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Tris(5,5′-dimethyl-2,2′-bipyridyl-κ^2N,N')iron(II) bis(perchlorate) 5,5′-dimethyl-2,2′-bipyridyl solvate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.078
wR factor = 0.238
Data-to-parameter ratio = 16.9

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

The low-spin iron(II) ion in the title compound, $[Fe(C_{12}H_{12}N_2)_3](ClO_4)_2\cdot C_{12}H_{12}N_2$, possesses an octahedral geometry formed by three bidentate 5,5′-dimethyl-2,2′-bipyridyl ligands. An uncoordinated ligand and two perchlorate anions are also present in the crystal structure.

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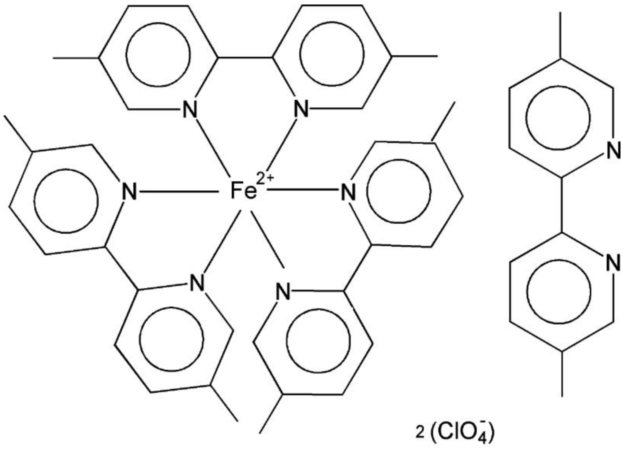
Comment

Only a limited number of X-ray crystal structures with the ligand 5,5′-dimethyl-2,2′-bipyridyl (abbreviated as dmbpy) has been published (Catalan *et al.*, 1995; Kooijman *et al.*, 2002; van Albada *et al.*, 2004).



(I)

The low-spin iron(II) ion in the title compound, (I), possesses an octahedral geometry, with an FeN_6 chromophore, formed by three bidentate dmbpy ligands, with Fe—N distances between 1.972 (4) and 1.995 (3) Å. In addition, a non-coordinated ligand and two non-coordinating perchlorate anions, of which one is disordered, are present in the asymmetric unit. The distances and angles of the iron(II) chromophore resemble those found in the compound [Fe(2,2′-bipyridine)$_3$](ClO$_4$)$_2$ (Batten *et al.*, 2000).

Experimental

All chemicals were obtained commercially and used without further purification. For the synthesis of the coordination compound, $Fe(ClO_4)_2$ (0.3 mmol) and dmbpy (1.0 mmol) were each dissolved in ethanol and were mixed carefully and left standing in a closed bottle. After a few weeks, very thin long red plate-like crystals formed (yield: 86%). Elemental analysis (%) found (calculated): C 58.2 (58.1), H 5.0 (4.9), N 11.5 (11.3).

Crystal data

$[Fe(C_{12}H_{12}N_2)_3](ClO_4)_2\cdot C_{12}H_{12}N_2$
$M_r = 991.69$
Triclinic, $P\overline{1}$
$a = 11.670$ (2) Å
$b = 13.466$ (3) Å
$c = 14.792$ (3) Å
$\alpha = 94.77$ (3)°
$\beta = 90.82$ (3)°
$\gamma = 96.26$ (3)°
$V = 2302.0$ (8) Å^3
$Z = 2$
$D_x = 1.431$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 32869 reflections
$\theta = 2.7$–27.5°
$\mu = 0.51$ mm^{-1}
$T = 173$ (2) K
Plate, red
0.35 × 0.10 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.842$, $T_{\max} = 0.990$
32869 measured reflections
10528 independent reflections
6461 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.108$
$\theta_{\max} = 27.5°$
$h = -15 \rightarrow 15$
$k = -17 \rightarrow 17$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.238$
$S = 1.03$
10528 reflections
622 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.13P)^2 + 0.9561P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.98$ e Å^{-3}
$\Delta\rho_{\min} = -0.94$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N51	1.972 (4)	Fe1—N41	1.987 (4)
Fe1—N61	1.973 (3)	Fe1—N11	1.991 (3)
Fe1—N21	1.983 (3)	Fe1—N31	1.995 (3)

N51—Fe1—N61	81.75 (14)	N21—Fe1—N11	81.42 (13)
N51—Fe1—N21	92.40 (14)	N41—Fe1—N11	95.48 (14)
N61—Fe1—N21	172.76 (14)	N51—Fe1—N31	94.61 (14)
N51—Fe1—N41	174.93 (13)	N61—Fe1—N31	90.17 (13)
N61—Fe1—N41	94.54 (14)	N21—Fe1—N31	94.56 (13)
N21—Fe1—N41	91.55 (14)	N41—Fe1—N31	81.90 (14)
N51—Fe1—N11	88.26 (14)	N11—Fe1—N31	175.17 (14)
N61—Fe1—N11	94.09 (13)		

One of the two perchlorate anions was found to be disordered over two sites; the two components were refined as rigid groups with population parameter 0.5. All non-H atoms were refined anisotropically. The H atoms were placed in calculated positions and refined with a riding model, with $U_{\rm iso} = 1.2U_{\rm eq}$(C) and C—H = 0.95 Å for aromatic H atoms, and $U_{\rm iso} = 1.5U_{\rm eq}$(C) and C—H = 0.98 Å for methyl H atoms. The crystals showed weak diffracting ability, which could account for the rather high $R_{\rm int}$ value.

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *COLLECT* and *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1990); software used to prepare material for publication: *SHELXTL*.

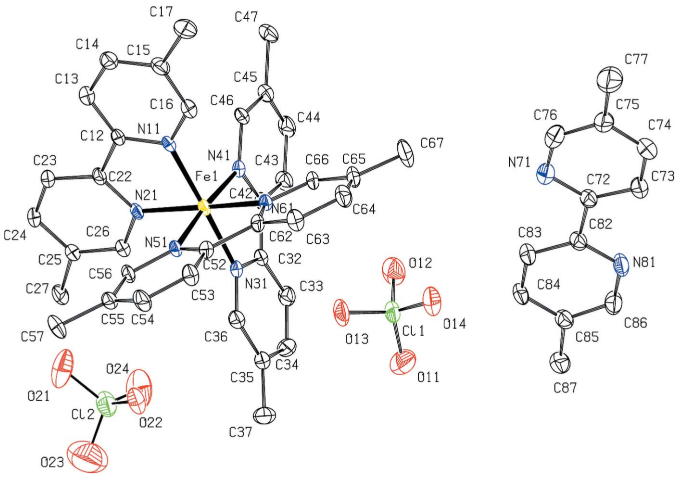


Figure 1
A view of the title structure with the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity. Only one component of the disordered perchlorate anion is shown.

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