Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

trans-Bis(benzoato-*O*)tetrakis(methanol-*O*)iron(II): ligand bulk is not structure-determining

Sarah E. McMullen and Karl S. Hagen*

Department of Chemistry, Emory University, 1515 Pierce Drive, Atlanta, GA 30322, USA
Correspondence e-mail: khagen@emory.edu

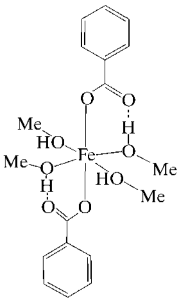
Received 10 August 2001
Accepted 5 November 2001
Online 22 December 2001

The title compound, $[Fe(C_7H_5O_2)_2(CH_4O)_4]$, is a centrosymmetric six-coordinate Fe^{II} complex coordinated by two axial monodentate benzoate ligands and four methanol ligands in the equatorial plane [$Fe-O_{benzoate}$ 2.0935 (7) Å, and $Fe-O_{methanol}$ 2.1310 (7) and 2.1290 (7) Å]. The benzoate ligands adopt monodentate ligation, rather than a bridged polymeric structure, because of strong intra- and intermolecular hydrogen bonds to the methanol ligands. This structure is nearly identical to that obtained with a much bulkier carboxylate ligand [Chavez, Que & Tolman (2001). *Chem. Commun.* pp. 111–112].

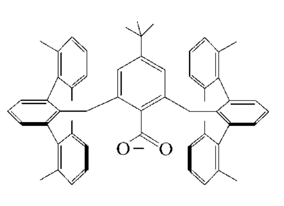
Comment

A recent report of an Fe^{II} complex having the same core structure as the title compound, (I), but with a bulky trisubstituted benzoate, attributed the atypical structure to the extreme steric bulk of the carboxylate (Chavez *et al.*, 2001). We demonstrate here that the structure can also be obtained using unsubstituted benzoic acid (Fig. 1).

The core geometry in (I) is very similar to that in the complex $[Fe(II)_2(MeOH)_4]$, (III), where (II) is 4-*tert*-butyl-2,6-bis[2,2″,6,6″-tetramethyl-*m*-terphenyl-2′-yl)methyl]phenyl carboxylate. The Fe–carboxylate bonds are slightly shorter in (I) [Fe—O1 2.0935 (7) Å] than in (III) [2.13 (2) Å], while the Fe–methanol bonds are comparable [Fe—O3 2.1310 (7) Å and Fe—O4 2.1290 (7) Å in (I), and 2.162 (2) and 2.091 (2) Å in (III)]. The charge on the benzoate ligand is delocalized over the carboxylate group [C1—O1 1.260 (1) Å and C1—O2 1.266 (1) Å]. The methanol ligands in (I) are hydrogen bonded to the uncoordinated O atom of the benzoate [intramolecular O2···O3 2.623 (1) Å and intermolecular O4···O2^{i} 2.634 (1) Å (Table 1); symmetry code: (i) $x, \frac{1}{2}-y, \frac{1}{2}+z$]. Intermolecular hydrogen bonds link the molecules into layers parallel to the *bc* plane, and the phenyl rings in adjacent layers are interleaved. The short Fe—O bond in (III) involves the methanol molecule that forms a strong hydrogen bond to the uncoordinated carboxylate O atom [O···O 2.582 (3) Å], whereas the longer bond involves a methanol that forms a weaker hydrogen bond to a solvate methanol [intramolecular O···O 3.199 (3) Å]. This difference can be attributed to the bulky carboxylate in (III).



(I)



(II)

The most closely related metal–carboxylate complexes with four additional coordinated alcohol ligands are the isomorphous Ca^{2+} and Cd^{2+} complexes of a natural product, griseocheline, from *Streptomyces griseus* (Scharfenberg-Pfeiffer & Czugler, 1991). Each of two tridentate ligands are coordinated to the metal through one carboxylate and two alcohols. However, the complexes have pseudo-twofold symmetry, with a *cis* arrangement of the two carboxylates. More common structures with simpler ligands are generally centrosymmetric with amine donors in the equatorial plane. A related structure is *trans*-bis(4-bromobenzoato-*O*)(2-dimethylaminoethanol-*N*,*O*)copper(II), in which the free O atom on the benzoate forms a hydrogen bond to the coordinated alcohol (Turpeinen *et al.*, 1996). The centrosymmetric

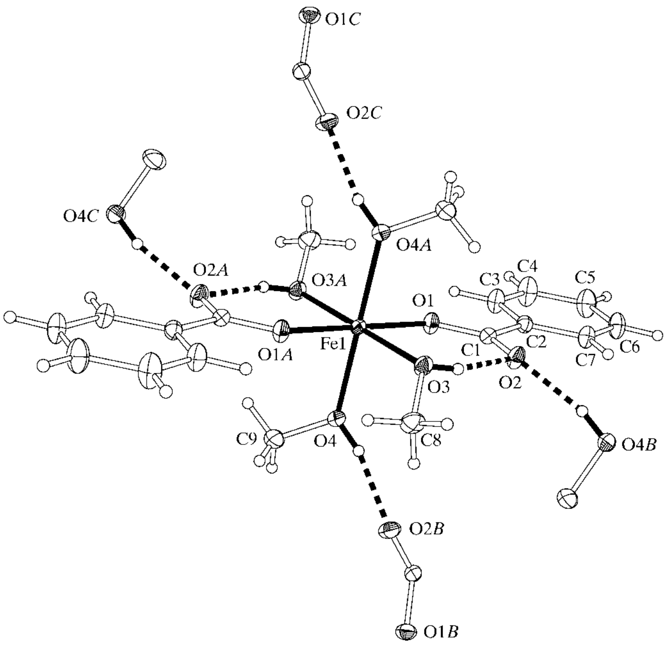


Figure 1
A view of the molecular structure of (I) with 50% probability displacement ellipsoids. H atoms are drawn as small spheres of arbitrary radii and intra- and intermolecular hydrogen bonds are indicated by dotted lines. [Symmetry codes: (*A*) $1-x, -y, 1-z$; (*B*) $x, \frac{1}{2}-y, -\frac{1}{2}+z$; (*C*) $1-x, -\frac{1}{2}+y, \frac{n}{2}-z$ ($n = 3$ for O4 and $n = 1$ for O1 and O2).]

DOI: 10.1107/S010827010101873X

structures are also preferred in the Fe^{II} and Co^{II} acetate and trifluoroacetate complexes, $[M(R\mathrm{COO})_2(\mathrm{NH_2CH_2py})_2]$, with the diamine 2-aminomethylpyridine (Payne, 1998).

Experimental

Compound (I) was crystallized from a methanol solution containing $Fe(BPh_4)_2$ (0.25 mmol), benzoic acid (1.0 mmol) and triethylamine (1 mmol). Crystals of triethylammonium tetraphenylborate, $(Et_3NH)(BPh_4)$, were also isolated from this solution.

Crystal data

$[Fe(C_7H_5O_2)_2(CH_4O)_4]$
$M_r = 426.24$
Monoclinic, $P2_1/c$
$a = 9.863$ (1) Å
$b = 12.065$ (2) Å
$c = 8.605$ (1) Å
$\beta = 101.45$ (1)°
$V = 1003.5$ (3) Å^3
$Z = 2$
$D_x = 1.411$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9801 reflections
$\theta = 2.7$–32.9°
$\mu = 0.79$ mm^{-1}
$T = 100$ (2) K
Block, colourless
0.40 × 0.25 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector on D8 diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995; Sheldrick, 2001)
$T_{\min} = 0.79$, $T_{\max} = 0.89$
11 390 measured reflections
2922 independent reflections
2731 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 30°$
$h = -13 \rightarrow 13$
$k = -16 \rightarrow 16$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.027$
$wR(F^2) = 0.078$
$S = 1.08$
2922 reflections
176 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0495P)^2 + 0.2157P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.58$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}

The H-atom parameters were freely refined, giving C—H = 0.86 (3)–0.98 (2) Å and $U_{\rm iso} = 0.029$ (4)–0.10 (1) Å^2.

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H1···O2	0.82 (2)	1.83 (2)	2.623 (1)	161 (2)
O4—H2···O2$^{\rm i}$	0.82 (2)	1.83 (2)	2.634 (1)	166 (2)

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART* and *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

We acknowledge the National Science Foundation (grant No. CHE-9974864) for funds to purchase the diffractometer.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1620). Services for accessing these data are described at the back of the journal.

References

Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2000). *SAINT*. Version 6.02a. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SMART*. Version 5.624. Bruker AXS Inc., Madison, Wisconsin, USA.
Chavez, F. A., Que, L. Jr & Tolman, W. B. (2001). *Chem. Commun.* pp. 111–112.
Payne, S. C. (1998). PhD thesis, Emory University, USA.
Scharfenberg-Pfeiffer, D. & Czugler, M. (1991). *Pharmazie*, **46**, 781–783.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. Version 2.03. University of Göttingen, Germany.
Turpeinen, U., Klinga, M., Mutikainen, I. & Hamalainen, R. (1996). *Z. Kristallogr.* **211**, 261–262.