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Key indicators

Single-crystal X-ray study
T = 273 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.040
wR factor = 0.102
Data-to-parameter ratio = 16.3

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

Porous solvent-free μ_2 -bis(salicylidene)propane-1,3-diaminato-bis[[bis(salicylidene)propane-1,3-diaminato]iron(III)]

The title compound, $[\text{Fe}_2(\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_2)_3]$, was isolated from the reaction between iron(III) perchlorate hydrate and a mixture of 1,3-bis(salicylideneamino)propane and triethylamine in ethanol. Recrystallization from a dichloroethane solution resulted in a porous solvent-free array of molecules. The molecule lies on a special position with twofold symmetry.

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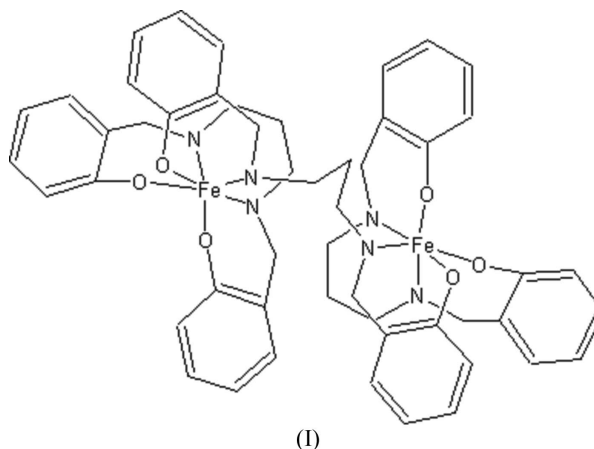
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Comment

Tetradentate bis(salicylidene) Schiff base ligands of the type $\text{HOC}_6\text{H}_4\text{CH}=\text{N}(\text{CH}_2)_n\text{N}=\text{CHC}_6\text{H}_4\text{OH}$, where $n = 1, 2$ or 3 , and their metal complexes have been extensively studied because of their physico-chemical properties, reactivity patterns, and use as catalysts and models for a variety of biological systems (Gerli *et al.*, 1991; Reddy *et al.*, 1998; Fanning *et al.*, 1995; Pasini *et al.*, 2000; Babu *et al.*, 1997). A recent search of the Cambridge Structural Database (CSD, Version 5.26 of November 2004; Allen, 2002) yielded over 500 structures of various derivatives of $\text{HOC}_6\text{H}_4\text{CH}=\text{N}(\text{CH}_2)_3\text{N}=\text{CHC}_6\text{H}_4\text{OH}$ and its metal complexes.

Although the structure of $\text{HOC}_6\text{H}_4\text{CH}=\text{N}(\text{CH}_2)_3\text{N}=\text{CHC}_6\text{H}_4\text{OH}$, H_2Salpn (Elderman *et al.*, 1991), and six of its iron complexes, including $\text{Fe}^{\text{III}}(\text{Salpn})(\mu_2\text{-Salpn})\text{Fe}^{\text{III}}(\text{Salpn})\text{-solvent}$, where solvent = ethanol or acetonitrile (Elmali *et al.*, 1993; Babu *et al.*, 1997; Gerli *et al.*, 1991; Reddy *et al.*, 1998; Fanning *et al.*, 1995; You *et al.*, 2004), have been reported, to our knowledge the structure of a solvent-free modification of the latter species has not been described to date. Here, we describe the synthesis and structure of the title solvent-free compound, $\text{Fe}^{\text{III}}(\text{Salpn})(\mu_2\text{-Salpn})\text{Fe}^{\text{III}}(\text{Salpn})$, (I). We note that this compound crystallizes in a different space group from the solvate structure determined by Babu *et al.* (1997).



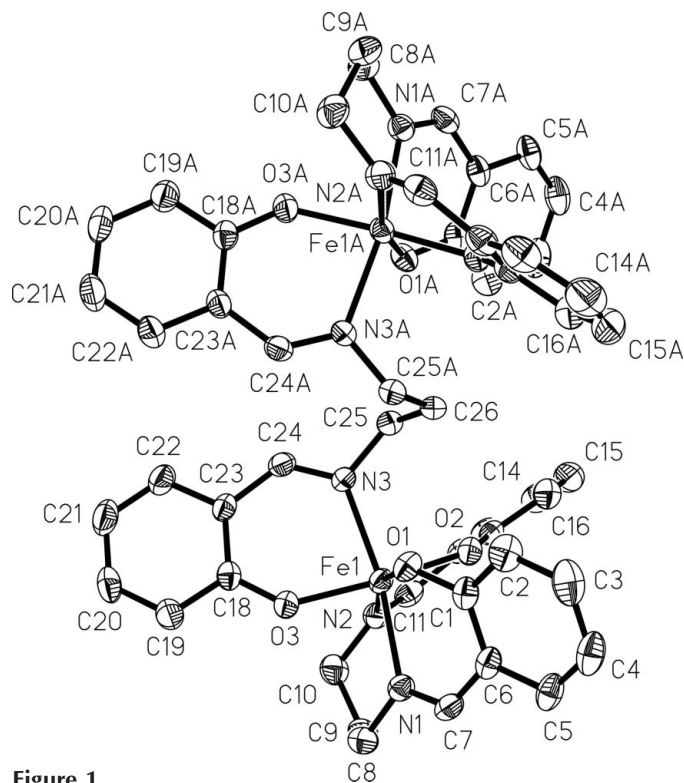


Figure 1

A drawing of the molecular structure of (I), showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity. Atoms with the label suffix A are generated by the symmetry operation $(-x, y, \frac{1}{2} - z)$.

A view of (I) (Fig. 1) reveals the presence of two Fe^{III} ions accompanied by two terminal tetradentate-*N,N,O,O* Salpn $^{2-}$ anions and one bridging bis(bidentate-*N,O*) Salpn $^{2-}$ species. The complete molecule is generated by twofold symmetry (atom C26 is located on the twofold axis). The coordination about the Fe atom is approximately octahedral, with the equatorial sites occupied by two O atoms and one N atom from the terminal Salpn $^{2-}$ and one O atom from bridging Salpn $^{2-}$. The axial sites are occupied by N atoms from terminal (N1) and bridging Salpn. Overall, a *fac*- FeN_3O_3 coordination geometry results. The deviations of the *cis* bond angles from 90° can be correlated with the constraints associated with the tetradentate and bidentate binding modes of Salpn $^{2-}$. The *N,N*-metallocyclic ring adopts a chair conformation. The overall geometry of (I) is similar to that reported for $\text{Fe}^{\text{III}}(\text{Salpn})(\mu_2\text{-Salpn})\text{Fe}^{\text{III}}(\text{Salpn})\cdot\text{solvent}$ (solvent = acetonitrile; Babu *et al.*, 1997).

A view of the packing in (I) along the *c* axis is shown in Fig. 2. There may be some weak C—H...O interactions (Table 2) involved in stabilizing the conformation of (I). The intermolecular voids are clearly discernible. A calculation of the solvent-accessible volume yielded 352 \AA^3 of empty space per unit cell in the structure (Spek, 2003, 2004). This is consistent with the final Fourier difference map, which shows no peak greater than 0.5 e \AA^{-3} after the final refinement. The vacant regions may arise from geometrical packing constraints, or may be due to loss of solvent of crystallization without structure collapse.

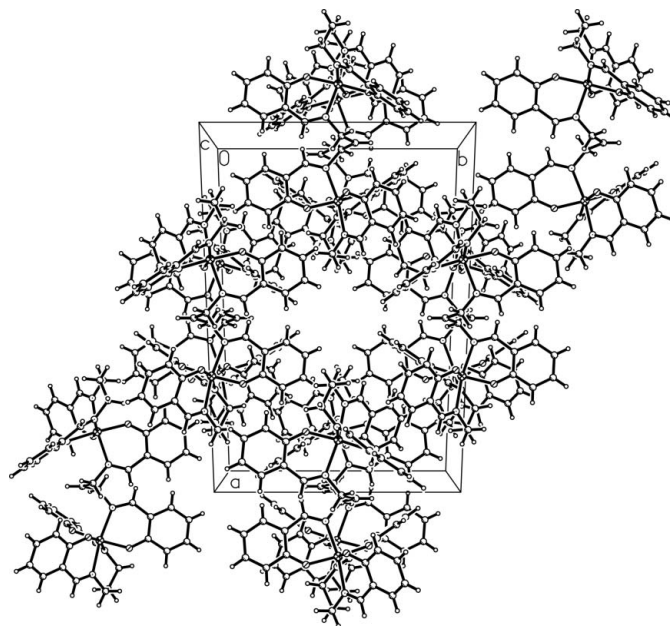


Figure 2

The packing of (I), viewed along the *c* axis.

Experimental

Compound (I), $\text{Fe}^{\text{III}}(\text{Salpn})(\mu_2\text{-Salpn})\text{Fe}^{\text{III}}(\text{Salpn})$, was isolated in good yield from $\text{Fe}(\text{ClO}_4)_3 \cdot x\text{H}_2\text{O}$ (354 mg), 1,3-bis(salicylidene-imino)propane (423 mg) and $(\text{CH}_3\text{CH}_2)_3\text{N}$ in ethanol (50 ml), following a procedure similar to that described by Babu *et al.* (1997). Solvent-free dark-red crystals of (I) were obtained from the slow evaporation of a dichloromethane solution of $\text{Fe}^{\text{III}}(\text{Salpn})(\mu_2\text{-Salpn})\text{Fe}^{\text{III}}(\text{Salpn})$.

Crystal data

$[\text{Fe}_2(\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_2)_3]$
 $M_r = 952.65$
 Monoclinic, $C2/c$
 $a = 24.463 (4) \text{ \AA}$
 $b = 14.818 (2) \text{ \AA}$
 $c = 15.911 (3) \text{ \AA}$
 $\beta = 121.138 (2)^\circ$
 $V = 4936.8 (14) \text{ \AA}^3$
 $Z = 4$

$D_x = 1.282 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 1015 reflections
 $\theta = 1.6\text{--}26.0^\circ$
 $\mu = 0.64 \text{ mm}^{-1}$
 $T = 273 (2) \text{ K}$
 Block, dark red
 $0.44 \times 0.40 \times 0.24 \text{ mm}$

Data collection

Bruker SMART APEX CCD area-detector diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.766$, $T_{\text{max}} = 0.861$
 13618 measured reflections

4846 independent reflections
 3692 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = -29 \rightarrow 29$
 $k = -18 \rightarrow 14$
 $l = -15 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.102$
 $S = 1.01$
 4846 reflections
 298 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 2.0537P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Fe1—O1	1.9227 (14)	Fe1—N2	2.1491 (19)
Fe1—O3	1.9458 (15)	Fe1—N1	2.1725 (18)
Fe1—O2	1.9603 (15)	Fe1—N3	2.1859 (17)
O1—Fe1—O3	99.21 (7)	O2—Fe1—N1	100.32 (7)
O1—Fe1—O2	88.95 (6)	N2—Fe1—N1	82.50 (7)
O3—Fe1—O2	170.59 (6)	O1—Fe1—N3	94.35 (6)
O1—Fe1—N2	163.35 (7)	O3—Fe1—N3	85.01 (6)
O3—Fe1—N2	90.10 (7)	O2—Fe1—N3	89.74 (6)
O2—Fe1—N2	83.14 (7)	N2—Fe1—N3	100.22 (7)
O1—Fe1—N1	84.55 (7)	N1—Fe1—N3	169.84 (7)
O3—Fe1—N1	85.20 (7)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C10—H10A...O3	0.97	2.50	3.052 (4)	116
C26—H26A...O2	0.95 (2)	2.56 (3)	3.213 (2)	126 (2)

Atom H26A attached to atom C26 was positioned geometrically and freely refined. All other H atoms were placed in idealized geometry, with C—H distances in the range 0.93–0.97 Å, and were refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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