

{Bis[2-(2-oxido-1-naphthylmethylidene-amino)phenyl] disulfide}chloroiron(III)Ayhan Elmali,^{a*}† Yalcın Elerman^{a†} and Ingrid Svoboda^b^aDepartment of Engineering Physics, Faculty of Sciences, University of Ankara, 06100 Besevler, Ankara, Turkey, and ^bStrukturforschung, Fachbereich Materialwissenschaft, Technische Universität Darmstadt, Petersenstraße 23, D-64287 Darmstadt, Germany

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The crystal structure of the title compound, {bis[2-(2-oxido-2-naphthylideneamino)phenyl] disulfide- $\kappa^5 O, N, S, N', O'$ }chloroiron(III), [FeCl(C₃₄H₂₂N₂O₂S₂)], has been determined. The structure consists of monomeric iron(III) complexes with distorted octahedral coordination. The disulfide functions as a pentadentate ligand and the Fe^{III} atom is coordinated through two N, two O and one S atom, and one chloride ion. The distance between the second S atom and the Fe^{III} atom is a non-bonding 3.8473 (14) Å.

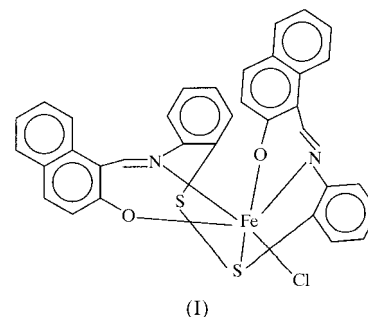
Comment

The complexes of transition metal ions with Schiff bases are a large and growing class of compounds of both stereochemical and magnetochemical interest (Garnovskii *et al.*, 1993). Characteristically, several distinct compounds may be isolated from one reaction mixture. The nature of the Schiff base complexes appears to be dependent on several factors which include the solvent system, steric constraints imposed by ligand side groups, crystal packing considerations and hybridization states defined by the metal atom. However, it has been observed that on rare occasions, some other factors, which include coordination preferences of the metal ion and specific intermolecular interactions, may also effect the result (Elmali *et al.*, 1992; Elmali, Atakol *et al.*, 1993a,b; Elmali, Elerman *et al.*, 1993a,b; Gerloch & Mabbs, 1967).

We have reported previously the crystal structures of several dimeric and monomeric Schiff base complexes of Fe^{III} (Elmali *et al.*, 1992, 1993, 1994a,b, 2000; Elmali, Atakol *et al.*, 1993a,b; Elmali, Elerman *et al.*, 1993a,b; Elerman *et al.*, 1997). As part of a general study of complexes of polydentate Schiff base ligands, we became interested in ligands containing sulfur donors. We report here the results of the reaction of iron(III) with the polydentate Schiff base ligand formed from 2-hydroxynaphthalene-1-carbaldehyde and 2,2'-diaminophenyl disulfide.

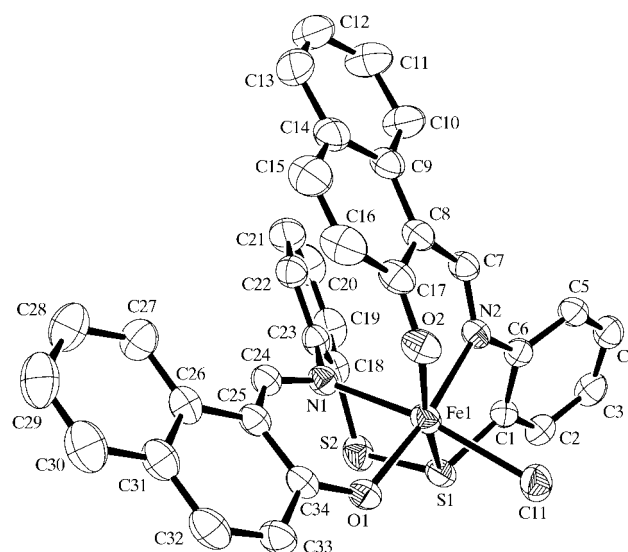
† Alexander von Humboldt fellow.

The structure of the title compound, (I), consists of monomeric iron(III) complexes with distorted octahedral coordination (Fig. 1). The Fe^{III} ion is coordinated by the two



phenolic O atoms, the two imine N atoms, one S and one Cl atom. In order to achieve this coordination, the ligands form three six-membered chelate rings and one five-membered chelate ring. Two of the six-membered rings involve phenolic O and imine N atoms, and for one of these rings the imine N atom (N2) is also included in the five-membered ring and the phenolic O atom (O2) is *trans* to the S atom (S1), which is involved in the five-membered ring. The third six-membered ring includes both the uncoordinated and coordinated S atoms, and the second imine N atom. The distance between S2 and Fe1 is 3.8473 (14) Å, and S2 is not considered to be coordinated.

All the O—Fe—O, O—Fe—Cl, O—Fe—N, O—Fe—S, N—Fe—S and Cl—Fe—S angles deviate from 90° and the arrangement is 'axial'. The N1—Fe—Cl1 angle is found to be only 164.39 (5)°, instead of 180°. The two Fe—O bond distances are only slightly different and are similar to those found in other structures (Davies & Gatehouse, 1970; Gerloch *et al.*, 1967; Bertrand & Breece, 1974). The two Fe—N bond distances show a similar difference and are slightly longer than

**Figure 1**

The molecular structure (ORTEP-3; Farrugia, 1997) of the title compound showing the atom-labelling scheme and displacement ellipsoids at the 50% probability level. All H atoms have been omitted for clarity.

the value of 2.10 Å found in other structures (Gerloch & Mabbs, 1967; Davies & Gatehouse, 1970; Elmali, Atakol *et al.*, 1993a; Elerman *et al.*, 1997). The S··S distance of 2.0580 (10) Å is in good agreement with the values found in other structures (Riley & Seff, 1972; Bonds & Ibers, 1972; Bertrand & Breece, 1974). The magnetic moment of the compound, 5.93 BM (or μ_B), is normal for high-spin iron(III).

Experimental

The title compound was synthesized according to the well established method of Bertrand & Breece (1974).

Crystal data

[FeCl(C ₃₄ H ₂₂ N ₂ O ₂ S ₂)]	Mo K α radiation
$M_r = 645.96$	Cell parameters from 25 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 2.96\text{--}13.87^\circ$
$a = 8.558$ (3) Å	$\mu = 0.827$ mm ⁻¹
$b = 12.989$ (3) Å	$T = 293$ (2) K
$c = 24.991$ (5) Å	Plate, dark red
$V = 2778.0$ (13) Å ³	$0.55 \times 0.40 \times 0.08$ mm
$Z = 4$	
$D_x = 1.544$ Mg m ⁻³	

Data collection

Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.018$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 25.97^\circ$
Absorption correction: empirical via ψ scans (North <i>et al.</i> , 1968)	$h = -10 \rightarrow 7$
$T_{\text{min}} = 0.593$, $T_{\text{max}} = 0.774$	$k = -16 \rightarrow 1$
5939 measured reflections	$l = -30 \rightarrow 1$
5069 independent reflections	3 standard reflections
4728 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: 0.02%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0392P)^2 + 0.4861P]$
$R[F^2 > 2\sigma(F^2)] = 0.024$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.067$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.078$	$\Delta\rho_{\text{max}} = 0.27$ e Å ⁻³
5069 reflections	$\Delta\rho_{\text{min}} = -0.24$ e Å ⁻³
379 parameters	Absolute structure: Flack (1983)
H-atom parameters constrained	Flack parameter = -0.012 (11)

Table 1

Selected geometric parameters (Å, °).

N1—Fe1	2.1834 (17)	S1—S2	2.0580 (10)
N2—Fe1	2.1396 (16)	S1—Fe1	2.5694 (7)
O1—Fe1	1.9302 (14)	Cl1—Fe1	2.3084 (7)
O2—Fe1	1.9122 (15)		
O2—Fe1—O1	95.51 (6)	N2—Fe1—Cl1	93.21 (5)
O2—Fe1—N2	84.53 (6)	N1—Fe1—Cl1	164.39 (5)
O1—Fe1—N2	171.85 (6)	O2—Fe1—S1	161.90 (5)
O2—Fe1—N1	90.66 (7)	O1—Fe1—S1	101.35 (5)
O1—Fe1—N1	83.61 (6)	N2—Fe1—S1	77.84 (4)
N2—Fe1—N1	88.24 (6)	N1—Fe1—S1	84.78 (5)
O2—Fe1—Cl1	104.95 (6)	Cl1—Fe1—S1	80.35 (3)
O1—Fe1—Cl1	94.65 (5)		

H atoms bonded to C atoms were refined using a riding model (C—H = 0.93 Å) and H-atom displacement parameters were restricted to be 1.2 U_{eq} of the parent atom.

Data collection: *CAD-4 Diffractometer Control Software* (Enraf-Nonius, 1993); cell refinement: *CAD-4 Diffractometer Control Software*; data reduction: *X-RED* (Stoe & Cie, 1991); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1428). Services for accessing these data are described at the back of the journal.

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