

## Crystal Structure of {Bis[*N*-(2-hydroxyethylamino)ethylsalicylaldiminato]-iron(III)} Chloride

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Ligands such as diethylenetriamine, triethylenetetramine, tetraethylenepentaamine and *N*-2-hydroxyethyl-ethylenediamine give condensation reactions with salicylaldehyde and form polydentate ligands with a linear structure. These polydentate ligands are suitable for several types of coordination geometry.<sup>1,2</sup> The complexes formed by these ligands and

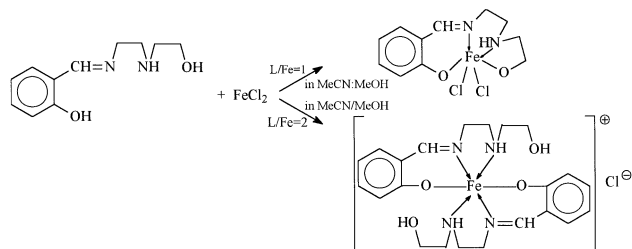


Fig. 1 Synthesis and chemical structure.

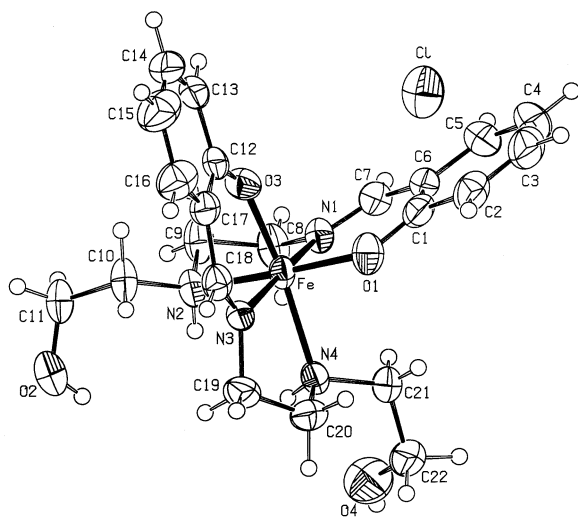


Fig. 2 ORTEP drawing of the title compound with atom labeling.

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transition metals have been reported in the literature concerned with coordination compounds for the last forty years.<sup>2-5</sup>

The ligand was obtained by the reaction of salicylaldehyde and *N*-(2-hydroxyethyl)ethylenediamine in ethanolic media at a mole ratio of 1:1. The complex was prepared as follows: 0.415 g (0.002 mol) of the ligand *N*-(2-hydroxyethylamino)-ethylsalicylaldimine was dissolved in 80 mL of hot acetonitrile. Then, a solution of 0.200 g (0.001 mol) FeCl<sub>2</sub>·4H<sub>2</sub>O in 20 mL of hot methanol was added to it. Finally, a solution of 0.5 mL piperidine was added to this mixture. The final mixture was kept for approximately three days on the bench and the precipitated crystals were filtered out.

In this study, the structure of the complex formed with *N*-(2-hydroxyethylamino)ethylsalicylaldimine and Fe(II) was investigated. There were two complexes obtained with different stoichiometry depending upon the Fe(II)/ligand ratio employed. If the Fe(II) ligand ratio had 1/1, a new complex was formed and published.<sup>6</sup> On the other hand, for a Fe(II)/ligand ratio of 1/2, a new complex was obtained; the molecular structure is shown in Fig. 1.

As can be seen from Fig. 2, the coordination of iron(III) is a distorted octahedral. The two phenolic oxygens, which participate in the coordination, form two neighboring corners of an octahedral. The coordination sphere is depicted in a very simple manner in Fig. 3.

The crystal and experimental data are given in Table 1 and the final atomic parameters are presented in Table 2. Selected bond distances and angles are given in Table 3 and the hydrogen bond geometry is given in Table 4. The coordination in the

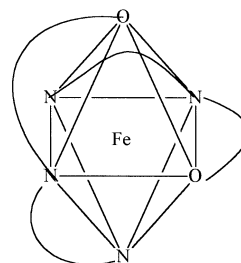


Fig. 3 Coordination sphere of the Fe complex.

Table 1 Crystal and experimental data

|   |
|---|
| Formula: C <sub>22</sub> H <sub>30</sub> N <sub>4</sub> O <sub>4</sub> FeCl |
| Formula weight= 505.809   |
| Space group: P2 <sub>1</sub> /n   |
| Crystal system: monoclinic      Z = 4                                       |
| a = 9.8497(12) Å  |
| b = 24.9281(15) Å   |
| c = 10.4705(13) Å   |
| β = 115.735(2)°   |
| V = 2315.9(6) Å <sup>3</sup>  |
| D <sub>x</sub> = 1.451 g/cm <sup>3</sup>                                    |
| μ (Mo Kα) = 0.8 mm <sup>-1</sup>  |
| T = 295 K   |
| Color = brown   |
| Radiation Mo Kα (λ = 0.71073 Å)   |
| 0.25 × 0.20 × 0.15  |
| 2θ <sub>max</sub> = 50.8°   |
| No. of reflection = 4070  |
| No. of reflection used = 2925 (I > 1σ(I))                                   |
| No. of parameters = 289   |
| R = 0.045   |
| Rw = 0.049  |
| Goodness-of-fit = 0.91  |
| (Δ/σ) <sub>max</sub> final cycle: 0.0002                                    |
| (Δρ) <sub>max</sub> = 0.46 eÅ <sup>-3</sup>                                 |
| (Δρ) <sub>min</sub> = -0.12 eÅ <sup>-3</sup>                                |
| Measurement: Enraf-Nonius CAD-4   |
| Program system: Enraf-Nonius MolEN  |
| Structure determination: Sir88-MolEN  |
| Refinement: full matrix least-square (MolEN)                                |
| Treatment of hydrogen atoms: geometric calculation                          |

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms

| Atom | x          | y          | z          | B <sub>eq</sub> |
|------|------------|------------|------------|-----------------|
| Fe   | 0.25450(6) | 0.36103(2) | 0.36895(6) | 2.73(1)         |
| Cl   | -0.0020(2) | 0.48876(6) | -0.1949(2) | 5.75(3)         |
| O1   | 0.4181(3)  | 0.3241(1)  | 0.3609(3)  | 3.86(7)         |
| O2   | -0.2418(3) | 0.4482(2)  | 0.2582(4)  | 5.7(1)          |
| O3   | 0.2419(3)  | 0.3140(1)  | 0.5062(3)  | 3.78(7)         |
| O4   | 0.3041(4)  | 0.5180(2)  | 0.1081(4)  | 6.5(1)          |
| N1   | 0.3964(3)  | 0.4103(1)  | 0.5185(3)  | 3.06(8)         |
| N2   | 0.0942(3)  | 0.4102(1)  | 0.3967(3)  | 3.15(8)         |
| N3   | 0.1023(3)  | 0.3175(1)  | 0.2124(3)  | 2.72(7)         |
| N4   | 0.2377(3)  | 0.4095(1)  | 0.1936(3)  | 3.08(8)         |
| C1   | 0.5604(4)  | 0.3232(2)  | 0.4570(4)  | 3.54(9)         |
| C2   | 0.6553(5)  | 0.2830(2)  | 0.4453(5)  | 4.6(1)          |
| C3   | 0.8032(5)  | 0.2797(2)  | 0.5394(5)  | 6.1(1)          |
| C4   | 0.8658(5)  | 0.3165(2)  | 0.6485(5)  | 5.9(1)          |
| C5   | 0.7769(5)  | 0.3563(2)  | 0.6632(5)  | 5.0(1)          |
| C6   | 0.6234(4)  | 0.3602(2)  | 0.5694(4)  | 3.57(9)         |
| C7   | 0.5392(4)  | 0.4031(2)  | 0.5927(4)  | 3.4(1)          |
| C8   | 0.3223(4)  | 0.4550(2)  | 0.5540(5)  | 4.1(1)          |
| C9   | 0.1733(4)  | 0.4338(2)  | 0.5399(5)  | 4.2(1)          |
| C10  | -0.0459(4) | 0.3818(2)  | 0.3789(5)  | 3.8(1)          |
| C11  | -0.1690(4) | 0.4176(2)  | 0.3801(5)  | 3.9(1)          |
| C12  | 0.1810(4)  | 0.2657(2)  | 0.4886(4)  | 3.32(9)         |
| C13  | 0.1940(4)  | 0.2365(2)  | 0.6086(4)  | 4.3(1)          |
| C14  | 0.1371(5)  | 0.1864(2)  | 0.5980(5)  | 5.0(1)          |
| C15  | 0.0610(6)  | 0.1619(2)  | 0.4671(5)  | 5.6(1)          |
| C16  | 0.0411(5)  | 0.1900(2)  | 0.3476(5)  | 4.7(1)          |
| C17  | 0.0988(4)  | 0.2416(2)  | 0.3539(4)  | 3.31(9)         |
| C18  | 0.0627(4)  | 0.2695(2)  | 0.2235(4)  | 3.16(9)         |
| C19  | 0.0426(5)  | 0.3435(2)  | 0.0737(4)  | 3.8(1)          |
| C20  | 0.1675(5)  | 0.3755(2)  | 0.0646(4)  | 4.1(1)          |
| C21  | 0.3855(4)  | 0.4310(2)  | 0.2079(4)  | 4.2(1)          |
| C22  | 0.3743(5)  | 0.4711(2)  | 0.0961(5)  | 4.7(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 Selected bond distances (Å) and angles (°)

|          |          |
|----------|----------|
| Fe-O1    | 1.889(3) |
| Fe-O3    | 1.900(3) |
| Fe-N1    | 2.004(3) |
| Fe-N2    | 2.117(4) |
| Fe-N3    | 1.994(3) |
| Fe-N4    | 2.143(4) |
| O1-Fe-O3 | 94.0(1)  |
| O1-Fe-N1 | 90.8(1)  |
| O1-Fe-N2 | 171.9(1) |
| O1-Fe-N3 | 92.8(1)  |
| O1-Fe-N4 | 89.2(1)  |
| O3-Fe-N1 | 92.3(1)  |
| O3-Fe-N2 | 87.5(1)  |
| O3-Fe-N3 | 90.8(1)  |
| O3-Fe-N4 | 171.7(1) |
| N1-Fe-N2 | 81.1(1)  |
| N1-Fe-N3 | 175.0(1) |
| N1-Fe-N4 | 95.3(1)  |
| N2-Fe-N3 | 95.1(1)  |
| N2-Fe-N4 | 90.4(1)  |
| N3-Fe-N4 | 81.5(1)  |

Table 4 Hydrogen-bonding geometry (Å)

|                |          |
|----------------|----------|
| O2...H2.....Cl | 3.172(4) |
| N2...H2A...Cl  | 3.180(3) |
| N4...H4A...Cl  | 3.464(3) |
| C7...H7.....O4 | 3.448(5) |
| C10...H101..O3 | 3.064(5) |
| C10...H102..N3 | 3.158(5) |
| C20...H202..Cl | 3.769(5) |
| C21...H211..O1 | 3.052(5) |

N4O2 system is spherical. The complex is formed by the coordination of two ligands in a medial manner; the two oxygens are not in apical position, but side by side. The iminic nitrogen and the phenolic oxygens are closer to iron (1.889 – 2.004 Å) than aminic nitrogens (2.117 – 2.143 Å). The hydroxyl groups bonded to amine groups are in a free position, and do not take place in the coordination. They form hydrogen bonds with the ionic chlorides, which explains why amine nitrogens are further away from iron.

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