1,1,1-Tris(2-hydroxybenzylideneamino)-methylpropane iron(III) chloroform solvate

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Key indicators

Single-crystal X-ray study

$T = 123$ K

Mean σ(C–C) = 0.003 Å

R factor = 0.036

wR factor = 0.087

Data-to-parameter ratio = 14.1

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

The X-ray crystal structure of the hexadentate Schiff base complex, $[Fe(C_{27}H_{26}N_3O_3)]\cdot CHCl_3$, is presented. This species contains an iron atom coordinated within an octahedral N_3O_3 environment.

Comment

As a consequence of our recent studies on the coordination chemistry of tetradentate Schiff bases with 1,3-propanediamine (Reglinski *et al.*, 2002*a,b*), we wished to extend our catalogue of complexes to include hexadentate species with similar steric influences around the imine regions. To this end we prepared 1,1,1-tris(2-hydroxybenzylideneamino)methylpropane from the relevant triamine (Fleischer *et al.*, 1971; Hellmann *et al.*, 1995) and salicylaldehyde. To ensure that the ligand behaved in the expected manner we prepared a sample of the iron(III) complex, (I), from ferrous acetate *via* air oxidation.
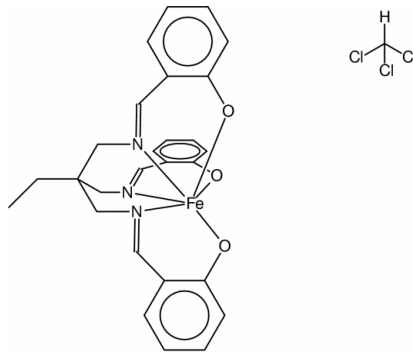
(I)

Experimental

On recrystallization of the title complex from chloroform/diethyl ether using vapour diffusion methods, we obtained deep-red crystals of (I) suitable for X-ray analysis (Found C, 54.46; H, 4.15; N, 6.75: Expected for $C_{27}H_{26}N_3O_3Fe\cdot CHCl_3$ C, 54.62; H, 4.42; N, 6.82).

Crystal data

$[Fe(C_{27}H_{26}N_3O_3)]\cdot CHCl_3$

$M_r = 615.75$

Orthorhombic, *Pbca*

$a = 13.6479$ (2) Å

$b = 18.8071$ (2) Å

$c = 21.6016$ (3) Å

$V = 5544.64$ (13) Å^3

$Z = 8$

$D_x = 1.475$ Mg m^{-3}

Mo $K\alpha$ radiation
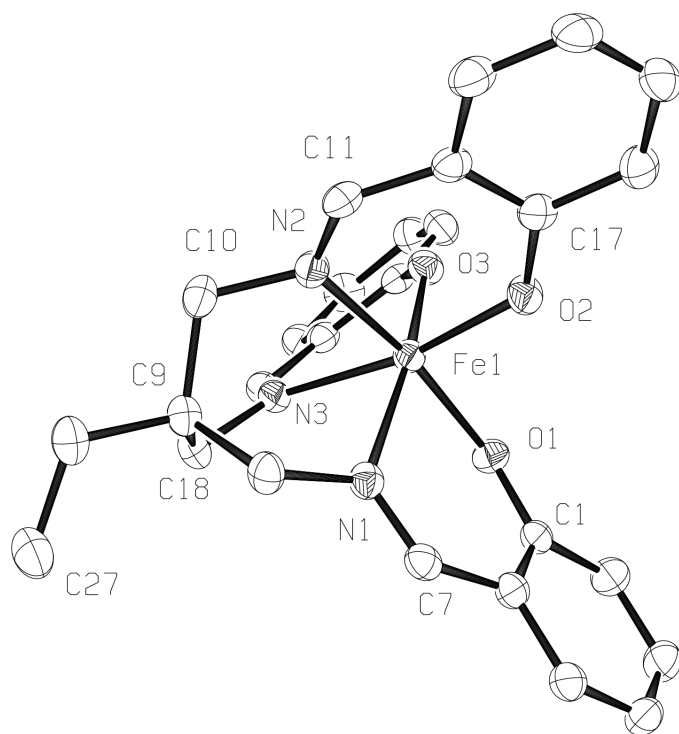
Cell parameters from 6345 reflections

$\theta = 3.2$–27.5°

$\mu = 0.87$ mm^{-1}

$T = 123$ (2) K

Prism, red

$0.27 \times 0.15 \times 0.12$ mm

Figure 1
*ORTEP*II view of (I), with non-H atoms drawn as 50% probability ellipsoids. The solvent molecule has been omitted

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: none
11957 measured reflections
6345 independent reflections
4670 reflections with $I > 2\sigma(I)$

$R_{int} = 0.029$
$\theta_{max} = 27.5°$
$h = -17 \rightarrow 17$
$k = -24 \rightarrow 24$
$l = -27 \rightarrow 28$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.087$
$S = 1.03$
6345 reflections
451 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 2.1937P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—N1	2.1690 (16)	Fe1—O1	1.9454 (13)
Fe1—N2	2.1514 (15)	Fe1—O2	1.9323 (13)
Fe1—N3	2.1262 (15)	Fe1—O3	1.9498 (13)
O2—Fe1—O1	93.16 (6)	O3—Fe1—N2	103.37 (6)
O2—Fe1—O3	91.22 (6)	N3—Fe1—N2	84.82 (6)
O1—Fe1—O3	91.94 (5)	O2—Fe1—N1	101.43 (6)
O2—Fe1—N3	169.63 (6)	O1—Fe1—N1	84.39 (6)
O1—Fe1—N3	96.83 (6)	O3—Fe1—N1	166.98 (6)
O3—Fe1—N3	85.67 (6)	N3—Fe1—N1	82.38 (6)
O2—Fe1—N2	86.28 (6)	N2—Fe1—N1	80.73 (6)
O1—Fe1—N2	164.68 (6)		

All H atoms were found in a difference Fourier synthesis and refined isotropically. The C—H bond lengths are 0.90 (2)–1.00 (2) Å.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1988); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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