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Instrumental Achievement

Crystal Structure of the *N*-(2-Salicylidene-ethyl)-ethanolaminato-dichloro-iron(III) Complex

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The structre of monomeric Fe complexes in the presence of a tetradentate ONNO Schiff-base ligand has been reported.[1-3] In those complexes the Fe ion lies at the base of a distorted pyramid coordination; the Cl^- ion completes the pyramid. In the title compound with an ONNO-type Schiff-base ligand the coordination of the Fe ion is a distorted octahedral. The Fe ion is at a distance of 0.1513(14)Å from the equatorial plane (N1, O2, Cl1, Cl2) and the apical positions are occupied by O1 and N2. The bond lengths with the donor atoms of the Schiff-base ligand Fe–O [1.912(6)–2.122(5)Å] and Fe–N [2.099(8)–2.197(9)Å], respectively, are shorter than the bond lengths with Fe–Cl [2.287(3)–2.317(2)Å]. The bond angles around the Fe are in the range of 74.9(2) to 97.4(2)°. The chelating moiety (O1, C1, C6, C7, N1) is planar with the Fe ion at a distance of 0.365(1)Å. The five-membered chelating moieties are in an envelope-like shape. The *para*-positioned atom (C9 and C11) are at a distance of 0.571(9) and 0.337(15)Å from the best planes passing through (Fe, N1, N2, C8) and (Fe, O2, N2, C10), respectively. The bond lengths are in agreement with the critical literature.

Table 1 shows the crystal and experimental data, while the final atomic parameters are given in Table 2. The bond distances and angles are given in Table 3. The intermolecular hydrogen-bond geometry is given in Table 4. The yellow-crystalline ligand *N*-(2-salicylidene-ethyl)ethanolamine is a reaction product of *N*-(2-aminoethyl)ethanolamine and salicylaldehyde, taken in equal molar ratio at room temperature in a (1:2) solution of ethanol:diethylether. This ligand was filtered after one day, and its 0.208 g (1 mmol) was dissolved in 50 ml of hot MeCN. To this solution 0.199 g (1 mmol) of $FeCl_3 \cdot 4H_2O$ in hot 20 ml of MeOH was

Table 1 Crystal and experimental data

Formula:	$C_{11}H_{15}Cl_2FeN_2O_2$
Formula weight=334.01	
Crystal system: monoclinic	
Space group: *C*2/*c*	Z=8
a=21.615(2)Å	
b=10.193(1)Å	
c=15.063(2)Å	
β=123.968(3)°	
V=2752.5(5)Å^3	
D_x=1.612 g/cm^3	
μ(Mo K$_\alpha$)=1.481 mm^{-1}	
T=295 K	
Dark red	
F(0 0 0)=1368	
Crystal size: 0.4×0.3×0.15 mm	
Radiation=Mo K$_\alpha$	
R=0.058	
R_w=0.063	
No. of reflections used=1290	
No. of parameters=161	
Goodness-of-fit=0.92	
Measurements: Enraf Nonius CAD-4 diffractometer	
Program system: CAD-4 EXPRESS software	
Structure determination: MolEN	
Treatment of hydrogen atoms: geometric calculation except the hydrogens of hydroxy groups	
Refinement: full matrix least-squares (MolEN)	

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

Atom	*x*	*y*	*z*	B_{eq}/Å^2
Fe	0.36113(5)	0.0528(1)	0.09220(8)	2.69(2)
Cl1	0.3864(1)	0.2603(3)	0.0632(2)	5.04(7)
Cl2	0.2691(1)	0.0110(3)	-0.0858(2)	4.68(6)
O1	0.4453(2)	-0.0322(6)	0.1070(4)	3.1(1)
O2	0.4272(3)	0.0929(7)	0.2588(4)	4.0(2)
N1	0.3379(3)	-0.1271(8)	0.1355(5)	3.3(2)
N2	0.2815(3)	0.1135(9)	0.1313(5)	3.6(2)
C1	0.4591(4)	-0.159(1)	0.1039(6)	3.1(2)
C2	0.5168(5)	-0.192(1)	0.0895(6)	4.3(3)
C3	0.5351(5)	-0.323(1)	0.0924(7)	5.5(3)
C4	0.4992(6)	-0.423(1)	0.1066(8)	5.8(3)
C5	0.4430(5)	-0.392(1)	0.1191(7)	4.5(3)
C6	0.4229(4)	-0.262(1)	0.1191(6)	3.5(2)
C7	0.3648(5)	-0.238(1)	0.1375(6)	3.8(2)
C8	0.2828(4)	-0.119(1)	0.1660(6)	4.8(2)
C9	0.2357(4)	-0.001(1)	0.1171(7)	5.4(3)
C10	0.3185(5)	0.179(1)	0.2352(7)	6.4(3)
C11	0.3977(6)	0.183(2)	0.2952(9)	9.2(4)

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

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Table 3 Bond distances (Å) and angles (°)

Fe - Cl1	2.287(3)	N2 - C10	1.46(1)
Fe - Cl2	2.317(2)	C1 - C2	1.42(2)
Fe - O1	1.912(6)	C1 - C6	1.40(1)
Fe - O2	2.122(5)	C2 - C3	1.38(2)
Fe - N1	2.099(8)	C3 - C4	1.37(2)
Fe - N2	2.197(9)	C4 - C5	1.36(2)
O1 - C1	1.33(1)	C5 - C6	1.40(2)
O2 - C11	1.39(2)	C6 - C7	1.45(2)
N1 - C7	1.26(1)	C8 - C9	1.48(1)
N1 - C8	1.50(1)	C10 - C11	1.42(1)
N2 - C9	1.46(1)		
Cl1 - Fe - Cl2	95.2(1)	C7 - N1 - C8	118.6(9)
Cl1 - Fe - O1	97.4(2)	Fe - N2 - C9	107.7(7)
Cl1 - Fe - O2	88.4(2)	Fe - N2 - C10	112.0(5)
Cl1 - Fe - N1	172.5(2)	C9 - N2 - C10	116.1(9)
Cl1 - Fe - N2	95.5(3)	O1 - C1 - C2	118.1(9)
Cl2 - Fe - O1	101.4(2)	O1 - C1 - C6	124.3(9)
Cl2 - Fe - O2	168.4(2)	C2 - C1 - C6	118(1)
Cl2 - Fe - N1	90.0(2)	C1 - C2 - C3	119(1)
Cl2 - Fe - N2	93.8(2)	C2 - C3 - C4	123(1)
O1 - Fe - O2	89.0(2)	C3 - C4 - C5	118(1)
O1 - Fe - N1	86.9(3)	C4 - C5 - C6	121(1)
O1 - Fe - N2	159.0(3)	C1 - C6 - C5	121(1)
O2 - Fe - N1	85.4(3)	C1 - C6 - C7	121.8(9)
O2 - Fe - N2	74.9(2)	C5 - C6 - C7	118(1)
N1 - Fe - N2	78.7(3)	N1 - C7 - C6	126(1)
Fe - O1 - C1	130.8(6)	N1 - C8 - C9	109.8(9)
Fe - O2 - C11	116.1(5)	N2 - C9 - C8	110.6(7)
Fe - N1 - C7	126.9(8)	N2 - C10 - C11	116(1)
Fe - N1 - C8	114.4(6)	O2 - C11 - C10	113(1)

mixed. The dark-red crystals were taken after two days in air. During this period Fe(II) ions oxidize into Fe(III) ions. The chemical reaction of the title compound is shown in Fig. 1.

Table 4 Hydrogen-bonding geometry (Å,°)

D—H...A	H....A	D—A	D—H...A
O2—H2'...O1^{i}	1.751	2.661(8)	167.9
N2—H2A...Cl1^{ii}	2.563	3.395(8)	146.4

Symmetry code: (i)–*x*, *y*, 1/2–*z*; (ii) 1/2–*x*, 3/2–*y*, 1–*z*.

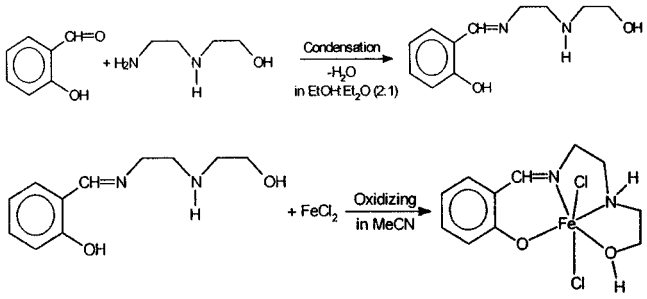

Fig. 1 Synthesis and chemical structure.

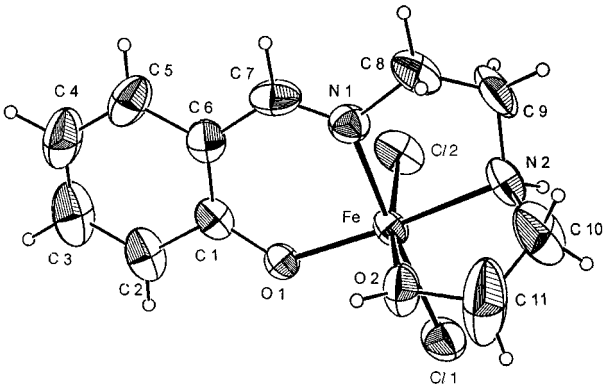

Fig. 2 ORTEP drawing of the title compound with atom labeling.

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