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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$

R factor = 0.032

wR factor = 0.083

Data-to-parameter ratio = 14.1

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Dichlorobis(2-formylpyridineanil)iron(II)
acetonitrile solvate

The structure of the title compound {systematic name: dichlorobis[2-(phenyliminomethyl)pyridine]iron(II) acetonitrile solvate}, *cis*-(2-C₅H₄NCH=NPh)₂FeCl₂·C₂H₃N, consists of an iron centre bound by two bidentate pyridylimine ligands and two *cis*-disposed chloride ligands.

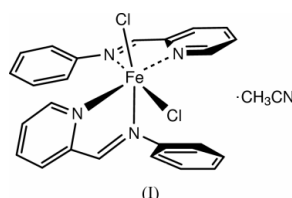
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Comment

The synthesis of divalent late transition metal complexes supported by chelating multidentate ligands containing both the imino and pyridine moieties has been the subject of considerable recent research activity due, in part, to their connection to olefin polymerization catalysis (Gibson & Spitzmesser, 2003). In particular, the application of group 10 metals supported by iminopyridine ligands (Laine *et al.*, 1999) and group 8 metals by bis(imino)pyridine ligands (Britovsek *et al.*, 1998) has allowed access to highly active ethylene polymerization catalysts capable of producing a wide variety of polymeric materials.



Here, we report the synthesis and the crystal structure of *cis*-(2-C₅H₄NCH=NPh)₂FeCl₂·C₂H₃N, (I). The positive FAB mass spectrum exhibits a molecular ion peak along with a fragmentation peak corresponding to the loss of chloride from the molecular ion.

The X-ray analysis of (I) shows a distorted octahedral complex in which the Fe atom is bound to two *cis*-coordinated *N,N'*-chelating ligands [the pyridyl N atoms being *trans*; angle = 159.60 (6)°] and two chlorides. The Fe–N(pyridine) distances [2.1744 (15) and 2.1799 (16) Å] are similar and notably shorter than the Fe–N(imine) distances [Fe1–N4 = 2.2474 (16) Å and Fe1–N2 = 2.3144 (14) Å]. The explanation for the disparity in the Fe–N(imine) distances is uncertain but it is worthy of note that the corresponding *trans* Fe–Cl distances [Fe1–Cl1 = 2.3796 (6) Å and Fe1–Cl2 = 2.4120 (5) Å] are also markedly different, with the shorter Fe–N(imine) distance (Fe1–N4) being accompanied by a longer *trans* Fe–Cl distance (Fe1–Cl1) and *vice versa*. The two imine linkages, C6–N2 and C18–N4, are both short, reflecting a retention of their double-bond character. The

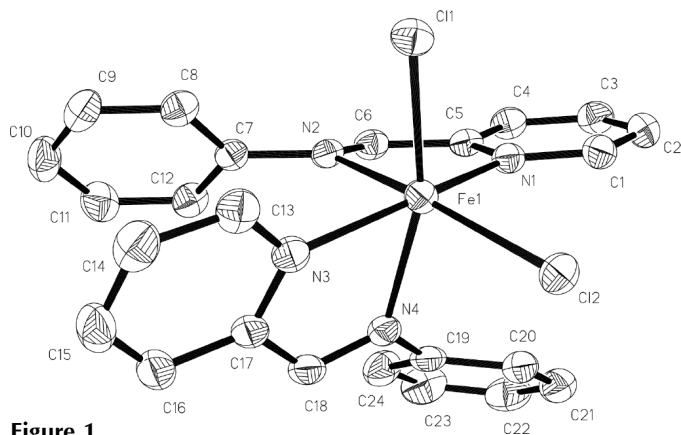


Figure 1
The molecular structure of (I), showing the atom-numbering scheme and 50% displacement ellipsoids. H atoms are drawn as spheres of arbitrary radius. H atoms and the acetonitrile solvent molecule have been omitted for clarity.

phenyl rings of each ligand are similarly oriented with respect to the pyridyl-imine N–C–C–N planes [43.90 (9) and 46.46 (10)°]. There are no intermolecular packing interactions of note.

Experimental

Under an atmosphere of nitrogen, a suspension of iron(II) chloride (0.222 g, 1.75 mmol) in tetrahydrofuran (30 ml) was cooled to 195 K and 2-formylpyridineanil (0.600 g, 3.30 mmol) was added to this solution *via* a solids addition tube. The dark green solution mixture was allowed to reach room temperature and then stirred overnight. The volatiles were removed under reduced pressure and the remaining solid was extracted into acetonitrile (30 ml), stirred for 3 h and then filtered. The solution was layered with hexane (60 ml) and left to crystallize at 248 K. After 3 d, dark green crystals suitable for single-crystal X-ray diffraction analysis were obtained (0.200 g, 24% yield).

Crystal data

[FeCl₂(C₁₂H₁₀N₂)₂]₂·C₂H₃N
M_r = 532.24
 Monoclinic, *P*2₁/*c*
a = 12.7838 (7) Å
b = 14.8194 (8) Å
c = 13.8905 (8) Å
 β = 109.677 (10)°
V = 2477.9 (2) Å³
Z = 4

D_x = 1.427 Mg m⁻³
 Mo *K* α radiation
 Cell parameters from 7807 reflections
 θ = 2.2–28.4°
 μ = 0.85 mm⁻¹
T = 150 (2) K
 Plate, black
 0.39 × 0.26 × 0.09 mm

Data collection

Bruker CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 T_{\min} = 0.715, T_{\max} = 0.928
 15046 measured reflections

4348 independent reflections
 3811 reflections with $I > 2\sigma(I)$
 R_{int} = 0.027
 θ_{max} = 25.0°
 h = -15 → 15
 k = -17 → 17
 l = -16 → 16

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)]$ = 0.033
 $wR(F^2)$ = 0.083
 S = 1.04
 4348 reflections
 308 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 0.2129P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}}$ = 0.002
 $\Delta\rho_{\text{max}}$ = 0.47 e Å⁻³
 $\Delta\rho_{\text{min}}$ = -0.24 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Fe1–N1	2.1744 (15)	Fe1–Cl1	2.3796 (6)
Fe1–N3	2.1799 (16)	Fe1–Cl2	2.4120 (5)
Fe1–N4	2.2474 (16)	N2–C6	1.270 (2)
Fe1–N2	2.3144 (14)	N4–C18	1.283 (3)
N3–Fe1–N4	74.08 (6)	Cl1–Fe1–Cl2	100.504 (19)
N1–Fe1–N2	73.10 (5)		

All H atoms were refined using a riding model, with bond lengths of 0.98 Å (solvent molecule) and 0.95 Å (all others). For methyl H atoms, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{parent})$; for all other H atoms, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$.

Data collection: SMART (Bruker, 1997); cell refinement: SMART; data reduction: SAINT (Bruker, 1999); program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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