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A dinuclear oxygen-bridged Schiff base iron(III) complex derived from *N*,*N*′-bis(2-hydroxybenzylidene)-1,2-diaminopropane

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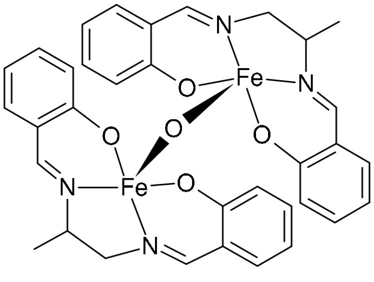
The title compound, μ-oxo-bis({2,2-[propane-1,2-diylbis(nitrilomethylidyne)]diphenolato}iron(III)), $[Fe_2(C_{17}H_{16}N_2O_2)_2O]$, is an unusual dinuclear iron(III) complex. Each Fe^{III} ion has a distorted square-pyramidal coordination geometry. In the basal plane, the Fe atom is coordinated by two N atoms and two O atoms of the Schiff base ligand. The apical position is occupied by a bridging O^{2-} ion, which links to the other Fe^{III} ion in the complex. In the crystal structure, pairs of dinuclear iron(III) units form dimers by way of a pair of intermolecular C—H$\cdots$O interactions. The dimers stack along the *a* axis and there are no other short intermolecular contacts.

Key indicators
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.08 Å
R factor = 0.056
wR factor = 0.161
Data-to-parameter ratio = 13.3

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

Comment

Investigation into the magnetic properties of molecule-based materials containing a polynuclear assembly has become a fascinating subject in the field of condensed matter physics and materials chemistry (Dalai *et al.*, 2002; Bhaduri *et al.*, 2003). Much attention has been focused on coordination complexes with novel magnetic properties, which may have potentially useful applications in materials science (Ray *et al.*, 2003). The prime strategy for designing these molecular materials is to use a suitable bridging ligand that determines the nature of the magnetic interactions (Koner *et al.*, 2003). Schiff base iron(III) complexes with O atoms as the bridging ligands have been of great interest in the past few years (Ashmawy *et al.*, 1991; Corazza *et al.*, 1987). Recently, we have reported some Schiff base complexes (You *et al.*, 2004; You & Zhu, 2004; You *et al.*, 2004). As an extension of this work, a novel dinuclear iron(III) complex, (I) (Fig. 1), is reported here. Its most interesting feature is a bridging oxide ligand.



(I)

Each iron(III) ion in (I) has a square-pyramidal coordination geometry, involving two N atoms and two O atoms from a Schiff base ligand, and one apical O5 atom. The O5 atom acts as a bridging function, and coordinates to both Fe1 and Fe2 atoms.The Fe$\cdots$Fe separation is 3.418 (4) Å. The significant distortion of each of the square pyramids is revealed by the

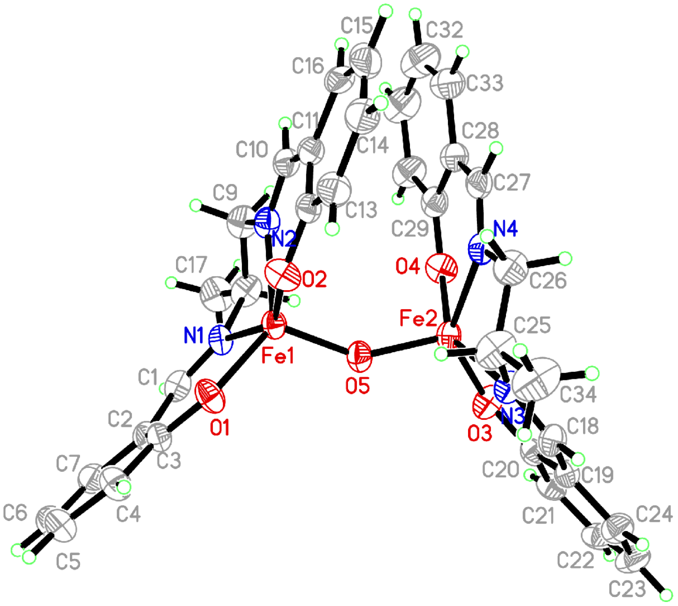


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

bond angles between the apical and basal donor atoms (Table 1), which show deviations, of 17.2 (2)° for the Fe1 grouping, and 16.4 (2)° for the Fe2 grouping, from the ideal 90° angle in a regular square pyramid. The smallest bond angles for the basal donor atoms [76.29 (19)° for N1—Fe1—N2 and 75.81 (19)° for N3—Fe2—N4] correlate with the strained ligand bite angle for the five-membered chelate rings. The other basal angles are closer to 90° [86.17 (18)–90.24 (15)° for Fe1 and 86.23 (18)–93.19 (17)° for Fe2].

The Fe—O5 bond lengths (Table 1) are substantially shorter than the average bond lengths between the Fe atoms and the basal donor atoms [2.005 (4) Å for the Fe1 moiety, and 2.012 (4) Å for the Fe2 moiety]. The Fe1—O5—Fe2 bond angle of 147.5 (2)° is comparable to the corresponding value of 146.69 (3)° observed in a similar Schiff base iron(III) complex (Ashmawy *et al.*, 1991).

Atoms C8 and C9 deviate by 0.759 (7) and 0.220 (7) Å, respectively, from the N1/Fe1/N2 plane. Atoms C25 and C26 deviate by 0.831 (7) and 0.320 (7) Å, respectively, from the N3/Fe2/N4 plane. The deviations of atoms Fe1 and Fe2 from the N1/N2/O2/O1 and N3/N4/O4/O3 square planes are 0.600 (2) and 0.573 (2) Å, respectively. The dihedral angle between the two benzene rings associated with the Fe1 moiety is 32.5 (6)°, with a corresponding value of 38.2 (6)° for Fe2.

The average value of the C1═N1, C10═N2, C18═N3 and C27═N4 bond lengths in (I) is 1.269 (6) Å, which conforms to the value for a C═N double bond, while the average value of the C8—N1, C9—N2, C25—N3 and C26—N4 bond lengths is 1.471 (7) Å, which conforms to the value for a C—N single bond.

In the crystal structure of (I), the dinuclear molecules form dimers by way of a pair of intermolecular C—H···O interactions (Table 2). The dimers stack along the *a* axis and there are no other short intermolecular contacts (Fig. 2).

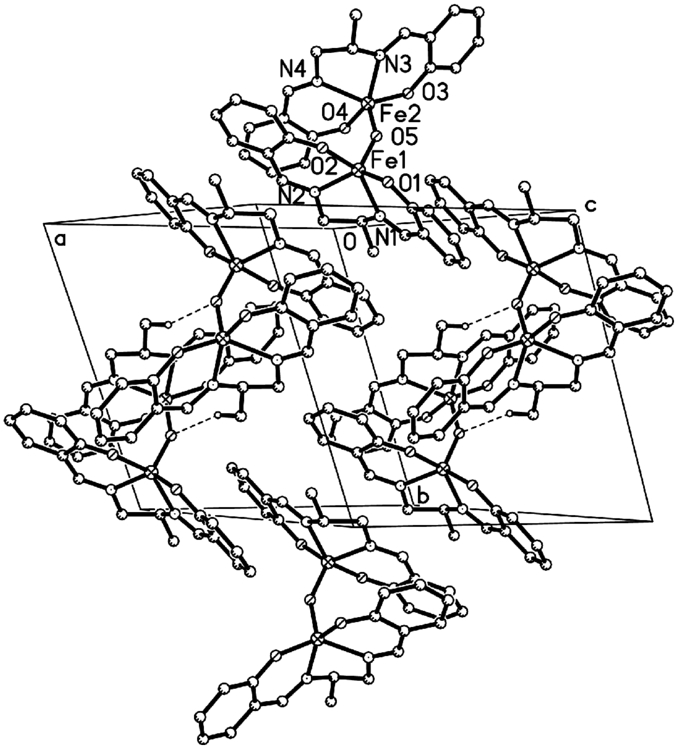


Figure 2
The crystal packing of (I), showing the C34—H34*A*···O5 interactions as dashed lines. All other H atoms have been omitted for clarity.

Experimental

1,2-Diaminopropane (0.1 mmol, 7.4 mg) and salicylaldehyde (0.2 mmol, 24.4 mg) were dissolved in methanol (5 ml). The mixture was stirred for 10 min at room temperature to give a clear yellow solution. To the solution was added a methanol solution (5 ml) of $Fe(NO_3)_3 \cdot 9H_2O$ (0.1 mmol, 34.2 mg), with stirring. The mixture was stirred for another 10 min, and allowed to stand in air for 12 d. Some brown block-shaped crystals of (I) were formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed three times with methanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 71.3%). Analysis found: C 59.1, H 4.7, N 8.2%; calculated for $C_{34}H_{32}Fe_2N_4O_5$: C 59.3, H 4.7, N 8.1%.

Crystal data

$[Fe_2(C_{17}H_{16}N_2O_2)_2O]$
M_r = 688.34
Triclinic, $P\overline{1}$
a = 11.462 (4) Å
b = 12.478 (4) Å
c = 13.230 (5) Å
α = 95.491 (6)°
β = 115.070 (5)°
γ = 110.291 (5)°
V = 1540.6 (9) Å^3

Z = 2
D_x = 1.484 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1812 reflections
θ = 2.2–24.3°
μ = 0.99 mm^{-1}
T = 298 (2) K
Block, brown
0.29 × 0.23 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.762, T_{max} = 0.858
8247 measured reflections

5385 independent reflections
2776 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.0°
$h = -13 \rightarrow 13$
$k = -10 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.161$
$S = 0.91$
5385 reflections
406 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0834P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.0001$
$\Delta\rho_{max} = 0.61$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O5	1.770 (4)	Fe2—O5	1.790 (3)
Fe1—O1	1.910 (4)	Fe2—O3	1.897 (4)
Fe1—O2	1.914 (4)	Fe2—O4	1.928 (4)
Fe1—N1	2.090 (4)	Fe2—N3	2.099 (4)
Fe1—N2	2.105 (5)	Fe2—N4	2.123 (4)
O5—Fe1—O1	114.84 (16)	O5—Fe2—O3	113.35 (17)
O5—Fe1—O2	107.35 (17)	O5—Fe2—O4	106.04 (16)
O1—Fe1—O2	90.24 (15)	O3—Fe2—O4	93.19 (17)
O5—Fe1—N1	100.94 (16)	O5—Fe2—N3	101.02 (16)
O1—Fe1—N1	87.01 (16)	O3—Fe2—N3	86.25 (17)
O2—Fe1—N1	149.90 (18)	O4—Fe2—N3	150.66 (17)
O5—Fe1—N2	105.87 (16)	O5—Fe2—N4	105.23 (17)
O1—Fe1—N2	138.24 (17)	O3—Fe2—N4	139.89 (17)
O2—Fe1—N2	86.17 (18)	O4—Fe2—N4	86.23 (18)
N1—Fe1—N2	76.29 (19)	N3—Fe2—N4	75.81 (19)

Table 2
Hydrogen-bond geometry (Å, °).

	D—H	H···*A*	*D*···*A*	*D*—H···*A*
C34—H34*A*···O5^{i}	0.96	2.56	3.454 (7)	155

Symmetry code: (i) $-x, -y+1, -z+1$.

All H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H distances of 0.93–0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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