

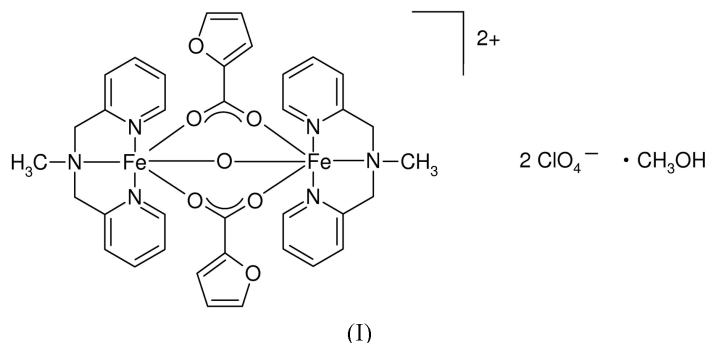
Anne Nielsen, Andrew D. Bond\*  
and Christine J. McKenzieUniversity of Southern Denmark, Department of  
Chemistry, Campusvej 55, 5230 Odense,  
Denmark

Correspondence e-mail: adb@chem.sdu.dk

## Key indicators

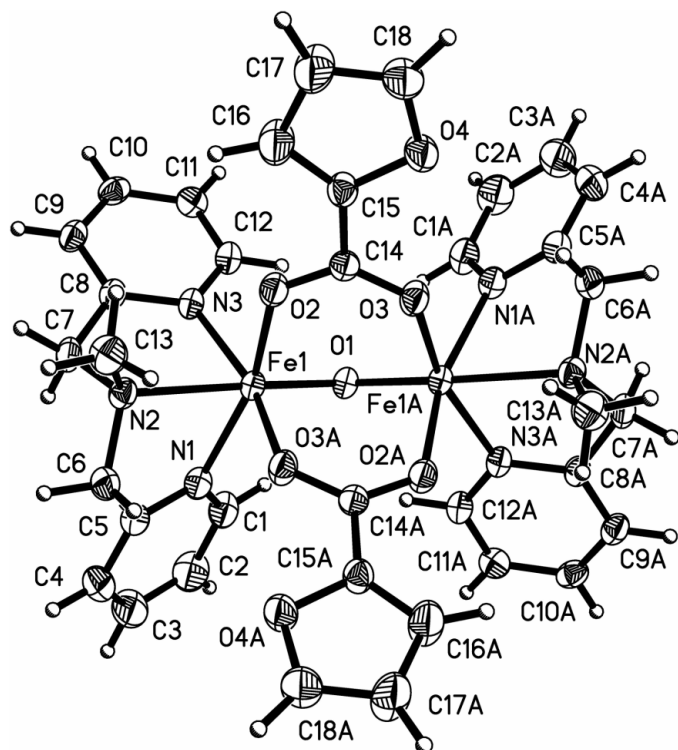
Single-crystal X-ray study  
 $T = 180$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å  
Disorder in solvent or counterion  
 $R$  factor = 0.043  
 $wR$  factor = 0.120  
Data-to-parameter ratio = 17.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>. $\mu$ -Oxo-bis( $\mu$ -2-furancarboxylato)bis{[*N*-methyl-*N,N*-bis(2-pyridylmethyl)amine]iron(III)}  
bis(perchlorate) methanol solvateIn the title compound,  $[\text{Fe}_2\text{O}(\text{C}_5\text{H}_3\text{O}_3)_2(\text{C}_{13}\text{H}_{15}\text{N}_3)_2](\text{ClO}_4)_2 \cdot \text{CH}_3\text{OH}$ , at 180 K, the complex lies on a twofold axis, as does the C atom of the disordered methanol molecule.Received 27 October 2004  
Accepted 22 November 2004  
Online 27 November 2004

## Comment

 $\mu$ -Oxo-bridged Fe(III)–Fe(III) complexes with neutral *N*-donor capping ligands commonly also contain oxo-anion groups that bridge between the Fe atoms. The title complex, (I), is chemically closely related to three previously reported Fe(III)–( $\mu$ -O)–Fe(III) complexes containing the *N*-methyl-*N,N*-bis(2-pyridylmethyl)amine (medpa) ligand: the di-ethoxylato-bridged analogue crystallizes as both a monohydrate (Nishino *et al.*, 1999) and a dihydrate (Jensen *et al.*, 1997), the di-phenylato-bridged analogue crystallizes as a hemihydrate (Dick *et al.*, 1997), and the di-(2,3,5-triiodo-phenylato)-bridged analogue crystallizes without solvent inclusion (Dick *et al.*, 1997).In the crystal structure of (I), the Fe(III)–Fe(III) complexes lie on twofold axes passing through the  $\mu$ -O atom and the midpoint of the Fe...Fe vector. The two furancarboxylato moieties form a dihedral angle of  $101.8(1)^\circ$  between their least-squares planes, and the methanol moiety lies between them with its C atom also on the twofold axis (Fig. 2). Similar sites are occupied by water molecules in the di-phenylato analogue and in the monohydrate of the di-ethoxylato-bridged analogue, and by perchlorate anions in the other two cited examples.

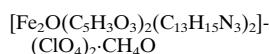
## Experimental

2-Furancarboxylic acid (0.016 g, 0.144 mmol) in 0.5 ml methanol and medpa( $\text{HClO}_4$ )<sub>2</sub> (0.059 g, 0.143 mmol; medpa = *N*-methyl-*N,N*-bis(2-pyridylmethyl)amine) in 5 ml hot methanol were added dropwise to a solution of  $\text{Fe}(\text{ClO}_4)_3 \cdot 9\text{H}_2\text{O}$  (0.074 g, 0.144 mmol) in 0.25 ml methanol. The yellow precipitate was dissolved by dropwise addition of 0.75 ml water. Olive-green crystals suitable for X-ray analysis precipitated overnight. Total yield: 21%.



**Figure 1**  
The complex cation in (I), showing displacement ellipsoids at the 50% probability level for non-H atoms. H atoms are shown as spheres of arbitrary radius. Suffix A denotes symmetry code  $1 - x, y, \frac{3}{2} - z$ .

#### Crystal data



$M_r = 1007.35$

Monoclinic,  $C2/c$

$a = 16.1111$  (6) Å

$b = 13.7316$  (5) Å

$c = 20.1356$  (10) Å

$\beta = 111.277$  (1)°

$V = 4151.0$  (3) Å<sup>3</sup>

$Z = 4$

$D_x = 1.612$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

Cell parameters from 5148

reflections

$\theta = 2.5$ – $25.1$ °

$\mu = 0.91$  mm<sup>-1</sup>

$T = 180$  (2) K

Block, green

$0.16 \times 0.06 \times 0.04$  mm

#### Data collection

Bruker–Nonius X8 APEXII CCD diffractometer

Thin-slice  $\omega$  and  $\varphi$  scans

Absorption correction: multi-scan (SADABS; Sheldrick, 2003)

$T_{\min} = 0.851$ ,  $T_{\max} = 0.965$

24686 measured reflections

5149 independent reflections

3740 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 28.3$ °

$h = -21 \rightarrow 21$

$k = -18 \rightarrow 18$

$l = -26 \rightarrow 26$

#### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.043$

$wR(F^2) = 0.120$

$S = 1.01$

5149 reflections

291 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0611P)^2$

$+ 6.5832P]$

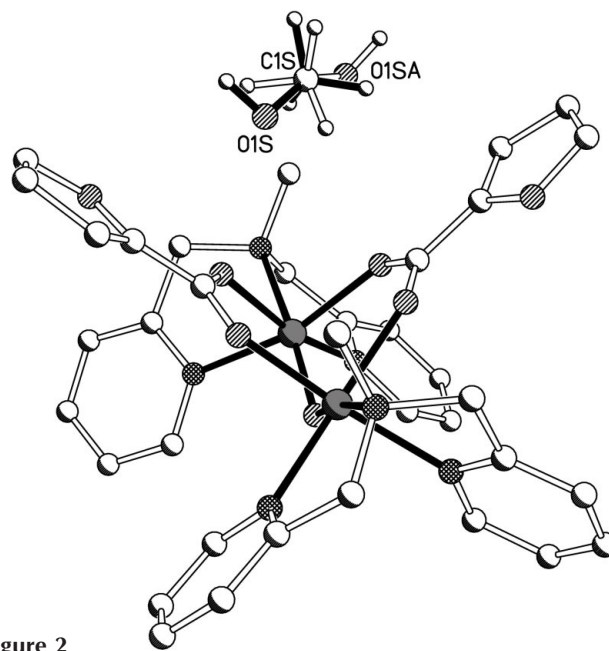
where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.32$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.70$  e Å<sup>-3</sup>

H atoms bound to C were positioned geometrically and allowed to ride during subsequent refinement, with C–H = 0.95 Å,  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  for  $\text{C}(sp^2)$ , C–H = 0.99 Å,  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  for methylene groups and C–H = 0.98 Å,  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$  for methyl groups. The methyl groups of the Fe(III)–Fe(III) complex were also allowed



**Figure 2**  
Environment of the disordered methanol molecule in (I). H atoms have been omitted from the cation, as have the perchlorate anions. Suffix A denotes symmetry code  $1 - x, y, \frac{3}{2} - z$ .

to rotate about their local threefold axes. The methanol solvent molecule was modelled as disordered, with its C atom lying on the twofold axis at  $(\frac{1}{2}, y, \frac{3}{4})$  and its O atom disordered over two sites related by the twofold rotation. The H atom of the hydroxyl group was evident in a difference Fourier map. For subsequent refinement, its position was adjusted along the observed O–H vector so that the O–H distance was 0.84 Å (the *SHELXL* default) and it was then allowed to ride on the O atom with  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$ . The H atoms of the methyl group were placed so as to form a staggered arrangement with the O–H bond and subsequently allowed to ride with C–H = 0.98 Å,  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C})$ . The highest residual peak in the electron density is associated with the perchlorate anion.

Data collection: *APEX2* (Bruker–Nonius, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

We are grateful to the Danish Natural Science Research Council (SNF) and Carlsbergfondet for provision of the X-ray equipment.

#### References

- Bruker Nonius (2004). *APEX2*. Version 1.0-22. Bruker Nonius BV, Delft, The Netherlands.
- Bruker (2003). *SAINT*. Version 7.06a. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dick, S. Weiss, A., Wagner, U., Wagner, F. & Grosse, G. (1997). *Z. Naturforsch. Teil B*, **52**, 372–384.
- Jensen, K. B., McKenzie, C. J., Simonsen, O., Toftlund, H. & Hazell, A. (1997). *Inorg. Chim. Acta*, **257**, 163–172.
- Nishino, S., Hosomi, H., Ohba, S., Matsushima, H., Tokii, T. & Nishida, Y. (1999). *J. Chem. Soc. Dalton Trans.* pp. 1509–1514.
- Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.