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Key indicators

Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(C-C) = 0.007$ Å
 R factor = 0.060
 wR factor = 0.145
Data-to-parameter ratio = 16.4

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

Bis[*N*-[(*Z*)-4-(3-methyl-5-oxo-1-phenyl-1*H*-pyrazol-4-ylidene)(phenyl)methyl]naphthylamido- κ^2 *N,O*]-iron(II)

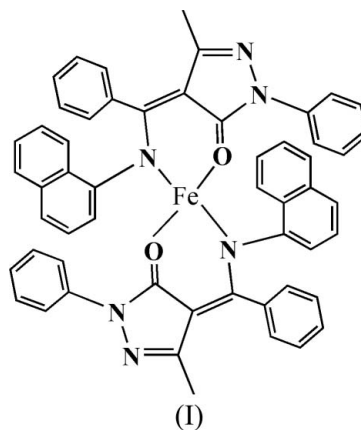
The Fe^{II} atom in the title compound, [Fe(C₂₇H₂₀N₃O)₂], is *N,O*-chelated by two deprotonated 3-methyl-4-[(1-naphthylamino)(phenyl)methylene]-1-phenyl-1*H*-pyrazol-5(4*H*)-one ligands in a severely distorted tetrahedral geometry.

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Comment

The crystal structure of the *N*-[(*Z*)-4-(3-methyl-5-oxo-1-phenyl-1*H*-pyrazol-4-ylidene)(phenyl)methyl]naphthylamide ligand (*L*) in the title compound, (I), has previously been determined (Wang *et al.*, 2003); it also forms a bis-chelated nickel(II) complex (Bao *et al.*, 2006), Ni(C₂₇H₂₀N₃O)₂·(C₂H₆O)₂, in which two solvent molecules of ethanol also coordinate to the metal ion to complete a slightly distorted *cis*-NiN₂O₆ octahedral coordination environment.



In compound (I), the Fe coordination, which involves only the *L* ligands, is intermediate between square planar and tetrahedral and could be described as severely compressed tetrahedral (Fig. 1 and Table 1) with both deprotonated, monoanionic ligands showing *N,O*-chelation to result in stable, six-membered chelate rings (—Fe—O—C—C—C—N—) with the ligand presumably showing electron delocalization. Short intramolecular C—H···O interactions may help to establish the molecular conformation and the crystal packing may be assisted by an intermolecular C—H···N interaction (Table 2).

Experimental

Iron(II) chloride (6.3 mg, 0.05 mmol) dissolved in ethanol (5 ml) was added to a chloroform (5 ml) solution of 3-methyl-4-[(1-naphthylamino)(phenyl)methylene]-1-phenyl-1*H*-pyrazol-5(4*H*)-one (Wang *et al.*, 2003) (40 mg, 0.1 mmol) and triethylamine (14 ml, 0.1 mmol). The brown mixture was filtered and the solution set aside for several

days to give red crystals of (I) in about 75% yield. Analysis calculated for $C_{54}H_{40}FeN_6O_2$: C 75.35, H 4.68, N 9.76%; found: C 75.28, H 4.66, N 9.78%.

Crystal data

$[Fe(C_{27}H_{20}N_3O)_2]$
 $M_r = 860.77$
 Monoclinic, $P2_1/c$
 $a = 13.1106$ (9) Å
 $b = 19.6822$ (13) Å
 $c = 16.8314$ (11) Å
 $\beta = 96.627$ (2)°
 $V = 4314.2$ (5) Å³

$Z = 4$
 $D_x = 1.325$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.40$ mm⁻¹
 $T = 292$ (2) K
 Block, red
 $0.20 \times 0.20 \times 0.10$ mm

Data collection

Bruker SMART CCD
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2001)
 $T_{min} = 0.924$, $T_{max} = 0.961$

31464 measured reflections
 9368 independent reflections
 4102 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.123$
 $\theta_{max} = 27.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.145$
 $S = 0.82$
 9368 reflections
 570 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0538P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta\sigma)_{max} = 0.002$
 $\Delta\rho_{max} = 0.35$ e Å⁻³
 $\Delta\rho_{min} = -0.52$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Fe1—O2	1.907 (2)	Fe1—N1	1.976 (3)
Fe1—O1	1.933 (2)	Fe1—N2	1.982 (3)
O2—Fe1—O1	149.08 (11)	O2—Fe1—N2	98.85 (12)
O2—Fe1—N1	94.56 (12)	O1—Fe1—N2	92.48 (11)
O1—Fe1—N1	97.24 (11)	N1—Fe1—N2	135.56 (13)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C27—H27 \cdots O2	0.93	2.34	2.928 (5)	121
C50—H50 \cdots O1	0.93	2.28	2.924 (5)	126
C48—H48A \cdots N3 ⁱ	0.96	2.59	3.504 (5)	160

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

Because of the poor quality of the crystals, a rather high value of R_{int} was obtained. We tried to obtain better quality crystals, but were unsuccessful. All H atoms were positioned geometrically ($C-H = 0.93$ and 0.96 Å) and refined as riding with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl groups and $x = 1.2$ for all other H atoms.

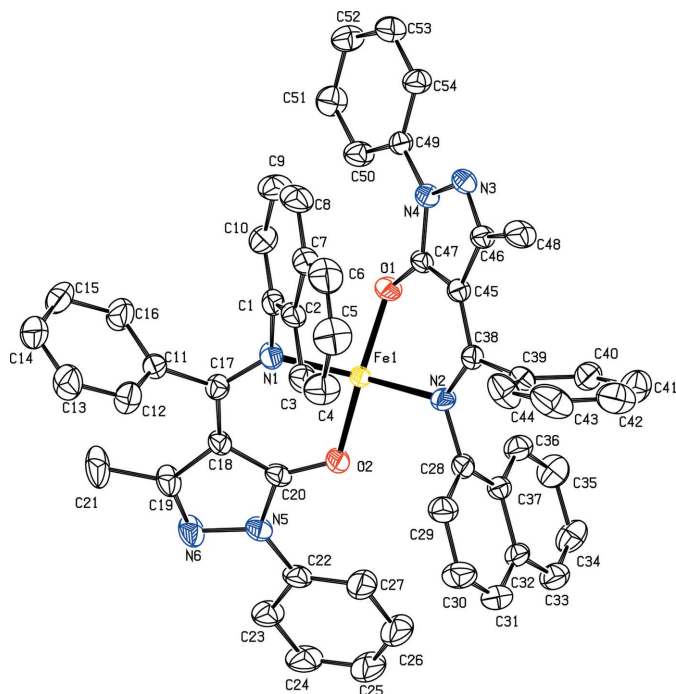


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted for clarity.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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