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Key indicators

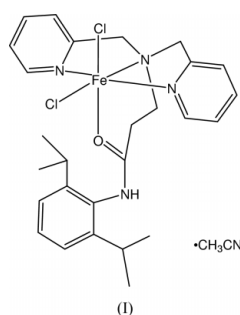
Single-crystal X-ray study
 $T = 91$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.029
 wR factor = 0.075
Data-to-parameter ratio = 26.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Dichloro{*N*-(2,6-diisopropylphenyl)-2-
[(di-2-pyridylmethyl)amino]ethanamido}-
iron(II) acetonitrile solvate

The title compound, $[\text{FeCl}_2(\text{C}_{27}\text{H}_{34}\text{N}_4\text{O})]\cdot\text{CH}_3\text{CN}$, was readily synthesized from the reaction of $\text{FeCl}_2\cdot 4\text{H}_2\text{O}$ and *N*-(2,6-diisopropylphenyl)-2-[(di-2-pyridylmethyl)amino]ethanamide in THF. The iron(II) metal center is in an octahedral geometry. There is an intermolecular hydrogen bond between the amide H and one chlorine of a neighboring complex.

Received 5 June 2003
Accepted 16 June 2003
Online 24 June 2003

Comment

Iron coordination complexes have been studied as small molecule analogs of metalloproteins (Marlin *et al.*, 2002; Rowland *et al.*, 2001; Sams *et al.*, 2001) and recently have gained considerable interest as catalysts for atom-transfer reactions and controlled 'living' radical polymerizations (Kamigaito *et al.*, 2001; Matyjaszewski & Xia, 2001; Patten & Matyjaszewski, 1999). During the course of our studies on using monoanionic tetradentate ligands to prepare copper(I)-based atom transfer radical polymerization catalysts, we investigated the complexation of a bis(picoly)amine-based ligand to iron(II) (Kobayashi *et al.*, 1998; Niklas *et al.*, 2000, 2001). The addition of $\text{FeCl}_2\cdot 4\text{H}_2\text{O}$ to a THF solution of *N*-(2,6-diisopropylphenyl)-2-[(di-2-pyridylmethyl)amino]ethanamide formed the complex. An IR spectrum showed bands corresponding to the presence of the amide N—H group (3410 cm^{-1}) and the amide carbonyl group (1607 cm^{-1}).



The structure of the complex is monomeric with one molecule of cocrystallized acetonitrile, and the iron(II) center is in near octahedral geometry, taking into account the constraints of the tethers between the N atoms. The amide group is hydrogen bonded to a Cl atom of the neighboring complex to form chains of complexes throughout the crystal.

Experimental

All chemicals were purchased from Acros and used without further purification. Acetonitrile was dried using appropriate procedures,

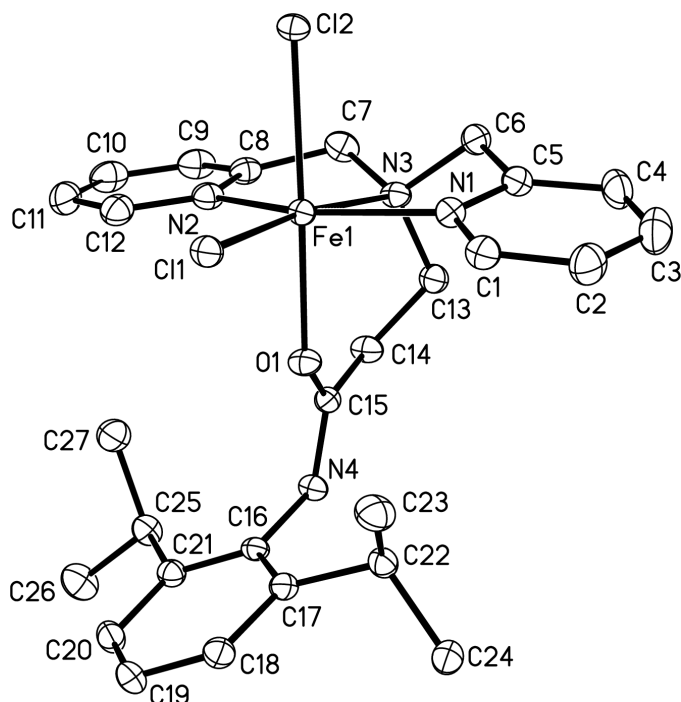


Figure 1
View of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

and the recrystallization was performed using standard Schlenk techniques. The synthesis of the ligand *N*-(2,6-diisopropylphenyl)-2-[(di-2-pyridylmethyl)amino]ethanamide will be described in a subsequent publication (Troeltzsch *et al.*, 2003). The ligand (1.02 g, 2.37 mmol) was dissolved in 25 ml of THF, and then 0.472 g (2.38 mmol) of $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ was added. The solution was stirred and then concentrated to dryness. The solid was dissolved in boiling, dry acetonitrile, and upon cooling amber needles formed, suitable for X-ray analysis [m.p. 446 K (decomposition)]. Elemental composition, calculated: C 58.21, H 6.23, N 11.70%; found: C 57.09, H 6.14, N 11.69%. IR (Thin film, NaCl plates) ν (cm^{-1}): 3410 (*broad*), 1607 (*s*), 1563 (*m*).

Crystal data

$[\text{FeCl}_2(\text{C}_{27}\text{H}_{34}\text{N}_4\text{O})] \cdot \text{CH}_3\text{CN}$
 $M_r = 598.39$
Orthorhombic, $P2_12_12_1$
 $a = 13.1579$ (4) Å
 $b = 14.2791$ (5) Å
 $c = 15.7210$ (5) Å
 $V = 2953.71$ (17) Å³
 $Z = 4$
 $D_x = 1.346$ Mg m⁻³

Data collection

Bruker SMART 1000
diffractometer
 ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2000)
 $T_{\min} = 0.829$, $T_{\max} = 0.857$
27 291 measured reflections

Mo $K\alpha$ radiation
Cell parameters from 7272
reflections
 $\theta = 2.5\text{--}31.4^\circ$
 $\mu = 0.72$ mm⁻¹
 $T = 91$ (2) K
Block, brown
 $0.27 \times 0.27 \times 0.22$ mm
9258 independent reflections
8527 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$
 $\theta_{\text{max}} = 31.5^\circ$
 $h = -19 \rightarrow 19$
 $k = -20 \rightarrow 20$
 $l = -22 \rightarrow 22$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.075$
 $S = 1.07$
9258 reflections
348 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0464P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.22$ e Å⁻³
Absolute structure: Flack (1983);
3928 Friedel pairs
Flack parameter = -0.015 (8)

Table 1

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>
N4—H4A···Cl2 ⁱ	0.88	2.32	3.1951 (13)	173

Symmetry code: (i) $\frac{3}{2} - x, 2 - y, z - \frac{1}{2}$.

H atoms were placed in idealized positions and refined using a model whereby the H atom was riding on the parent C atom with an U_{iso} value equal to 1.2 (1.5 for methyl H) times the U_{eq} value of the parent atom.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL97*.

The Bruker SMART 1000 diffractometer was funded in part by NSF Instrumentation grant CHE-9808259 to the University of California, Davis. Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research (Grant #35150-AC7).

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