fine structure: *SHELXL*93 (Sheldrick, in preparation). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93 and local programs.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71611 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AL1072]

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Synthesis and Structure of the New Complex $Et_3NH[Fe(tben)]$

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Abstract

Triethylammonium (2,2′,2″,2‴-{1,2-ethanediylbis[nitrilobis(methylene)]}tetraphenolato-*N*,*N*′,*O*,*O*′,*O*″,*O*‴)-ferrate(III), $[NH(C_2H_5)_3][Fe(C_{30}H_{28}N_2O_4)]$, has been synthesized and its structure determined by X-ray diffraction. The anion, $[Fe(tben)]^-$, shows pseudo-octahedral geometry in which the hexadentate ligand is coordinated to the high-spin ferric center by four phenolate O and two N donor atoms in facial NO_2 arrangements. The O(1)—Fe—O(2) and the N(1)—Fe—N(2) bond angles of 103.6 (1) and 80.7 (1)°, respectively, deviate significantly from 90° as a result of the large bite angle required by the

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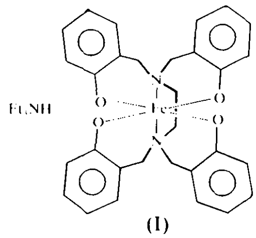
six-membered chelate rings for the equatorial configuration.

Comment

The coordination of tyrosine to Fe centers in proteins such as transferrins and purple acid phosphatases is a structural feature recently found for these metalloenzymes (Que, 1983). Herein we report the synthesis and crystal structure of a new Fe^{III} complex (I) as a model for these metalloenzymes.



(I)

The Fe—O(phenolate) bonds [average 1.923 (3) Å] that are in the equatorial plane containing the Fe and N atoms are significantly shorter than the other two [average 2.004 (4) Å], which are in mutually *trans* positions. This reflects the weak *trans* effect of the shorter Fe—O(phenolate) bonds, which also accounts for the longer Fe—N(amine) bonds [average 2.235 (4) Å]. On the other hand, the Fe—O(phenolate) bond distances *trans* to the ethylenediamine N atoms are comparable to the corresponding bond lengths observed in the $[Fe^{III}(hbed)]^-$ ion [average 1.887 (6) Å; hbed = *N*,*N*′-bis(2-hydroxybenzyl)ethylenediamine-*N*,*N*′-diacetate (Larsen, Jenkins, Memon & Lauffer, 1990)]. Magnetic susceptibility data for the complex in the temperature range 4.6-269 K fit the Curie Law with μ_{eff} = 5.48 BM and are in good agreement with the spin-only value of 5.92 BM for a high-spin Fe^{III} ion.

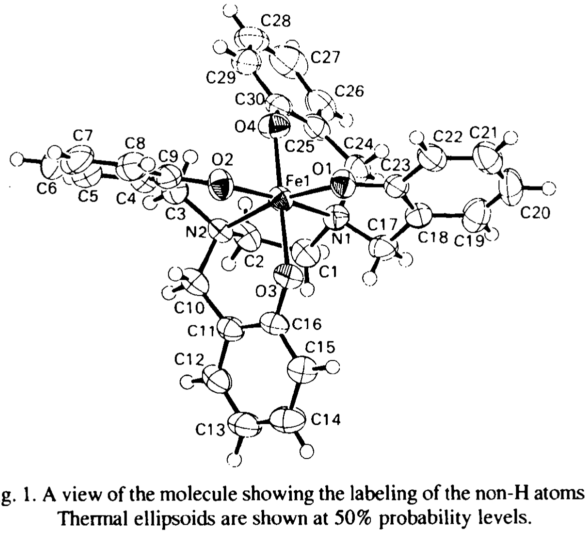


Fig. 1. A view of the molecule showing the labeling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels.

The five-membered ring formed by the ethylenediamine 'backbone' (FeN_2C_2) has the usual skew conformation with the torsion angle N(1)—C(1)—C(2)—N(2) = −52.0 (6)°. There is a linear hydrogen bond between the H(N) of the cation and O(3). The N···O distance is 2.910 (6) Å and the N—H···O angle is approximately 171°.

The triethylammonium cation is disordered. For one of the ethyl groups, it was possible to locate two independent positions for each of the C atoms. This was not feasible for the other two groups, although it is apparent from their anisotropic displacement parameters that they are also disordered.

Experimental

Reaction of tetrakis(2-hydroxybenzyl)ethylenediamine (H_4-tben) (Neves, Ceccato, Vencato, Mascarenhas & Erasmus-Buhr, 1992) with $FeCl_3.xH_2O$ in CH_3OH in the presence of Et_3N affords crystals of the title complex suitable for X-ray diffraction studies.

Crystal data

$[NH(C_2H_5)_3][Fe(C_{30}H_{28}N_2O_4)]$
M_r = 638.6
Monoclinic
$P2_1/n$
a = 14.053 (3) Å
b = 10.781 (5) Å
c = 21.406 (5) Å
β = 102.84 (2)°
V = 3161 (1) Å^3
Z = 4
D_x = 1.341 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 21 reflections
θ = 4.75-9.45°
μ = 0.521 mm^{-1}
T = 294 K
Prismatic
0.20 × 0.20 × 0.20 mm
Burgundy

Data collection

Rigaku AFC-7*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
6135 measured reflections
5885 independent reflections
2625 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.037
θ_{max} = 25°
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 12$
$l = -25 \rightarrow 24$
3 standard reflections
frequency: 150 min
intensity variation: −3.5%

Refinement

Refinement on F
R = 0.047
wR = 0.058
S = 1.31
2625 reflections
396 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: empirical (*TEXSAN*; Molecular Structure Corporation, 1985, 1992)
Extinction coefficient: 1.149 (1) × 10^{-7}
Atomic scattering factors from Cromer & Waber (1974)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Fe	0.46689 (5)	0.02001 (7)	0.21446 (4)	3.11 (3)
O(1)	0.4836 (3)	0.1002 (3)	0.2961 (2)	3.8 (2)
O(2)	0.3271 (2)	0.0079 (3)	0.1853 (2)	4.1 (2)
O(3)	0.4719 (2)	−0.1427 (3)	0.2647 (2)	3.5 (2)
O(4)	0.4668 (3)	0.1736 (3)	0.1656 (2)	4.0 (2)
N(1)	0.6296 (3)	0.0190 (4)	0.2268 (2)	3.3 (2)
N(2)	0.4782 (3)	−0.0957 (4)	0.1302 (2)	3.3 (2)
N(3)	0.6634 (5)	0.1029 (7)	0.6522 (3)	7.9 (4)
C(1)	0.6526 (4)	−0.0838 (5)	0.1855 (3)	3.8 (3)
C(2)	0.5803 (4)	−0.0912 (5)	0.1223 (3)	3.8 (3)
C(3)	0.4117 (4)	−0.0407 (5)	0.0722 (3)	4.2 (3)
C(4)	0.3069 (4)	−0.0401 (5)	0.0727 (3)	4.1 (3)
C(5)	0.2403 (5)	−0.0682 (6)	0.0160 (3)	5.2 (3)
C(6)	0.1415 (6)	−0.0681 (7)	0.0116 (4)	6.4 (4)
C(7)	0.1055 (4)	−0.0420 (7)	0.0640 (4)	6.0 (3)
C(8)	0.1677 (4)	−0.0162 (6)	0.1216 (3)	4.9 (3)
C(9)	0.2705 (4)	−0.0139 (5)	0.1281 (3)	3.9 (2)
C(10)	0.4436 (4)	−0.2262 (5)	0.1351 (3)	4.0 (3)
C(11)	0.4935 (4)	−0.3022 (5)	0.1911 (3)	3.5 (2)
C(12)	0.5209 (4)	−0.4239 (6)	0.1828 (3)	4.3 (3)
C(13)	0.5541 (4)	−0.5024 (5)	0.2339 (3)	4.9 (3)
C(14)	0.5610 (4)	−0.4586 (6)	0.2953 (3)	4.9 (3)
C(15)	0.5357 (4)	−0.3383 (6)	0.3051 (3)	4.1 (3)
C(16)	0.4997 (4)	−0.2576 (5)	0.2543 (3)	3.3 (2)
C(17)	0.6719 (4)	−0.0119 (5)	0.2949 (3)	3.8 (2)
C(18)	0.6547 (4)	0.0856 (5)	0.3418 (2)	3.6 (2)
C(19)	0.7336 (4)	0.1285 (6)	0.3896 (3)	5.0 (3)
C(20)	0.7217 (5)	0.2160 (7)	0.4346 (3)	5.6 (3)
C(21)	0.6294 (5)	0.2643 (6)	0.4317 (3)	5.0 (3)
C(22)	0.5511 (4)	0.2239 (5)	0.3851 (3)	4.2 (3)
C(23)	0.5618 (4)	0.1352 (5)	0.3398 (2)	3.2 (2)
C(24)	0.6706 (4)	0.1416 (5)	0.2123 (3)	3.6 (3)
C(25)	0.6265 (4)	0.2006 (5)	0.1489 (2)	3.7 (3)
C(26)	0.6874 (4)	0.2500 (6)	0.1120 (3)	4.6 (3)
C(27)	0.6487 (7)	0.3195 (6)	0.0584 (3)	6.0 (4)
C(28)	0.5504 (7)	0.3398 (6)	0.0409 (3)	5.9 (4)
C(29)	0.4882 (5)	0.2899 (5)	0.0765 (3)	4.6 (3)
C(30)	0.5255 (4)	0.2198 (5)	0.1312 (3)	3.7 (3)
C(31)	0.610 (1)	0.1786 (8)	0.5960 (6)	10.9 (7)
C(32)	0.512 (1)	0.120 (1)	0.5687 (4)	10.5 (6)
C(33)	0.6813 (6)	−0.0250 (8)	0.6364 (4)	7.7 (5)
C(34)	0.7422 (7)	−0.0395 (9)	0.5858 (5)	9.7 (5)
C(35*a*)	0.740 (1)	0.207 (1)	0.6738 (7)	5.9 (3)
C(35)	0.783 (1)	0.125 (1)	0.6994 (7)	5.4 (3)
C(36*a*)	0.765 (1)	0.233 (1)	0.7437 (7)	5.8 (3)
C(36)	0.798 (1)	0.177 (2)	0.7365 (9)	7.4 (4)

Table 2. *Selected geometric parameters* (Å, °)

Fe—O(1)	1.917 (3)	Fe—N(2)	2.227 (4)
Fe—O(2)	1.930 (3)	N(1)—C(1)	1.496 (6)
Fe—O(3)	2.050 (4)	N(2)—C(2)	1.481 (6)
Fe—O(4)	1.958 (4)	C(1)—C(2)	1.502 (7)
Fe—N(1)	2.243 (4)		
O(1)—Fe—O(2)	103.6 (1)	O(2)—Fe—N(2)	87.2 (2)
O(1)—Fe—O(3)	85.7 (1)	O(3)—Fe—O(4)	177.8 (1)
O(1)—Fe—O(4)	95.2 (2)	O(3)—Fe—N(1)	91.1 (1)
O(1)—Fe—N(1)	88.9 (1)	O(3)—Fe—N(2)	86.9 (1)
O(1)—Fe—N(2)	167.0 (1)	O(4)—Fe—N(1)	86.9 (2)
O(2)—Fe—O(3)	91.5 (1)	O(4)—Fe—N(2)	91.9 (2)
O(2)—Fe—O(4)	90.3 (2)	N(1)—Fe—N(2)	80.7 (1)
O(2)—Fe—N(1)	167.4 (1)		

The asymmetric unit contains one [Fe^{III}(tben)]$^-$ anion and one triethylammonium cation. With the exception of the disordered C atoms [C(35), C(35*a*), C(36) and C(36*a*)], all non-H atoms were refined anisotropically. H atoms on the anion were included in idealized positions. For the cation, H atoms on resolved disordered atoms were omitted. Data collection: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Cell refinement: *TEXSAN*. Data reduction: *TEXSAN*. Program(s) used to solve structure: *SAPI91* (Fan Hai-Fu, 1991). Program(s) used to refine structure: *DIRDIF92* (Beurskens *et al.*, 1992). Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71640 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST1080]

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Cd(alloxanthine)$_2$(NO$_3$)$_2$(H$_2$O)$_2$

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Abstract

The title complex, diaquadinitratobis{[1*H*]pyrazolo-[3,4-*d*]pyrimidine-4,6(5*H*,7*H*)-dione-N^1}cadmium-(II), was prepared as part of a study of the coordinating properties of the xanthine oxidase inhibiting molecule, alloxanthine. [Cd(NO$_3$)$_2$(C$_5$H$_4$-

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