

μ -Oxido-bis({4,4'-dibromo-2,2'-ethane-1,2-diylbis(nitrilomethylidene)-diphenolato}iron(III))

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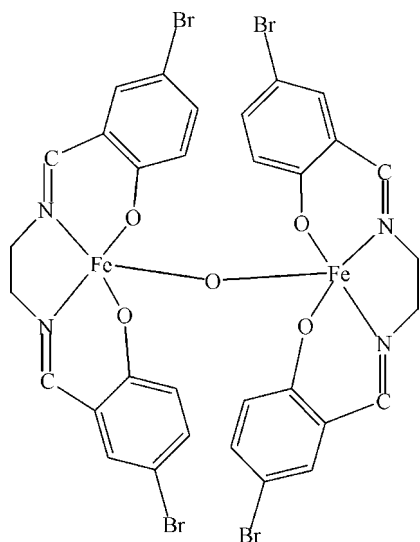
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.059; wR factor = 0.147; data-to-parameter ratio = 14.9.

In the title compound, $[\text{Fe}_2(\text{C}_{16}\text{H}_{12}\text{Br}_2\text{N}_2\text{O}_2)_2\text{O}]$, the complete molecule is generated by twofold symmetry, with the bridging O atom, which links the iron centres, lying on the rotation axis. The Fe(III) ion is chelated by the N,N,O,O -tetradentate Schiff base dianion, resulting in an FeN_2O_3 square-based pyramid, with the two N atoms in the basal plane.

Related literature

For related literature, see: Karacan & Somer (2004); Chen *et al.* (2006).



Experimental

Crystal data

$[\text{Fe}_2(\text{C}_{16}\text{H}_{12}\text{Br}_2\text{N}_2\text{O}_2)_2\text{O}]$	$V = 3486.3$ (7) Å ³
$M_r = 975.89$	$Z = 4$
Orthorhombic, $Pcca$	Mo $K\alpha$ radiation
$a = 21.094$ (2) Å	$\mu = 5.46$ mm ⁻¹
$b = 13.8168$ (18) Å	$T = 293$ (2) K
$c = 11.9619$ (12) Å	$0.43 \times 0.28 \times 0.22$ mm

Data collection

Bruker APEXII CCD diffractometer	11548 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2001)	3182 independent reflections
$T_{\min} = 0.202$, $T_{\max} = 0.380$	2257 reflections with $I > 2\sigma(I)$
(expected range = 0.160–0.301)	$R_{\text{int}} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$	213 parameters
$wR(F^2) = 0.147$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{\text{max}} = 1.05$ e Å ⁻³
3182 reflections	$\Delta\rho_{\text{min}} = -0.70$ e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

Fe1—O3	1.8162 (18)	Fe1—N2	2.116 (4)
Fe1—O2	1.926 (4)	Fe1—N1	2.141 (4)
Fe1—O1	1.930 (4)		
Fe1 ⁱ —O3—Fe1	139.4 (3)		

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2001); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2647).

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supplementary materials

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μ -Oxido-bis({4,4'-dibromo-2,2'-ethane-1,2-diylbis(nitrilomethylidene)]diphenolato}iron(III))

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Comment

Recently, Schiff base ligands, especially flexible symmetrical or unsymmetrical Schiff base ligands and their hydrogenated derivatives have been widely employed to assembly alkoxo- or phenoxo-bridged manganese clusters and polymers with novel topological structures and interesting magnetic, catalysis and photochemical properties. (Karacan & Somer, 2004; Chen *et al.*, 2006). In this paper, we report the structure of the title compound, (I).

As shown in Fig. 1, the Fe(III) ion in (I) is chelated by the dianionic Schiff base ligand in a tetradentate *N,N,O,O* coordination in an approximately square planar arrangement. An oxo ligand (site symmetry 2) caps the FeN_2O_2 grouping to result in a square based pyramid. The oxo ligand also bridges to a second, crystallographically generated Fe atom. The Fe—O capping distance is much shorter than the other bonds (Table 1). The $\text{Fe}\cdots\text{Fe}^i$ ($i = -x, y, 1/2 - z$) distance is 3.4066 (12) Å.

Experimental

A mixture of iron(III) chloride (1 mmol) and *N,N'*-bis(2-hydroxy-5-bromobenzyl)ethylenediamine (1 mmol) in 20 ml methanol was refluxed for two hours. The above cooled solution was filtered and the filtrate was evaporated naturally at room temperature. Two days later, brown blocks of (I) were obtained with a yield of 32%. Anal. Calc. for $\text{C}_{32}\text{H}_{24}\text{Br}_4\text{Fe}_2\text{N}_4\text{O}_5$: C 39.34, H 2.46, N 5.74%; Found: C 39.32, H 2.48, N 5.69%.

Refinement

The H atoms were included in calculated positions (C—H = 0.93–0.97 Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

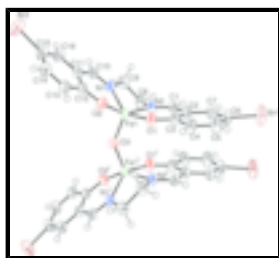


Fig. 1. The molecular structure of (I), drawn with 50% probability displacement ellipsoids for the non-hydrogen atoms. Symmetry code: (i) $-x, y, 1/2 - z$.

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Crystal data

$[\text{Fe}_2(\text{C}_{16}\text{H}_{12}\text{Br}_2\text{N}_2\text{O}_2)_2\text{O}]$	$F_{000} = 1904$
$M_r = 975.89$	$D_x = 1.859 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pcca</i>	Mo $K\alpha$ radiation
Hall symbol: -P 2a 2ac	$\lambda = 0.71073 \text{ \AA}$
$a = 21.094 (2) \text{ \AA}$	Cell parameters from 3182 reflections
$b = 13.8168 (18) \text{ \AA}$	$\theta = 3.0\text{--}25.4^\circ$
$c = 11.9619 (12) \text{ \AA}$	$\mu = 5.46 \text{ mm}^{-1}$
$V = 3486.3 (7) \text{ \AA}^3$	$T = 293 (2) \text{ K}$
$Z = 4$	Block, brown
	$0.43 \times 0.28 \times 0.22 \text{ mm}$

Data collection

Bruker APEX II CCD diffractometer	3182 independent reflections
Radiation source: fine-focus sealed tube	2257 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.049$
$T = 293(2) \text{ K}$	$\theta_{\text{max}} = 25.4^\circ$
φ and ω scans	$\theta_{\text{min}} = 3.0^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2001)	$h = -25 \rightarrow 25$
$T_{\text{min}} = 0.202$, $T_{\text{max}} = 0.380$	$k = -16 \rightarrow 16$
11548 measured reflections	$l = 0 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.059$	H-atom parameters constrained
$wR(F^2) = 0.147$	$w = 1/[\sigma^2(F_o^2) + (0.077P)^2]$
$S = 1.00$	where $P = (F_o^2 + 2F_c^2)/3$
3182 reflections	$(\Delta/\sigma)_{\text{max}} = 0.018$
213 parameters	$\Delta\rho_{\text{max}} = 1.05 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.70 \text{ e \AA}^{-3}$
	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations

between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Fe1	0.07194 (4)	0.12987 (5)	0.31467 (6)	0.0342 (2)
Br1	0.10796 (4)	0.66034 (5)	0.24478 (7)	0.0776 (3)
Br2	0.28253 (3)	-0.28115 (5)	0.48690 (6)	0.0663 (3)
C1	0.0405 (3)	0.3280 (4)	0.4134 (4)	0.0404 (13)
H1	0.0230	0.3670	0.4687	0.048*
C2	0.0734 (3)	0.3750 (4)	0.3197 (5)	0.0392 (12)
C3	0.1029 (3)	0.3259 (4)	0.2294 (5)	0.0405 (13)
C4	0.1303 (3)	0.3799 (4)	0.1413 (5)	0.0514 (15)
H4	0.1477	0.3477	0.0803	0.062*
C5	0.1316 (3)	0.4776 (4)	0.1447 (6)	0.0574 (17)
H5	0.1504	0.5126	0.0872	0.069*
C6	0.1039 (3)	0.5260 (4)	0.2367 (5)	0.0519 (16)
C7	0.0744 (3)	0.4748 (4)	0.3235 (5)	0.0476 (14)
H7	0.0557	0.5077	0.3829	0.057*
C8	0.0009 (3)	0.1958 (4)	0.5204 (5)	0.0432 (14)
H8A	-0.0382	0.1650	0.4970	0.052*
H8B	-0.0091	0.2462	0.5741	0.052*
C9	0.0461 (3)	0.1213 (4)	0.5721 (4)	0.0423 (13)
H9A	0.0765	0.1534	0.6202	0.051*
H9B	0.0224	0.0751	0.6166	0.051*
C10	0.1146 (3)	-0.0002 (4)	0.5091 (4)	0.0385 (13)
H10	0.1140	-0.0181	0.5840	0.046*
C11	0.1561 (3)	-0.0556 (4)	0.4331 (4)	0.0370 (12)
C12	0.1643 (3)	-0.0340 (3)	0.3168 (4)	0.0356 (11)
C13	0.2041 (3)	-0.0949 (4)	0.2516 (5)	0.0410 (13)
H13	0.2075	-0.0849	0.1749	0.049*
C14	0.2370 (3)	-0.1675 (4)	0.3005 (5)	0.0474 (14)
H14	0.2640	-0.2058	0.2576	0.057*
C15	0.2308 (3)	-0.1858 (4)	0.4169 (5)	0.0463 (14)
C16	0.1901 (3)	-0.1313 (4)	0.4831 (5)	0.0428 (14)
H16	0.1856	-0.1448	0.5589	0.051*
N1	0.0797 (2)	0.0703 (3)	0.4793 (4)	0.0363 (10)
N2	0.0348 (2)	0.2370 (3)	0.4227 (4)	0.0360 (10)
O1	0.10571 (18)	0.2321 (3)	0.2223 (3)	0.0435 (9)
O2	0.13721 (19)	0.0409 (3)	0.2686 (3)	0.0414 (9)
O3	0.0000	0.0842 (4)	0.2500	0.0388 (12)

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Fe1	0.0433 (5)	0.0267 (4)	0.0327 (4)	0.0006 (3)	0.0003 (3)	-0.0002 (3)
Br1	0.1084 (7)	0.0293 (4)	0.0950 (6)	-0.0024 (3)	0.0277 (5)	0.0093 (4)
Br2	0.0660 (5)	0.0586 (5)	0.0744 (5)	0.0225 (3)	0.0087 (4)	0.0251 (4)
C1	0.044 (3)	0.036 (3)	0.041 (3)	0.001 (2)	0.001 (3)	-0.007 (2)
C2	0.042 (3)	0.031 (3)	0.045 (3)	0.000 (2)	-0.001 (3)	0.002 (2)
C3	0.042 (3)	0.037 (3)	0.043 (3)	-0.003 (2)	0.003 (3)	0.001 (3)
C4	0.062 (4)	0.042 (3)	0.050 (3)	0.001 (3)	0.007 (3)	0.011 (3)
C5	0.067 (4)	0.040 (3)	0.066 (4)	-0.007 (3)	0.013 (3)	0.009 (3)
C6	0.064 (4)	0.029 (3)	0.064 (4)	-0.004 (3)	0.005 (3)	0.012 (3)
C7	0.051 (4)	0.032 (3)	0.060 (4)	0.001 (3)	0.004 (3)	0.002 (3)
C8	0.050 (4)	0.038 (3)	0.042 (3)	0.001 (3)	0.011 (3)	0.004 (3)
C9	0.052 (3)	0.041 (3)	0.034 (3)	-0.001 (3)	0.010 (3)	0.001 (2)
C10	0.051 (4)	0.030 (3)	0.035 (3)	-0.006 (2)	-0.001 (3)	0.001 (2)
C11	0.041 (3)	0.030 (3)	0.041 (3)	-0.001 (2)	-0.003 (2)	0.003 (2)
C12	0.043 (3)	0.027 (2)	0.037 (3)	-0.001 (2)	0.001 (2)	-0.001 (2)
C13	0.049 (3)	0.034 (3)	0.041 (3)	0.003 (2)	0.001 (3)	0.001 (2)
C14	0.048 (3)	0.038 (3)	0.057 (4)	0.005 (3)	0.008 (3)	0.002 (3)
C15	0.044 (3)	0.043 (3)	0.052 (3)	0.003 (3)	0.005 (3)	0.016 (3)
C16	0.047 (3)	0.038 (3)	0.043 (3)	-0.001 (3)	0.002 (3)	0.010 (3)
N1	0.045 (3)	0.029 (2)	0.035 (2)	0.0013 (19)	0.002 (2)	-0.0019 (19)
N2	0.042 (3)	0.025 (2)	0.041 (2)	-0.0042 (18)	0.005 (2)	0.0019 (19)
O1	0.055 (3)	0.030 (2)	0.045 (2)	-0.0026 (17)	0.0101 (19)	0.0019 (17)
O2	0.056 (2)	0.034 (2)	0.0346 (18)	0.0113 (17)	-0.0020 (18)	0.0024 (16)
O3	0.045 (3)	0.028 (3)	0.043 (3)	0.000	-0.009 (2)	0.000

Geometric parameters (\AA , $^\circ$)

Fe1—O3	1.8162 (18)	C8—C9	1.533 (8)
Fe1—O2	1.926 (4)	C8—H8A	0.9700
Fe1—O1	1.930 (4)	C8—H8B	0.9700
Fe1—N2	2.116 (4)	C9—N1	1.493 (7)
Fe1—N1	2.141 (4)	C9—H9A	0.9700
Br1—C6	1.861 (6)	C9—H9B	0.9700
Br2—C15	1.905 (6)	C10—N1	1.272 (7)
C1—N2	1.268 (6)	C10—C11	1.474 (7)
C1—C2	1.470 (7)	C10—H10	0.9300
C1—H1	0.9300	C11—C16	1.404 (7)
C2—C7	1.380 (7)	C11—C12	1.434 (7)
C2—C3	1.419 (8)	C12—O2	1.314 (6)
C3—O1	1.299 (6)	C12—C13	1.422 (7)
C3—C4	1.414 (8)	C13—C14	1.354 (8)
C4—C5	1.351 (8)	C13—H13	0.9300
C4—H4	0.9300	C14—C15	1.421 (8)
C5—C6	1.414 (9)	C14—H14	0.9300
C5—H5	0.9300	C15—C16	1.388 (8)

C6—C7	1.402 (8)	C16—H16	0.9300
C7—H7	0.9300	O3—Fe1 ⁱ	1.8162 (18)
C8—N2	1.483 (7)		
O3—Fe1—O2	104.71 (16)	N1—C9—C8	108.2 (4)
O3—Fe1—O1	108.58 (16)	N1—C9—H9A	110.1
O2—Fe1—O1	92.29 (16)	C8—C9—H9A	110.1
O3—Fe1—N2	101.14 (16)	N1—C9—H9B	110.1
O2—Fe1—N2	152.39 (17)	C8—C9—H9B	110.1
O1—Fe1—N2	88.51 (16)	H9A—C9—H9B	108.4
O3—Fe1—N1	108.78 (15)	N1—C10—C11	124.6 (5)
O2—Fe1—N1	87.89 (16)	N1—C10—H10	117.7
O1—Fe1—N1	141.27 (17)	C11—C10—H10	117.7
N2—Fe1—N1	74.66 (16)	C16—C11—C12	120.4 (5)
N2—C1—C2	123.4 (5)	C16—C11—C10	115.4 (5)
N2—C1—H1	118.3	C12—C11—C10	124.2 (5)
C2—C1—H1	118.3	O2—C12—C13	118.8 (4)
C7—C2—C3	119.8 (5)	O2—C12—C11	122.4 (5)
C7—C2—C1	115.0 (5)	C13—C12—C11	118.7 (5)
C3—C2—C1	125.1 (5)	C14—C13—C12	120.3 (5)
O1—C3—C2	123.1 (5)	C14—C13—H13	119.8
O1—C3—C4	117.3 (5)	C12—C13—H13	119.8
C2—C3—C4	119.6 (5)	C13—C14—C15	120.5 (5)
C5—C4—C3	120.9 (6)	C13—C14—H14	119.8
C5—C4—H4	119.6	C15—C14—H14	119.8
C3—C4—H4	119.6	C16—C15—C14	121.3 (5)
C4—C5—C6	119.2 (6)	C16—C15—Br2	118.6 (4)
C4—C5—H5	120.4	C14—C15—Br2	120.0 (4)
C6—C5—H5	120.4	C15—C16—C11	118.5 (5)
C7—C6—C5	121.4 (5)	C15—C16—H16	120.7
C7—C6—Br1	119.0 (5)	C11—C16—H16	120.7
C5—C6—Br1	119.5 (4)	C10—N1—C9	115.4 (4)
C2—C7—C6	119.1 (6)	C10—N1—Fe1	126.6 (4)
C2—C7—H7	120.5	C9—N1—Fe1	117.8 (3)
C6—C7—H7	120.5	C1—N2—C8	119.7 (5)
N2—C8—C9	106.0 (4)	C1—N2—Fe1	127.2 (4)
N2—C8—H8A	110.5	C8—N2—Fe1	113.0 (3)
C9—C8—H8A	110.5	C3—O1—Fe1	132.5 (3)
N2—C8—H8B	110.5	C12—O2—Fe1	133.4 (3)
C9—C8—H8B	110.5	Fe1 ⁱ —O3—Fe1	139.4 (3)
H8A—C8—H8B	108.7		

Symmetry codes: (i) $-x, y, -z+1/2$.

Fig. 1

