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Key indicators

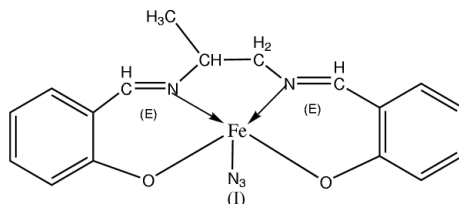
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.008\text{ \AA}$
Disorder in main residue
 R factor = 0.072
 wR factor = 0.225
Data-to-parameter ratio = 14.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Azido[bis(2-oxidophenylmethyleneimino)-
propane-1,2-diamine(2-)]iron(III)The title compound, $[\text{Fe}(\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_2)(\text{N}_3)]$, is mononuclear. The Fe^{III} atom is coordinated by four atoms from one Schiff base ligand and one N atom from an azide anion. In the crystal structure, the molecules are held together by hydrogen bonds, forming chains extending in the a direction.

Received 18 May 2004

Accepted 28 May 2004

Online 12 June 2004

Comment

Recently, we have reported a Schiff base iron(III) complex (You *et al.*, 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.

Compound (I) is neutral (Fig. 1). The Fe^{III} cation has a distorted square-pyramidal geometry, being coordinated by two N atoms and two O atoms from the Schiff base ligand L [L is the bis(2-hydroxyphenylmethyleneimino)-1,2-diaminopropane anion] and one N atom from an azide anion. The four coordinating atoms of L constitute the basal plane and the angles subtended at the Fe atom are close to 90° , varying from $81.73(19)$ to $94.59(13)^\circ$. Atom N3 of the azide anion occupies the apical position and the N3–Fe bond is approximately perpendicular to the basal plane, the angles involving N3 varying from $92.51(19)$ to $97.74(19)^\circ$.

The average Fe–O bond length of $1.89(2)\text{ \AA}$ is similar to the average value of $1.880(2)\text{ \AA}$ observed in another Schiff

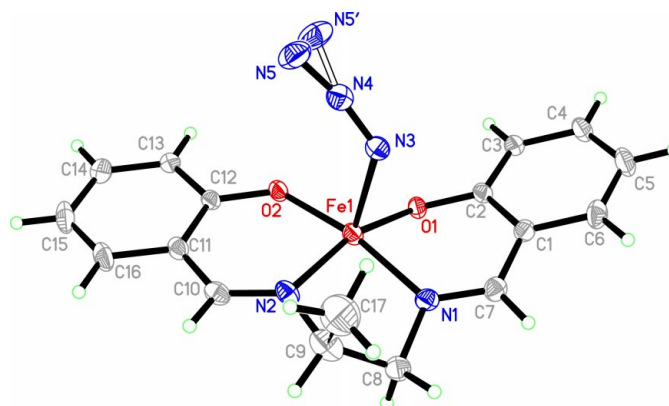


Figure 1

The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms are drawn as small spheres of arbitrary radii. Both disorder components of the azide ligand are shown.

base iron(III) complex (Elmali *et al.*, 2000). The average Fe–N(imine) bond distance of 1.985 (5) Å is also similar to the average value of 2.082 (2) Å observed in the same complex. The coordinating N atom in the azide anion in (I) is a little further from the Fe atom [2.134 (5) Å] than the average Fe–N(imine) bond distance in the structure.

In the crystal structure of (I), the molecules are connected by weak C–H...N and C–H...O hydrogen bonds involving the azide anion and the Schiff base ligand, forming chains extending along the *a* axis.

Experimental

1,2-Diaminopropane and salicylaldehyde were available commercially and were used without further purification. 1,2-Diaminopropane (0.2 mmol, 14.8 mg) and salicylaldehyde (0.4 mmol, 48.8 mg) were dissolved in ethanol (10 ml). The mixture was stirred for 2 h to obtain a clear orange solution of H₂L (0.2 mmol), where H₂L is bis(2-hydroxyphenylmethyleneimino)-1,2-diaminopropane. To this solution of H₂L was added an ethanol solution (10 ml) of Fe(NO₃)₃·9H₂O (0.2 mmol, 80.8 mg), and NaN₃ (0.2 mmol, 13 mg) in distilled water (4 ml), with stirring. After allowing the resulting solution to stand in air for 12 d, black crystals of (I) were formed on slow evaporation of the solvent. The crystals were isolated, washed with ethanol and dried in a vacuum desiccator using CaCl₂ (yield 68.8%). Elemental analysis: found: C 54.88, H 4.32, N 18.64%; calculated for C₁₇H₁₆FeN₅O₂: C 53.99, H 4.26, N 18.52%.

Crystal data

[Fe(C ₁₇ H ₁₆ N ₂ O ₂)(N ₃)]	$D_x = 1.497 \text{ Mg m}^{-3}$
$M_r = 378.20$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 3662 reflections
$a = 8.7388 (8) \text{ \AA}$	$\theta = 4\text{--}27^\circ$
$b = 13.7957 (13) \text{ \AA}$	$\mu = 0.92 \text{ mm}^{-1}$
$c = 14.4499 (14) \text{ \AA}$	$T = 298 (2) \text{ K}$
$\beta = 105.620 (2)^\circ$	Block, black
$V = 1677.7 (3) \text{ \AA}^3$	$0.18 \times 0.18 \times 0.10 \text{ mm}$
$Z = 4$	

Data collection

Siemens SMART CCD area-detector diffractometer	3528 independent reflections
φ and ω scans	2159 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.045$
$T_{\text{min}} = 0.852$, $T_{\text{max}} = 0.914$	$\theta_{\text{max}} = 27.0^\circ$
9418 measured reflections	$h = -5 \rightarrow 11$
	$k = -17 \rightarrow 17$
	$l = -18 \rightarrow 18$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1146P)^2 + 0.6771P]$
$R[F^2 > 2\sigma(F^2)] = 0.072$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.225$	$(\Delta/\sigma)_{\text{max}} = 0.009$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.86 \text{ e \AA}^{-3}$
3528 reflections	$\Delta\rho_{\text{min}} = -0.40 \text{ e \AA}^{-3}$
236 parameters	
H-atom parameters constrained	

Table 1

Hydrogen-bonding geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
C7–H7...N5 ⁱ	0.93	2.53	3.45 (4)	175
C10–H10...N3 ⁱⁱ	0.93	2.51	3.405 (6)	160
C13–H13...O2 ⁱⁱⁱ	0.93	2.56	3.313 (6)	139

Symmetry codes: (i) $x - 1, y, z$; (ii) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (iii) $1 - x, 2 - y, -z$.

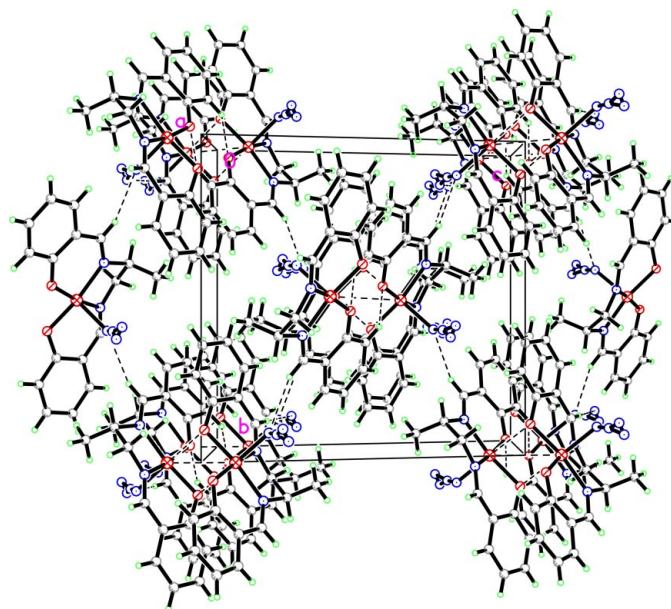


Figure 2

The crystal packing of (I), showing the hydrogen bonds (dashed lines) forming chains extending in the *a* direction.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C–H distances in the range 0.93–0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, apart from the H atoms on C17, for which $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C17})$. One of the N atoms of the azide anion was found to be disordered and was split into two alternative sites of 0.5 occupancy. The U_{iso} value of C17 is quite large, but it was not considered as a disordered atom.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); 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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