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Bis[1-ferrocenyl-3-(2,6-dimethylphenyl)-aminobut-2-en-1-onato]nickel(II)

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Key indicators

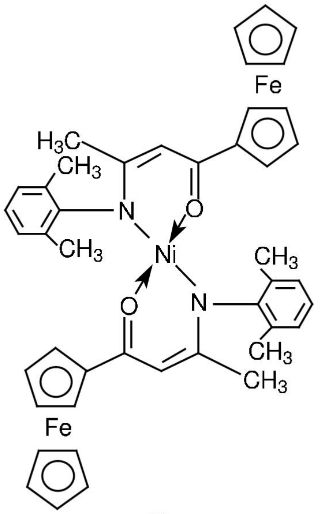
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.091
Data-to-parameter ratio = 15.7

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

In the title compound, [Ni($C_{22}H_{22}$FeNO)$_2$], the Ni^{II} ion lies on an inversion center and has a square planar coordination geometry formed by bidentate enaminone ligands. The Ni—N and Ni—O bond distances are 1.928 (2) and 1.850 (2) Å, respectively.

Comment

Enaminones and their complexes have attracted much attention because of their potential applications in the fields of coordination chemistry and catalysis (Kim *et al.*, 2001). The ferrocenyl enaminone used as a ligand in this work has recently been prepared in our laboratory (Shi *et al.*, 2005). As a part of an ongoing investigation on ferrocenyl enaminone compounds, we have prepared its nickel(II) complex, (I), and determined its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The Ni^{II} cation, located on an inversion center, assumes a square-planar coordination geometry formed by bidentate enaminone monoanions. The Ni—N and Ni—O bond distances (Table 1) are comparable to those in [{$C_5H_5FeC_5H_4$-C(O)CH═C(CH_3)NCH_2}$_2$Ni] [1.851 (4) and 1.842 (3) Å; Shi, Shen *et al.*, 2004] and [CH_3C(O)CH═C(CH_3)N(CH_2)$_3$-NC(CH_3)═C(CH_3CO)N(═O)Ni] [1.910 (4) and 1.832 (3) Å; Kwiatkowski *et al.*, 1990].

The mean C—C bond distance [1.423 (4) Å] in the C_5H_4 ring is essentially identical to that in the C_5H_5 ring [1.412 (5) Å]. In addition, the average Fe—C and Fe$\cdots$*Cg* distances [2.043 (3) and 1.6459 (4) Å, respectively] for the C_5H_4 ring are identical to the average Fe—C and Fe$\cdots$*Cg*

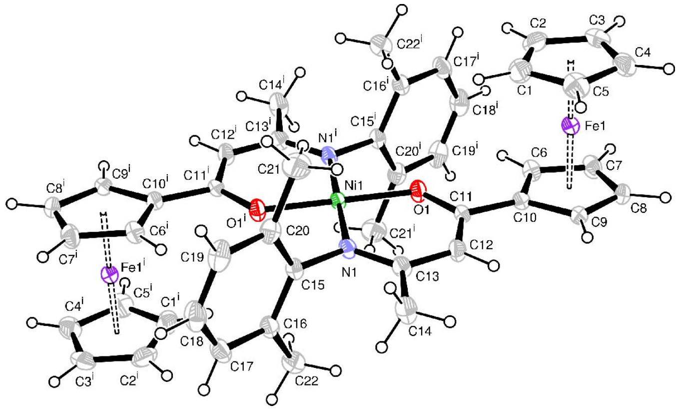


Figure 1

The molecular structure of (I), shown with 30% probability displacement ellipsoids (spheres of arbitrary radius for the H atoms) [symmetry code: (i) $1 - x, 1 - y, 1 - z$].

distances [2.038 (3) and 1.6464 (4) Å, respectively] for the C_5H_5 ring (*Cg* is the centroid of the respective five membered ring). The C_5H_4 ring is nearly parallel to the coordination plane, with a smaller dihedral angle of 6.53 (16)°.

The substituted benzene rings are nearly perpendicular to the coordination plane [dihedral angle = 86.73 (14)°], minimizing the steric repulsions between the methyl groups of dimethylphenyl and enaminone.

Experimental

1-Ferrocenyl-3-(2,6-dimethylphenyl)aminobut-2-en-1-one] (*L*) was synthesized in the manner reported previously (Shi, Yang *et al.*, 2004). $Ni(CH_3COO)_2$, $NaOC_2H_5$ and *L* were mixed in an ethanol solution in 1:2:2 molar ratio. The mixture was refluxed for 24 h to obtain (I). Single crystals of (I) were obtained by recrystallization from CH_2Cl_2/petroleum ether solution; m.p. 485–486 K.

Crystal data

$[Ni(C_{22}H_{22}FeNO)_2]$
$M_r = 803.22$
Triclinic, $P\overline{1}$
$a = 7.165$ (2) Å
$b = 9.709$ (3) Å
$c = 13.999$ (5) Å
$\alpha = 92.878$ (5)°
$\beta = 102.008$ (4)°
$\gamma = 102.174$ (5)°
$V = 926.6$ (5) Å^3
$Z = 1$
$D_x = 1.439$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 931 reflections
$\theta = 3.3$–26.1°
$\mu = 1.32$ mm^{-1}
$T = 293$ K
Block, orange-yellow
0.20 × 0.16 × 0.12 mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.802$, $T_{max} = 0.846$
5299 measured reflections
3697 independent reflections
2983 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 26.3$°
$h = -8 \rightarrow 7$
$k = -12 \rightarrow 9$
$l = -17 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.091$
$S = 1.08$
3697 reflections
235 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 0.3411P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.35$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Ni1—N1	1.928 (2)	C10—C11	1.477 (3)
Ni1—O1	1.850 (2)	C11—C12	1.372 (4)
O1—C11	1.287 (3)	C12—C13	1.397 (4)
N1—C13	1.331 (3)	C13—C14	1.524 (4)
N1—C15	1.444 (3)		
N1—Ni1—O1	93.47 (8)	C10—C11—C12	119.2 (2)
C11—O1—Ni1	128.62 (16)	C11—C12—C13	124.9 (2)
C13—N1—Ni1	124.74 (18)	N1—C13—C12	123.7 (2)
C15—N1—Ni1	120.81 (16)	N1—C13—C14	120.6 (2)
C13—N1—C15	114.5 (2)	C12—C13—C14	115.7 (2)

H atoms were placed in calculated positions (C—H = 0.96 Å for methyl groups, C—H = 0.98 Å for ferrocenyl ring H atoms and C—H = 0.93 Å for all other H atoms). They were refined as riding, with $U_{iso}(H) = 1.5U_{eq}(C)$ (for methyl) or $1.2U_{eq}(C)$. Torsion angles for methyl H atoms were refined from the electron density.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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References

Bruker (1998). *SMART* (version 5.051) and *SAINT* (version 5.01). Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Kim. J., Hwang, J. W., Kim, Y., Lee, M. H., Han,Y. & Do, Y. (2001). *J. Organomet. Chem.* **620**, 1–7.
Kwiatkowski, M., Kwiatkowski, E. & Olechnowicz, A. (1990). *J. Chem. Soc. Dalton Trans.* pp. 2497–2502.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Shi, Y.-C., Shen, W.-B., Yang, H.-M., Song, H.-B. & Hu, X.-Y. (2004). *Polyhedron*, **23**, 749–754.
Shi, Y.-C., Sui, C.-X., Song, H.-B. & Jian, P.-M. (2005). *J. Coord. Chem.* **58**, 363–371.
Shi, Y.-C., Yang, H.-M., Shen, W.-B., Yan, C.-G. & Hu, X.-Y. (2004). *Polyhedron*, **23**, 15–21.