Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Hexakis(1-oxido-1*H*-benzotriazol-3-ium-*κO*)-iron(III) tris(perchlorate) acetonitrile disolvate

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In the title mononuclear complex, $[Fe(C_6H_5N_3O)_6](ClO_4)_3\cdot 2C_2H_3N$, the Fe^{III} ion shows a close-to-ideal octahedral geometry coordinated by six O atoms from six 1-hydroxybenzotriazole ligands. The ligand benzotriazole exists as a zwitterion.

Received 9 May 2005
Accepted 12 August 2005
Online 17 August 2005

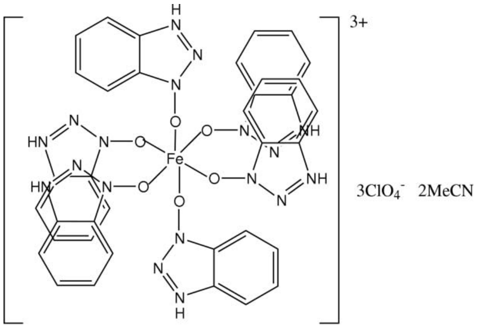
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.114
Data-to-parameter ratio = 16.9

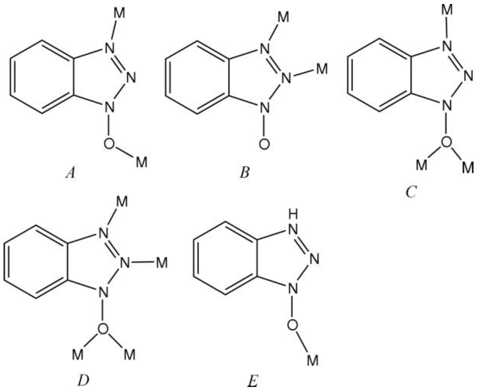
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

1-Hydroxybenzotriazole with a substituted donor group on the azole ring has recently been used to synthesize polymeric and polynuclear coordination complexes (Tangoulis *et al.*, 2000; Diamantopoulou, Perlepes *et al.*, 2002; Papaefstathiou *et al.*, 2002; Diamantopoulou, Raptopoulou *et al.*, 2002). There are three potential donor atoms in 1-hydroxybenzotriazole, resulting in coordination flexibility. 1-Hydroxybenzotriazole exists in a deprotonated form in reported complexes, coordinated to the metal atoms in a μ_2–, μ_3– and/or μ_4-fashion, as shown in the scheme.



(I)



In this paper, we report a new complex, (I), which crystallizes in the hexagonal space group $P6_3/m$. The Fe^{III} ion shows close-to-ideal octahedral geommetry, coordinated by six O atoms from six 1-hydroxybenzotriazole ligands. As required by the crystallographic symmetry of the Fe site, all six Fe—O

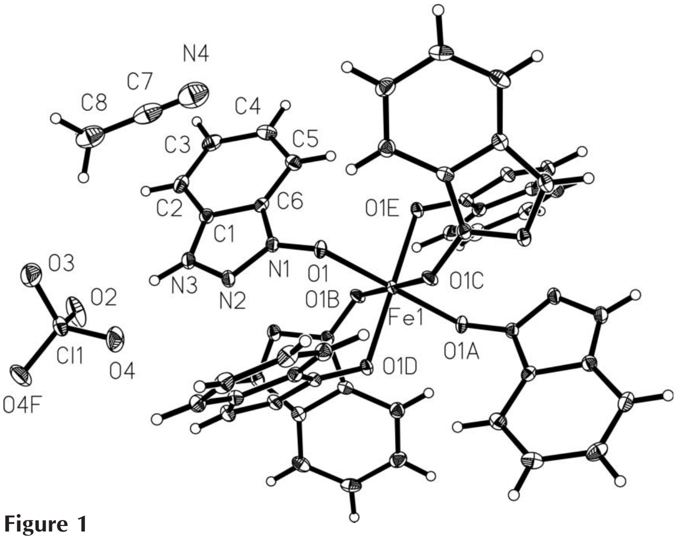


Figure 1
The coordination environment of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii. [Symmetry codes: (A) $-x, -y, -z$; (B) $x - y, x, -z$; (C) $-x + y, -x, z$; (D) $y, -x + y, -z$; (E) $-y, x - y, z$; (F) $x, y, -z + 1/2$.]

bond lengths are identical at 2.0095 (14) Å. The three *trans*-O—Fe—O angles are 180°. However, the *cis*-O—Fe—O angles deviate from ideal octahedral values [87.30 (6) and 92.70 (6)°]. The ligand 1-hydroxybenzotriazole exists as a zwitterion as a result of tautomerism. Atom N3 forms hydrogen bonds with perchlorate atoms O2 and O4, with N···O distances of 2.8912 (19) and 3.139 (3) Å, respectively.

Experimental

Iron(III) perchlorate (0.077 g, 0.5 mmol), 1-hydroxybenzotriazole (0.135 g, 1 mmol) and acetonitrile (7 ml) were stirred in a beaker at 313 K for 30 min. The resulting clear solution was filtered and allowed to stand for 3 d. The resulting colorless regular hexagonal block-shaped crystals were recovered by filtration in 62% yield. Analysis found: C 38.46, H 2.96, N 22.37%; calculated for $C_{40}H_{36}Cl_3FeN_{20}O_{18}$: C 38.53, H 2.91, N 22.46%.

Crystal data

$[Fe(C_6H_5N_3O)_6](ClO_4)_3 \cdot 2C_2H_3N$
$M_r = 1247.09$
Hexagonal, $P6_3/m$
$a = 10.2197$ (4) Å
$c = 28.3118$ (14) Å
$V = 2560.79$ (19) Å^3
$Z = 2$
$D_x = 1.617$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 164 reflections
$\theta = 2.5$–26°
$\mu = 0.55$ mm^{-1}
$T = 293$ (2) K
Hexagonal block, colorless
0.32 × 0.30 × 0.18 mm

Data collection

Bruker APEX area-dector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.845$, $T_{max} = 0.908$
2319 measured reflections
2319 independent reflections
1935 reflections with $I > 2\sigma(I)$
$\theta_{max} = 29.0°$
$h = -11 \rightarrow 0$
$k = -12 \rightarrow 13$
$l = -34 \rightarrow 38$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.114$
$S = 1.14$
2319 reflections
137 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 5.4288P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.18$ e Å^{-3}
$\Delta\rho_{min} = -0.75$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O1	2.0095 (14)		
O1^{i}—Fe1—O1	180.00 (10)	O1—Fe1—O1^{ii}	87.30 (6)
O1^{i}—Fe1—O1^{ii}	92.70 (6)		

Symmetry code: (i) $-x, -y, -z$; (ii) $x - y, x, -z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*B*···O2	0.92 (3)	1.97 (3)	2.8912 (19)	171 (3)
N3—H3*B*···O4	0.92 (3)	2.57 (3)	3.139 (3)	121 (2)

The H atoms of the benzene rings were placed at calculated positions (C—H = 0.93–0.96 Å) and refined in the riding model, with U_{iso}(H) = 1.2 times the U_{eq}(parent atom). The H atoms of acetronitrile and atom H3*B* (attached to N3) were located in a Fourier map and refined isotropically. The highest peak was located 0.94 Å from atom Cl1.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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