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μ-Oxo-bis[N,N'-ethylenebis(2-thioacetophenonidenimine)iron(III)]†

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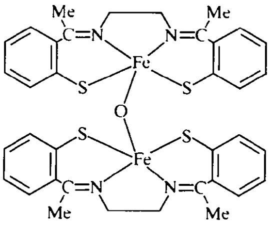
Abstract

The dinuclear title compound, $[Fe_2O(C_{18}H_{18}N_2S_2)_2]$ or $[Fe(EBTA)]_2O$, consists of two $[Fe(EBTA)]^+$ fragments connected by an oxo bridge, with distances Fe1—O1 1.773 (4) and Fe2—O1 1.763 (4) Å, and a bond angle Fe1—O1—Fe2 of 160.6 (2)°. The two iron(III) centres exhibit square-pyramidal coordination, with the basal plane formed by the two N and two S atoms of the $EBTA^{2-}$ ligand. The Fe atoms are located 0.69 (3) Å above the pyramid base. Bond lengths to the donor atoms lie in the range 2.112 (5)–2.215 (5) Å for Fe—N and in the range 2.307 (2)–2.357 (2) Å for Fe—S.

Comment

$[Fe(EBTA)]_2O$ [$EBTA^{2-}$ is N,N'-ethylenebis(2-thioacetophenonidenimine)], (I), is obtained by oxidation of Fe(EBTA) with molecular oxygen; Fe(EBTA) results from a template reaction with ethylenediamine, 2-thioacetophenone and anhydrous iron(II) acetate. The structure and the labelling scheme of $[Fe(EBTA)]_2O$ are shown in Fig. 1. In the dinuclear iron(III) complex, two $[Fe(EBTA)]^+$ fragments are connected by an almost symmetrical oxo bridge, with distances Fe1—O1 1.773 (4) and Fe2—O1 1.763 (4) Å, and a bond angle Fe1—O1—Fe2 of 160.6 (2)°.



(I)

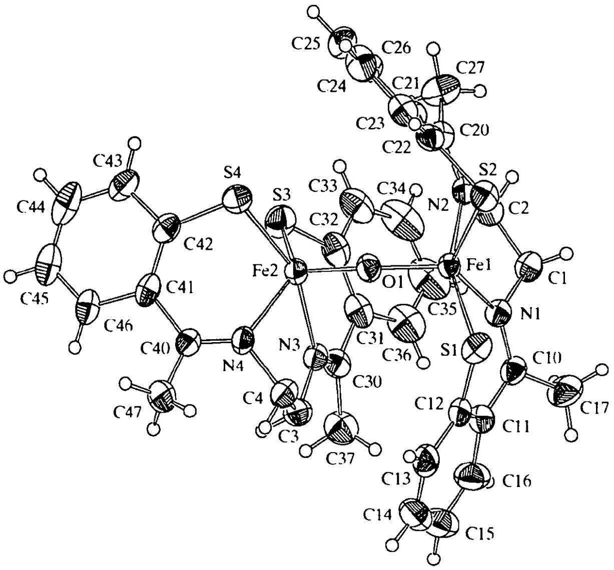


Fig. 1. *ZORTEP* (Zsolnai, 1994) plot of $[Fe(EBTA)]_2O$ with the atomic numbering scheme and 50% probability ellipsoids.

The Fe atoms have a fivefold coordination in the form of a square pyramid, with the oxo bridge in the axial position and the donor atoms of the $EBTA^{2-}$ ligand forming the pyramid base. The latter is slightly folded along its diagonal, so that the N and S atoms are alternately 0.26 (3) Å above and below the base. The Fe atoms are located 0.69 (3) Å above the pyramid base. The six-membered chelate rings containing one N and one S atom have a twist conformation, whereas the five-membered chelate rings containing the ethylene bridge form an envelope conformation with atom C2 0.45 (8) Å out of the N2—Fe1—N1—C1 plane and atom C4 0.78 (7) Å out of the N4—Fe2—N3—C3 plane. The two $EBTA^{2-}$ ligands are rotated with respect to each other by 103 (3)°. They are considerably folded. The two aromatic C_6 rings of the fragment Fe1(EBTA) form an angle of 69.4 (2)°, while the corresponding value of the $EBTA^{2-}$ ligand bound to Fe2 is 61.2 (2)°.

The Fe—S distances lie in the range 2.307 (2)–2.357 (2) Å. For the Fe—N distances, a slightly larger range of 2.112 (5)–2.215 (5) Å was found. In both cases, however, the values are in agreement with the corresponding distances in oxo-bridged iron(III) salen and thiosalen complexes (Coggon *et al.*, 1971; Gerloch *et al.*, 1969; Marini *et al.*, 1983; Ashmawy *et al.*, 1991).

Experimental

2-Thioacetophenone (1.38 g, 9.1 mmol; Coombes & Fenton, 1983) and iron(II) acetate (0.79 g, 4.55 mmol) were heated for

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† Systematic name: μ-oxo-bis{{2,2'-[1,2-ethanediylbis(nitriloethylidyne)]dibenzenethiolato-N,N',S,S'}iron(III)}.

15 min in boiling ethanol (50 ml). Ethylenediamine (0.3 ml) was then added and heating continued for 2 h. The black precipitate was filtered off, dissolved in CH_2Cl_2 and covered with a layer of hexane. Upon exposure to air at 293 K, brownish crystals of $[Fe(EBTA)]_2O$ were obtained.

Crystal data

$[Fe_2O(C_{18}H_{18}N_2S_2)_2]$
$M_r = 780.63$
Monoclinic
$P2_1/n$
$a = 11.663\,(4)$ Å
$b = 25.704\,(5)$ Å
$c = 12.070\,(4)$ Å
$\beta = 103.856\,(14)°$
$V = 3513.2\,(17)$ Å^3
$Z = 4$
$D_x = 1.476$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 18.5–25.2°$
$\mu = 9.124$ mm^{-1}
$T = 223\,(2)$ K
Plate
0.30 × 0.15 × 0.07 mm
Brown

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: ψ scan (Enraf–Nonius, 1985)
$T_{min} = 0.443$, $T_{max} = 0.528$
8821 measured reflections
5950 independent reflections
4395 reflections with $F > 2\sigma(F)$

$R_{int} = 0.067$
$\theta_{max} = 65°$
$h = -3 \rightarrow 13$
$k = 0 \rightarrow 30$
$l = -14 \rightarrow 14$
3 standard reflections every 100 reflections frequency: 60 min intensity decay: 2.0%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.151$
$S = 0.854$
5948 reflections
424 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.1131P)^2 + 13.2629P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.508$ e Å^{-3}
$\Delta\rho_{min} = -0.845$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Fe1—S1	2.350 (2)	N1—C1	1.474 (7)
Fe1—S2	2.341 (2)	C1—C2	1.508 (8)
Fe1—N1	2.161 (4)	N2—C2	1.466 (7)
Fe1—N2	2.129 (5)	N1—C10	1.295 (7)
Fe1—O1	1.773 (4)	N2—C20	1.294 (7)
Fe2—S3	2.307 (2)	N3—C3	1.478 (7)
Fe2—S4	2.357 (2)	C3—C4	1.520 (8)
Fe2—N3	2.215 (5)	N4—C4	1.477 (7)
Fe2—N4	2.112 (5)	N3—C30	1.289 (7)
Fe2—O1	1.763 (4)	N4—C40	1.283 (7)
S1—Fe1—S2	88.47 (6)	S3—Fe2—N3	88.20 (13)
S1—Fe1—N1	87.32 (13)	S3—Fe2—N4	129.06 (13)
S2—Fe1—N2	84.64 (12)	N3—Fe2—N4	75.7 (2)
N1—Fe1—N2	79.4 (2)	S3—Fe2—O1	117.74 (14)
S1—Fe1—O1	106.63 (14)	S4—Fe2—O1	105.94 (13)
S2—Fe1—O1	113.40 (13)	N3—Fe2—O1	93.3 (2)
N1—Fe1—O1	116.5 (2)	N4—Fe2—O1	111.3 (2)
N2—Fe1—O1	97.1 (2)	Fe2—O1—Fe1	160.6 (2)
S3—Fe2—S4	93.08 (7)		

Methyl H atoms were located from difference syntheses, idealized and refined as rigid groups allowed to rotate but not to tip; other H atoms were calculated at ideal positions and refined using a riding model. Computations were performed on a DEC VAX Station 3100, a CONVEX C3860 and a PC Pentium 120.

Data collection: *SDP* (Enraf–Nonius, 1985). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984) in *SDP*. Data reduction: *SDP*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1316). Services for accessing these data are described at the back of the journal.

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Bis(μ-3,7-diethyl-3,7-diazanonane-1,9-dithiolato)dimanganese(II)

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Abstract

The electroneutral title complex, bis(μ-3,7-diethyl-3,7-diazanonane-1,9-dithiolato-*S*,*N*,*N*′,*S*′:*S*′)dimanganese(II), $[Mn_2(C_{11}H_{24}N_2S_2)_2]$, is a dimercapto-bridged dimer