Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Aqua[*N*,*N*′-bis(2-oxidophenylmethyleneimino)-1,3-diaminopropane](thiocyanato)iron(III)

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Received 6 May 2004
Accepted 11 May 2004
Online 15 May 2004

The title compound (systematic name: aqua{2,2′-[propane-1,3-diylbis(nitrilomethylidene)]diphenolato}(thiocyanato)iron(III)), [Fe($C_{17}H_{16}N_2O_2$)(NCS)(H_2O)], has been synthesized and characterized. It is a mononuclear compound. The Fe^{III} atom has a slightly distorted octahedral geometry, coordinated by two N and two O donors of *L* [*L* is the *N*,*N*′-bis(2-hydroxyphenylmethyleneimino)-1,3-diaminopropane anion] and one N atom from a thiocyanate anion, together with a water molecule. In the crystal structure, the molecules are held together by intermolecular O—H···O hydrogen bonds, forming chains extending in the *b* direction.

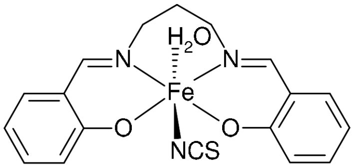
Key indicators

Single-crystal X-ray study
$T = 298$ K
Mean σ(C–C) = 0.011 Å
R factor = 0.058
wR factor = 0.137
Data-to-parameter ratio = 13.4

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

Comment

Recently, we have reported a few Schiff base complexes (You, Lin *et al.*, 2003; You, Qu *et al.*, 2003; You, Xiong *et al.*, 2004; You, Zhu & Liu, 2004). As an extension of our work on the structural characterization of Schiff base complexes, the title mononuclear iron(III) complex, (I), is reported here.



(I)

Compound (I) is an electronically neutral mononuclear iron(III) compound (Fig. 1). The Fe^{III} atom has a slightly distorted octahedral geometry and is coordinated by one Schiff base ligand *L* [*L* is the *N*,*N*′-bis(2-hydroxyphenylmethyleneimino)-1,3-diaminopropane anion], one thiocyanate anion and one water molecule. The Schiff base ligand *L* acts as

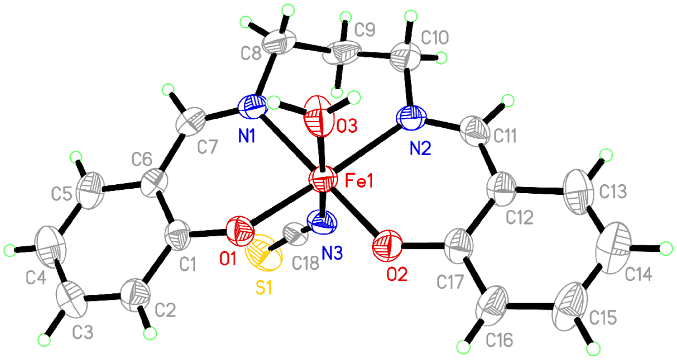


Figure 1
The structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

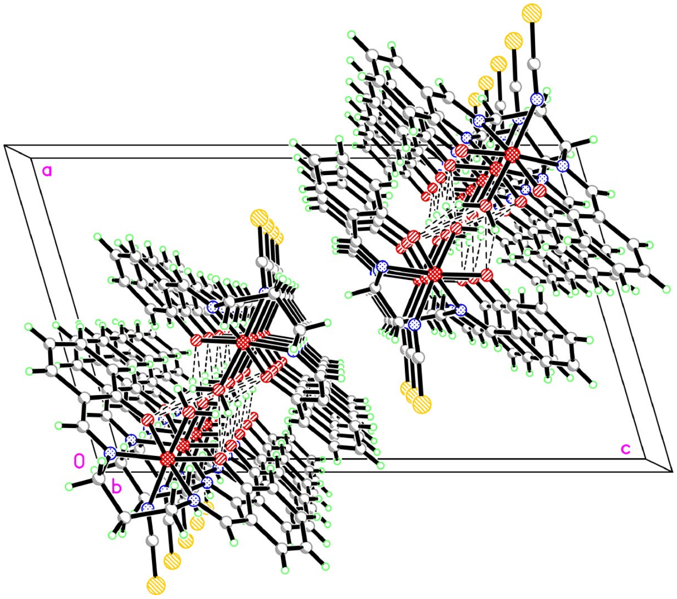


Figure 2
The crystal packing of (I), viewed along the *b* axis. Dashed lines indicate hydrogen bonds.

a tetradentate ligand and coordinates to atom Fe1 through two phenolate O atoms and two imine N atoms. The thiocyanate anion is a monodentate ligand and coordinates to the metal *via* the terminal N atom.

The three *trans* angles at the Fe^{III} atom are close to 180° (Table 1). The other angles subtended at the Fe^{III} atom are close to 90°, varying from 85.2 (3) to 97.4 (3)°, which indicates a slightly distorted octahedral geometry of the Fe^{III} atom. The average Fe—O(phenolate) bond length of 1.942 (6) Å is much longer than the average value of 1.880 (2) Å observed in another Schiff base iron(III) complex (Elmali *et al.*, 2000). The average Fe—N(imine) bond distance of 2.138 (7) Å is also much longer than the average value of 2.082 (2) Å observed in the same complex. The thiocyanate N—C—S angle of 176.6 (6)° in (I) is comparable with the value of 178.7 (4)° observed in a Schiff base nickel complex, recently reported by us (Zhu *et al.*, 2003). The dihedral angle between the two benzene rings is 8.7 (3)°. The conformation of the six-membered ring containing the Fe atom, the azomethine N atoms and the three C atoms of the connecting 1,3-diaminopropane is an asymmetric boat.

In the crystal structure of (I), the molecules are connected by intermolecular O—H···O hydrogen bonds (Fig. 2 and Table 2), forming chains extending in the *b* direction.

Experimental

Salicylaldehyde and 1,3-diaminopropane were available commercially and were used without further purification. Salicylaldehyde (0.2 mmol, 24.2 mg) and 1,3-diaminopropane (0.1 mmol, 7.4 mg) were dissolved in methanol (10 ml). The mixture was stirred for 1 h to give a clear yellow solution of H_2L (0.1 mmol), where H_2L is *N*,*N*′-bis(2-hydroxyphenylmethenylimino)-1,3-diaminopropane. To this solution of H_2L was added a methanol solution (10 ml) of $FeCl_3 \cdot 6H_2O$ (0.1 mmol, 270.3 mg) and a methanol solution (5 ml) of ammonium thiocyanate (0.1 mmol, 7.6 mg), with stirring. After keeping the resulting solution at room temperature in air for 18 d, brown 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 81.3%). Analysis found: C 52.1, H 4.6, N 9.9%; calculated for $C_{18}H_{18}FeN_3O_3S$: C 52.3, H 4.5, N 10.1%.

Crystal data

$[Fe(C_{17}H_{16}N_2O_2)(NCS)(H_2O)]$
$M_r = 412.26$
Monoclinic, $P2_1/n$
$a = 12.82$ (5) Å
$b = 6.96$ (2) Å
$c = 21.49$ (7) Å
$\beta = 106.51$ (6)°
$V = 1838$ (11) Å^3
$Z = 4$

$D_x = 1.490$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1105 reflections
$\theta = 3.1$–19.0°
$\mu = 0.96$ mm^{-1}
$T = 298$ (2) K
Block, brown
0.23 × 0.14 × 0.05 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.810$, $T_{max} = 0.954$
9280 measured reflections

3246 independent reflections
1452 reflections with $I > 2\sigma(I)$
$R_{int} = 0.108$
$\theta_{max} = 25.0°$
$h = -13 \rightarrow 15$
$k = -6 \rightarrow 8$
$l = -25 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.137$
$S = 0.85$
3246 reflections
243 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.72$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Fe1—O2	1.926 (6)	Fe1—O3	2.088 (6)
Fe1—O1	1.958 (6)	Fe1—N1	2.128 (7)
Fe1—N3	2.034 (7)	Fe1—N2	2.148 (7)
O2—Fe1—O1	91.6 (2)	N3—Fe1—N1	87.1 (3)
O2—Fe1—N3	97.4 (3)	O3—Fe1—N1	85.2 (3)
O1—Fe1—N3	94.5 (3)	O2—Fe1—N2	87.7 (3)
O2—Fe1—O3	90.3 (3)	O1—Fe1—N2	173.07 (17)
O1—Fe1—O3	87.8 (3)	N3—Fe1—N2	92.5 (3)
N3—Fe1—O3	171.9 (2)	O3—Fe1—N2	85.3 (3)
O2—Fe1—N1	175.13 (17)	N1—Fe1—N2	93.7 (3)
O1—Fe1—N1	86.4 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H17···O2^{i}	0.90 (1)	1.99 (3)	2.758 (9)	143 (4)
O3—H18···O1^{i}	0.90 (1)	1.86 (3)	2.663 (9)	148 (4)

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

The H atoms of the water molecules were located in a difference Fourier map and refined isotropically, with the O—H and H···H distances restrained to 0.89 (1) and 1.39 (1) Å, respectively. The other H atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, with C—H distances in the range 0.93–

0.97 Å, and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). An unassigned maximum residual density of 0.72 e Å^{-3} was observed 0.64 Å from atom H14*A*. The minimum residual density was observed 0.63 Å from atom Fe1. The ratio of observed to unique reflections is low (45%), and the value of $R_{\rm int}$ is greater than 0.10, probably due to the poor diffraction quality of the crystal.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); 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*.

The authors thank the Education Office of Anhui Province, People's Republic of China, for research grant No. 2004kj300zd.

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