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Key indicators

Single-crystal X-ray study
T = 293 K
 Mean σ (C–C) = 0.009 Å
R factor = 0.061
wR factor = 0.184
 Data-to-parameter ratio = 18.9

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

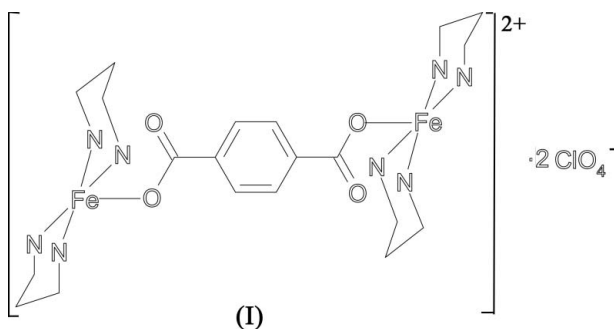
μ -Terephthalato- $\kappa^2O^1:O^4$ -bis[bis(propane-1,3-diamine- κ^2N,N')iron(II)] bis(perchlorate)

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The dinuclear title complex, $[Fe_2(C_8H_4O_4)(C_3H_{10}N_2)_4](ClO_4)_2$, is centrosymmetric about the central aromatic ring. The Fe centre is five-coordinated square pyramidal, being coordinated by four N atoms from two propane-1,3-diamine ligands and an O atom from a monodenate carboxylate residue.

Comment

Ferrous solutions are normally unstable in air and are easily oxidized to produce ferric compounds, especially in neutral or alkaline solutions. The title ferrous complex, (I), was formed and isolated from a mildly alkaline solution of ammonia and methanol.



The complex (Fig. 1) is disposed about a centre of inversion and features two $Fe[NH_2(CH_2)_3NH_2]_2$ entities bridged by a terephthalate anion; the charge-balance in the structure is provided by two perchlorate anions. The Fe–N bond lengths are in the range 2.024 (5)–2.047 (5) Å (Table 1). The associated N–C bond distances are experimentally equivalent, precluding imine functionality in any of the propane-1,3-diamine ligands. The Fe–O1 bond distance is 2.267 (4) Å, with the O1 atom occupying the apical position in the distorted square-pyramidal geometry about the Fe centre; the Fe atom lies 0.1131 (3) Å out of the basal plane defined by the four N atoms. The bond angles at Fe1 (Table 1) are consistent with a regular geometry.

Experimental

Terephthalic acid (0.5 mmol, 83 mg) and $FeSO_4 \cdot 7H_2O$ (1 mmol, 278 mg) were suspended in a mixed solvent of 30% aqueous ammonia and methanol (1:1 v/v, 10 ml). To this solution was added, with stirring, an acetonitrile solution (5 ml) of $NaClO_4$ (1 mmol, 124 mg) and propane-1,3-diamine (2 mmol, 144 mg). The suspension was stirred for 20 min and filtered. After keeping the filtrate in air for 10 d, large black block-shaped crystals of (I) formed at the bottom of

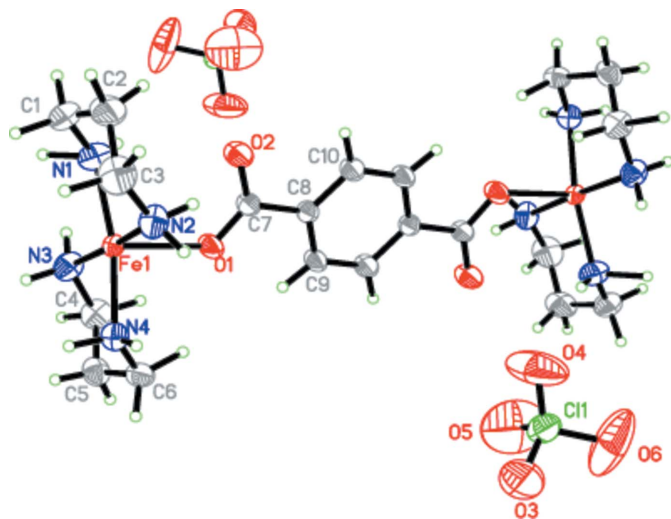


Figure 1

The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. The unlabelled atoms are related by the symmetry code $(1 - x, 1 - y, -z)$.

the vessel. The crystals were isolated, washed three times with water and dried in a vacuum desiccator over CaCl_2 (yield 81.2%). Analysis found: C 31.04, H 5.77, N 14.26%; calculated for $\text{C}_{20}\text{H}_{44}\text{Cl}_2\text{Fe}_2\text{N}_8\text{O}_{12}$: C 31.15, H 5.75, N 14.53%.

Crystal data

$[\text{Fe}_2(\text{C}_8\text{H}_4\text{O}_4)(\text{C}_3\text{H}_{10}\text{N}_2)_4](\text{ClO}_4)_2$
 $M_r = 771.23$
 Monoclinic, $P2_1/n$
 $a = 10.165$ (2) Å
 $b = 10.143$ (2) Å
 $c = 16.012$ (3) Å
 $\beta = 93.05$ (3)°
 $V = 1648.6$ (6) Å³

$Z = 2$
 $D_x = 1.554$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 1.11$ mm⁻¹
 $T = 293$ (2) K
 Block, black
 $0.34 \times 0.28 \times 0.22$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.696$, $T_{\max} = 0.784$

3980 measured reflections
 3781 independent reflections
 2463 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.184$
 $S = 1.03$
 3781 reflections
 200 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0957P)^2 + 1.6922P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.65$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.58$ e Å⁻³
 Extinction correction: SHELXL97
 Extinction coefficient: 0.024 (3)

Table 1

Selected geometric parameters (Å, °).

Fe1—O1	2.267 (4)	N1—C1	1.479 (8)
Fe1—N1	2.030 (4)	N2—C3	1.472 (9)
Fe1—N2	2.024 (5)	N3—C4	1.493 (7)
Fe1—N3	2.047 (5)	N4—C6	1.483 (7)
Fe1—N4	2.024 (5)		
O1—Fe1—N1	90.57 (18)	N2—Fe1—N3	90.7 (2)
O1—Fe1—N2	97.4 (2)	N2—Fe1—N4	168.6 (2)
O1—Fe1—N3	94.04 (18)	N3—Fe1—N4	88.4 (2)
O1—Fe1—N4	94.06 (15)	Fe1—N1—C1	119.4 (4)
N1—Fe1—N2	87.5 (2)	Fe1—N2—C3	121.2 (5)
N1—Fe1—N3	175.23 (19)	Fe1—N3—C4	116.6 (4)
N1—Fe1—N4	92.5 (2)	Fe1—N4—C6	116.2 (3)

H atoms were positioned geometrically and constrained to ride on their parent atoms at C—H distances of 0.93–0.97 Å and N—H = 0.90 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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References

- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997a). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
 Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.