

Crystal Structure of $[N,N'$ -Bis(5-chlorosalicylidene)-1,3-diaminopropane]iron(II)

Yalçın ELERMAN, Mehmet KABAK, and Ayhan ELMALI[†]

Department of Engineering Physics, Faculty of Engineering, Ankara University, 06100 Besevler, Ankara, Turkey

The crystal structure of the title compound has been determined. The coordination geometry about the iron(II) center is a tetrahedrally distorted square plane formed by the four-coordinate N_2O_2 donor set of the Schiff-base imine-phenol ligand. Molecules of the title compound are not planar. The two Schiff-base moieties, which themselves are reasonably planar, are inclined at an angle of $31.5(1)^\circ$.

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The complexes of transition metal ions with Schiff bases provide a large class of compounds of both stereochemical and magnetochemical interest due to their preparative accessibility, diversity and structural variability.¹ Metal derivatives of Schiff bases have been studied extensively, and copper(II) and nickel(II) complexes play a major role in both synthetic and structural research. Recently, we studied the crystal structures of $[N,N'$ -bis(5-bromosalicylidene)-1,3-diaminopropane]copper(II), $[N,N'$ -bis(5-bromosalicylidene)-1,3-diaminopropane]nickel(II) and $[N,N'$ -bis(5-chlorosalicylidene)-1,3-diaminopropane]copper(II).^{2,3} We report here the results of the reaction of iron(II) with the tetradentate ligand N,N' -bis(5-chloro-2-hydroxybenzylidene)-1,3-propanediamine, to form a monomeric

Schiff base complex of iron(II). The investigated compound (Fig. 1) was synthesized according to a well-established method.^{2,3}

The structure consists of monomeric iron(II) complexes (Fig. 2). The bond lengths and angles around the iron atom are in good agreement with the values found in other similar tetraordinated iron complexes.¹ The Fe atom is coordinated by two imine N atoms and two phenol O atoms from the imine-phenol ligand in a distorted square-planar coordination geometry. The dihedral angle between N1-Fe-N2 and the O1-Fe-O2 planes is $36.5(2)^\circ$. Each of the two halves of the Schiff base ligand of the title compound is reasonably planar, with the maximum deviation from the plane defined by atoms of each half of Schiff base ligand being $-0.314(4)\text{Å}$ for the C4 atom. However, the entire ligand is not planar, as the two halves are twisted with respect to one another. The least-squares planes through each half of the molecule are inclined at an angle is $36.3(1)^\circ$.

All H atoms bonded to C atoms were refined using a riding model. H-atom displacement parameters were restricted to be $1.5U_{eq}$ of the parent propanediamine C atoms and $1.2U_{eq}$ of the

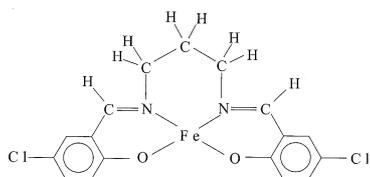


Fig. 1 Chemical structure.

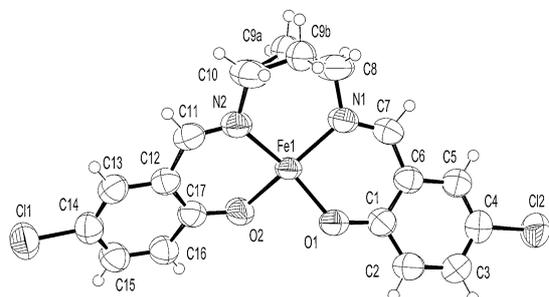


Fig. 2 The molecular structure of the title compound, showing the atom labeling scheme and 50% probability level displacement ellipsoids.

Table 1 Crystal and experimental data

Formula: $C_{17}H_{14}Cl_2FeN_2O_2$	
Formula weight = 405.05	
Crystal system: triclinic	
Space group: $P\bar{1}$	$Z = 2$
$a = 8.1492(8)\text{Å}$	$\alpha = 86.15(1)^\circ$
$b = 9.4290(14)\text{Å}$	$\beta = 68.82(1)^\circ$
$c = 11.8201(18)\text{Å}$	$\gamma = 89.99(1)^\circ$
$V = 844.7(2)\text{Å}^3$	
$D_x = 1.593\text{ g/cm}^3$	
$R = 0.030$	$wR = 0.087$
$(\Delta\rho)_{\max} = 0.005$	
$(\Delta\rho)_{\max} = 0.389\text{ eÅ}^{-3}$	
$(\Delta\rho)_{\min} = -0.217\text{ eÅ}^{-3}$	
No. of reflections used = 3167	
Measurements: Enraf-Nonius CAD-4 diffractometer	
Program system: CAD-4 EXPRESS Software	
Structure determination: SHELXS86	
Refinement: full-matrix least-squares F^2 (SHELXL97)	

[†] To whom correspondence should be addressed.
 E-mail: elmali@eng.ankara.edu.tr

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	x	y	z	$B_{eq}/\text{\AA}^2$
Fe1	4806(1)	2499(1)	5000(1)	4.12(1)
C11	4760(2)	8140(1)	370(1)	7.38(1)
C12	134(2)	-3142(1)	9628(1)	7.39(1)
O1	2503(3)	2001(3)	6202(3)	5.61(1)
O2	3709(4)	3002(3)	3792(3)	5.60(1)
N1	5977(4)	1173(4)	5847(3)	5.25(1)
N2	6822(4)	3825(4)	4154(3)	5.23(1)
C1	2041(5)	904(4)	7041(4)	5.04(1)
C2	255(5)	652(5)	7788(4)	5.70(1)
C3	-318(6)	-556(5)	8593(4)	5.97(1)
C4	887(6)	-1543(5)	8690(4)	5.86(1)
C5	2650(5)	-1292(4)	8076(4)	5.38(1)
C6	3266(5)	-59(4)	7245(4)	5.06(1)
C7	5154(5)	189(4)	6685(4)	5.22(1)
C8	7887(6)	1245(6)	5527(6)	7.79(2)
C9A	8931(11)	2236(9)	4607(8)	5.44(2)
C9B	8555(12)	2720(9)	5392(8)	5.37(3)
C10	8414(7)	3743(6)	4472(6)	7.50(2)
C11	6837(5)	4807(4)	3321(4)	5.24(1)
C12	5507(5)	5063(4)	2760(4)	4.98(1)
C13	5726(5)	6287(4)	1924(4)	5.40(1)
C14	4585(6)	6538(5)	1307(4)	5.69(1)
C15	3269(6)	5557(5)	1410(4)	5.96(1)
C16	3032(6)	4356(5)	2212(4)	5.56(1)
C17	4081(5)	4101(4)	2970(4)	4.96(1)

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* (\mathbf{a}_i \cdot \mathbf{a}_j).$$

Table 3 Bond distances (\AA) and angles ($^\circ$)

Fe1 – O1	1.935(3)	O1 – Fe1 – O2	89.6(1)
Fe1 – O2	1.973(3)	O1 – Fe1 – N2	153.4(1)
Fe1 – N1	2.000(3)	O2 – Fe1 – N2	91.8(1)
Fe1 – N2	1.977(3)	O1 – Fe1 – N1	91.8(1)
		O2 – Fe1 – N1	153.7(1)
		N2 – Fe1 – N1	98.4(1)

parent chlorosalicylidene C atoms. Table 1 shows the crystal and experimental data, while final atomic parameters are given in Table 2. The selected bond distances and angles are shown in Table 3. The C9 atom is disordered (occupancy factors are 0.52 and 0.48) and geometrical restraints were applied to the C8–C9 and C9–C10 distances to prevent anomalous bond distances in the propanediamine chelate ring. A similar disorder was observed in the similar structures of [N,N'-bis(5-bromosalicylidene)-1,3-diaminopropane]copper(II), [N,N'-bis(5-bromosalicylidene)-1,3-diaminopropane]nickel(II) and [N,N'-bis(5-chlorosalicylidene)-1,3-diaminopropane]copper(II).^{2,3}

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