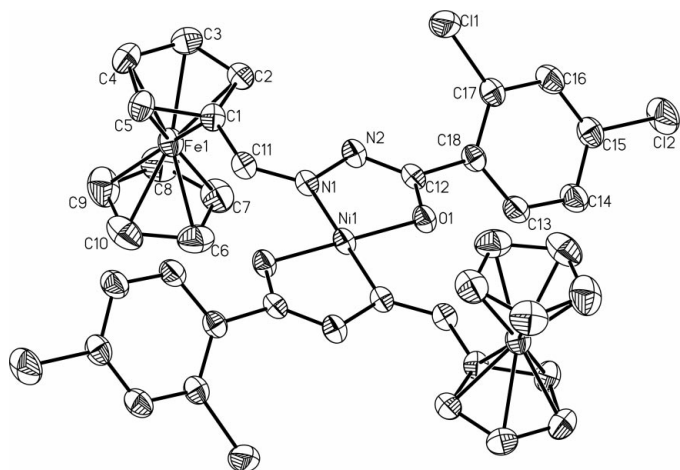


Figure 1
A view of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

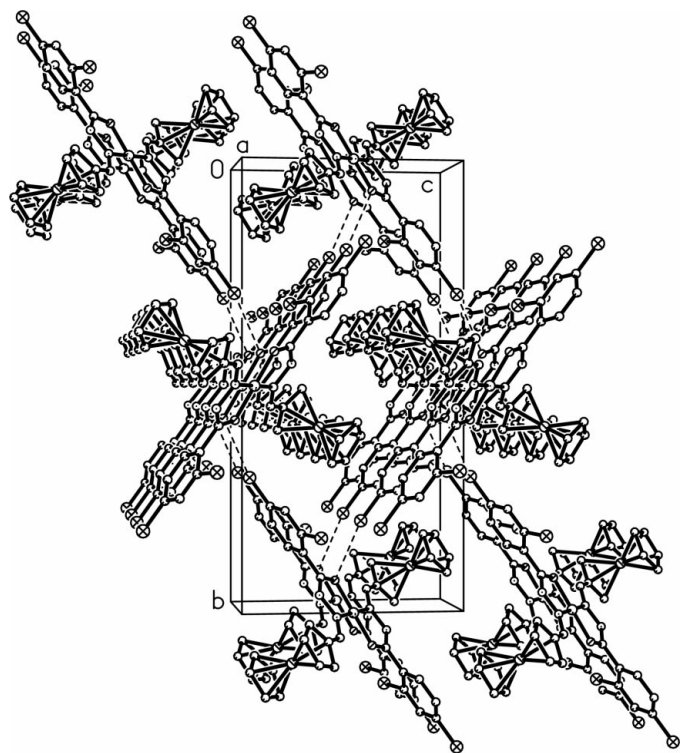


Figure 2
A packing diagram of (I), showing the Cl...O short contacts.

Experimental

Ferrocenecarboxaldehyde was dissolved in anhydrous ethanol and the resulting solution was added dropwise to a solution of 2,4-dichlorobenzoylhydrazine in anhydrous ethanol under reflux, with stirring. A red precipitate appeared immediately and the reaction mixture was allowed to reflux for 2 h with stirring. The mixture was cooled to room temperature and the product collected on a Buchner funnel, washed twice with ethanol and diethyl ether, recrystallized from anhydrous ethanol and dried *in vacuo*. The product obtained was dissolved in anhydrous ethanol, then a solution of Ni(OAc)₂-

4H₂O in anhydrous ethanol was added dropwise to it with stirring at room temperature. The mixture was stirred continuously for 20 min at room temperature and for 6–8 h under reflux. A red solid formed, was filtered off, and the filtrate collected. After four weeks, red crystals of (I) suitable for diffraction analysis had precipitated from the mother liquor.

Crystal data

[Ni(C₁₈H₁₃Cl₂FeN₂O)₂]
M_r = 858.82
 Monoclinic, *P*2₁/*c*
a = 7.032 (1) Å
b = 22.455 (4) Å
c = 11.075 (2) Å
 β = 91.36 (3)°
V = 1748.3 (5) Å³
Z = 2

D_x = 1.631 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 12.1–14.9°
 μ = 1.70 mm⁻¹
T = 293 (2) K
 Block, red
 0.30 × 0.25 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 T_{\min} = 0.599, T_{\max} = 0.716
 5629 measured reflections
 3082 independent reflections
 2869 reflections with $I > 2\sigma(I)$

*R*_{int} = 0.056
 θ_{\max} = 25.0°
 h = -8 → 8
 k = 0 → 26
 l = 0 → 13
 5 standard reflections every 300 reflections
 intensity decay: none

Refinement

Refinement on *F*²
 $R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.094$
 $S = 1.01$
 3082 reflections
 223 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.17 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.76 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Ni1—O1	1.8450 (17)	N1—C11	1.299 (3)
Ni1—N1	1.859 (2)	N1—N2	1.416 (3)
O1—C12	1.304 (3)	N2—C12	1.305 (3)
O1—Ni1—N1	83.73 (8)	C12—N2—N1	106.7 (2)
C12—O1—Ni1	110.27 (16)	O1—C12—N2	124.7 (2)
C11—N1—N2	118.2 (2)	O1—C12—C18	115.5 (2)
N2—N1—Ni1	114.56 (16)	N2—C12—C18	119.6 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C2—H2...N2	0.93	2.53	2.969 (4)	109
C11—H11...O1 ⁱ	0.93	2.42	2.957 (3)	117

Symmetry code: (i) 2 - *x*, 1 - *y*, -*z*.

The positions of all H atoms were fixed geometrically and refined as riding on their parent atoms (C—H 0.93 Å).

Data collection: *CAD-4 SDP/VAX* (Enraf–Nonius, 1989); cell refinement: *CAD-4 SDP/VAX*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 2000); software used to prepare material for publication: *SHELXTL*.

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