

Crystal Structure and Spectroscopic Properties of [Fe(HL)₂(NCS)₂] (HL = 2-methoxy-6- ((phenylimino)methyl)phenol)

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Abstract—In the crystal structure of the title compound, [Fe(NCS)₂(HL)₂] (HL is 2-methoxy-6-((phenylimino)methyl)phenol), the six-coordinated Fe(II) cation is bonded with two O-phenolate, two O-ether and two N-thiocyanate anions. In the complex, the ligand molecule acts as zwitterion. The complex **1** is characterized by elemental analysis, conductance measurement, room temperature magnetic susceptibility measurement and IR spectroscopic study. The complex **1** crystallizes in the monoclinic sp. gr. P2₁/n, Z = 4. In the crystal, the Fe^{II} cation is in a N₂O₄ core and is situated in a distorted octahedral environment. Furthermore, intermolecular hydrogen bonds link the molecules developing a 2D network parallel to ab plan.

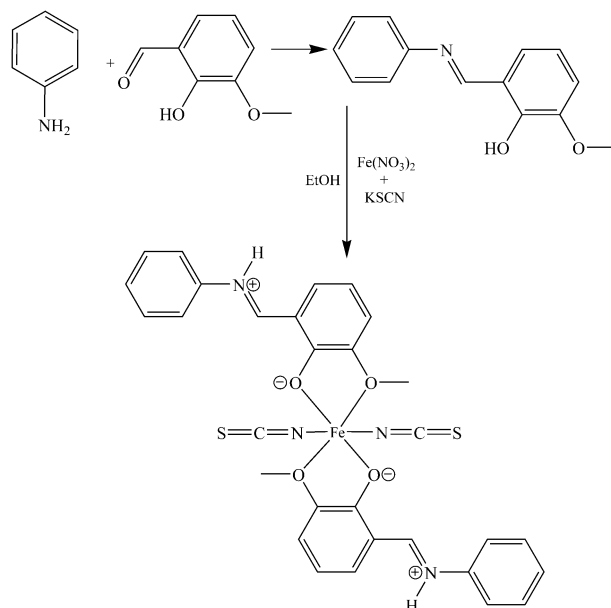
Keywords: o-vanillin, aniline, thiocyanate, X-ray.

Date of Submission: 01-03-2021

Date of Acceptance: 14-03-2021

I. Introduction

O-vanillin is very generous precursor in the preparation of Schiff bases used as ligands in coordination chemistry [1-4]. By appropriately choosing the type of amine with which the o-vanillin is condensed, ligands with peculiar structure can be synthesized [5,6]. Indeed, ligand with soft or hard donor atoms or with both or chiral ligands can be prepared easily [7-9]. The stereochemistry of the complexation reaction can be influenced by bulky substituents on the amine precursor [10,11]. It is possible to control the size of the cages with more or less flexible arms and the number of coordinating sites according to the type of metal. The methoxy group of o-vanillin makes the derived ligands versatile. In fact, methoxy can play an essential role in complexation. It can coordinate with metal or remains free [3,12], thus making it possible to receive another metal atom later. In this way, mononuclear (3d or 4f) [12,13], homonuclear (3d-3d or 4f-4f) [4,12,14-16] or heteronuclear (3d-4f) [17,18] complexes, with original structures, have been prepared. Several complexes obtained from o-vanillin with diverse properties such as magnetism [17,19,20], luminescence [18,21], catalytical [3,22], or optical [23,24] have been reported in recent years. Schiff bases based on o-vanillin with biological activities were obtained. Upon coordination the activities can be enhanced or reduced according to the metal and the coordination mode [15, 25-27]. Herein, we report an iron(II) complex obtained from a Schiff base based on o-vanillin (Scheme 1). The complex is characterized by spectroscopic study and X-ray diffraction technic.



Scheme 1. Synthesis procedure of the ligands.

II. Experimental

2.1. Materials and Measurements

Commercially available o-vanillin, aniline and iron nitrate were purchased from Aldrich and used without further purification. The IR spectrum was recorded on a Perkin Elmer Spectrum Two spectrophotometer ($4000\text{--}400\text{ cm}^{-1}$). The molar conductance of 10^{-3} M solution of the metal complex in DMF was measured at $25\text{ }^{\circ}\text{C}$ using a WTW LF-330 conductivity meter with a WTW conductivity cell. Room temperature magnetic susceptibility of the powdered sample was measured using a Johnson Matthey scientific magnetic susceptibility balance (Calibrant: $Hg[Co(SCN)_4]$).

2.2. Synthesis of the complex

To a solution of KSCN (0.194 g; 2 mmol) in 20 mL of ethanol was added solid $Fe(NO_3)_3 \cdot 9H_2O$ (0.3412 g, 0.1 mmol) under stirring. After 10 minutes, the solution was filtered and the solid was discarded. In another flask the ligand HL (2-methoxy-6-((phenylimino)methyl)phenol) was prepared by mixing o-vanillin (0.3043 g, 2 mmol) and aniline (0.1803 g; 2 mmol). The mixture was stirred under reflux for two hours. On cooling, the prepared solution of iron thiocyanate was added. Immediately the solution turned pinkish red. The mixture was refluxed for two hours under stirring and was filtered while hot. The filtrate was kept at 298 K . After one-week, wine-red crystalline compound was collected by filtration and washed thoroughly with chilled diethyl ether. Subsequently it was dried in a vacuum desiccator under $P_{4O_{10}}$. M.P. 128° C . Yield 48 %. Analysis calculated for $[C_{30}H_{26}FeN_4O_4S_2]$. C, 57.51; H, 4.18; N, 8.94; S, 10.24. Found: C, 57.54; H, 4.19; N, 8.95; S, 10.21 %. $\mu_{\text{eff}} (\mu_B)$: 5.27. $\Lambda_M (\text{S cm}^2 \text{ mol}^{-1})$: 11. IR (cm^{-1}): 3110, 2067, 1621, 1483, 1416, 1289, 1148.

2.3. X-ray data collection, structure determination and refinement

Details of the X- rays crystal structure solution and refinement are given in Table 1. The structures were solved by direct methods which revealed the position of all non- hydrogen atoms [28]. All the structures were refined on F^2 by a full- matrix least- squares procedure using anisotropic displacement parameters for all non- hydrogen atoms [29]. All hydrogen atoms were added in calculated positions and refined in riding mode on the parent atom [30].

Table-1. Crystal data and details of the structure determination for the complex $C_{30}H_{26}FeN_4O_4S_2$.

Chemical Formula	$C_{30}H_{26}FeN_4O_4S_2$
M (g/mol)	626.53
Temperature (K)	100(2)
Crystal system	Monoclinic
Space group	$P2_1/n$

a (Å)	9.7563(4)
b (Å)	16.5371(8)
c (Å)	18.5815(11)
β (°)	103.619(5)
V (Å ³)	2913.7(3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm ⁻¹)	5.86
Crystal size (mm)	0.03 × 0.02 × 0.01
D_x (g cm ⁻³)	1.428
T_{min} , T_{max}	0.684, 1.000
No. of measured reflections	25573
Independent reflections	5468
Observed reflections $I > 2\sigma(I)$	3403
R_{int}	0.079
Number of refined parameters	373
R_1 , wR_2 ($I > 2\sigma(I)$)	0.062, 0.172
R_1 , wR_2 (all data)	0.096, 0.197
Data/parameters/restraints	5468/373/0
GOF	1.03
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å ⁻³)	0.64, -0.49
h , k , l ranges	$-11 \leq h \leq 11$, $-19 \leq k \leq 19$, $-22 \leq l \leq 22$

III. Results and discussion

3.1. Preparation and characterization of the title complex

The ligand HL ((E)-2-methoxy-6-((phenylimino)methyl)phenol) reacts with iron(II) thiocyanate leading to $[Fe(HL)_2(NCS)_2]$ (**1**). During the formation of **1**, the neutral organic ligand, HL acts as a zwitterion. A quaternary iminium cationic group is formed by the migration of the phenolate proton resulting in the formation of negatively charged oxygen atom (Scheme 1). It seems that such type of dissociation does not occur when the ligand is not coordinating a metal ion. The title compound **1** in which the Fe(II) is coordinated by two thiocyanate groups through the negatively charged oxygen atom is neutral molecular complex. The analytical data are in accordance with the formulation of the complex: $[C_{30}H_{26}FeN_4O_4S_2]$. The infrared spectrum of the complex shows a medium absorption bands at 3110 cm^{-1} which is characteristic of the N—H stretching vibration. The band at 1621 cm^{-1} is attributed to the elongation of the C=N function. Additional bands, characteristic of the aromatic ring, are pointed in the range $[1580\text{--}1483\text{ cm}^{-1}]$. The intense and sharp band observed at 2067 cm^{-1} is indicative of the presence of a thiocyanate group. The molar conductivity is measured for a freshly prepared solution of complex in DMF. The value is $11\ \Omega^{-1}\cdot\text{cm}^2\cdot\text{mol}^{-1}$ for the complex, thus indicating neutral electrolyte with good stability of the complex in DMF [31]. The magnetic moment value of the title complex in solid state is $5.27\ \mu_B$. This value is consistent with high-spin state of the Fe(II) ($S = 2$) [32].

3.2. Crystal structure

The complex crystallizes in space group $P2_1/n$ of the monoclinic system. The asymmetric unit of the complex contains two thiocyanate anions, two zwitterionic ligand molecule and one iron(II) cation. The crystal data collection and refinement are recorded in Table 1. Drawings were made using *ORTEP3* for windows [30]. The molecular structure of the complex labelling of the atoms are displayed in Fig. 1. The selected bond lengths and angles are listed in Table 2. The X-ray diffraction analysis of the crystal formulated as $[Fe(NCS)_2(HL)_2]$, shows a discrete structure in which the iron(II) cation is coordinated to two O—bonded phenolate, two O—bonded ether, and two cyanate anions acting through their nitrogen atoms (Fig. 1). In the structure, the

thiocyanate moieties act as monodentate units while each of the two ligand molecules acts in bidentate fashion through their oxygen atoms. The ligand molecules are in their zwitterionic forms: the proton from the phenol moiety migrates to the azomethine nitrogen atom. The six-coordinated iron(II) cation is situated in a distorted octahedron. The basal plane of the environment around the Fe(II) cation is occupied by O1, O4, O3 and N4 while the apical positions are occupied by O2 and N3 atom. The values of the *cisoid* angles subtended by the atoms defining the basal planes are respectively $71.0(1)^\circ$ (O3—Fe—O4), $89.0(1)^\circ$ (O3—Fe—N4), $91.1(1)^\circ$ (O1—Fe—O4) and $106.4(1)^\circ$ (O1—Fe—N4) while the *transoid* angle values are $158.7(1)^\circ$ (O4—Fe—N4), and $157.6(1)^\circ$ (O3—Fe—O1). The angle value formed by Fe1 and the atoms in apical position is $154.2(1)^\circ$. These values are indicative of severely distorted octahedral environment. The thiocyanate groups are quasi-linear with S—C—N angle values of $176.3(5)^\circ$ and $178.3(4)^\circ$ which are comparable to the angle reported in the complex $[K(2,2,2\text{-crypt})][Fe^{II}(\text{TpivPP})(\text{NCS})]$ (2,2,2,-crypt is the cryptant-222 and TpivPP is $\alpha,\alpha,\alpha,\alpha$ -tetrakis(o-pivalamidophenyl) porphyrin) [33]. The anionic N-donor thiocyanate bind nonlinearly the iron(II) with angles Fe-N-CS respectively of $140.4(4)^\circ$ and $166.0(4)^\circ$. The Fe—O_{phenolate} distances (2.118(3) Å and 2.122 (3) Å) are shorter than the distances Fe—O_{ether} (2.375 (3) Å and 2.367 (3) Å) owing to the hard character of the phenolate oxygen atom. These values are longer than those reported for iron complexes formed with the ligands bis(O-vanillin)-O-phenylenediimine and bis(O-vanillin)-2,3-naphthalenediimine [34]. The stability of the crystalline structure is ensured by the existence of intramolecular hydrogen bonds type of N—H...O1 and C—H...S and intermolecular hydrogen bond type of C—H...S (Table 3, Fig. 2, Fig. 3).

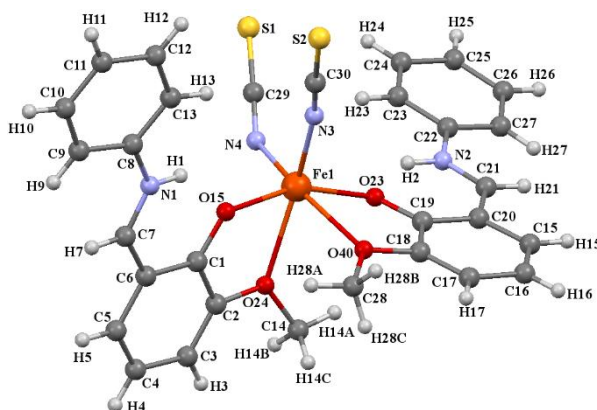


Fig. 1. Crystal structure of the title complex showing 30% probability ellipsoids and H atoms are shown as small sphere.

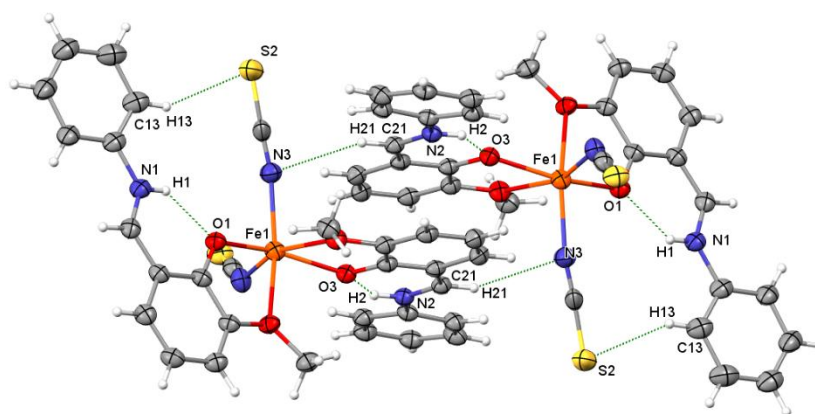


Fig. 2. Inter and intra molecular hydrogen bonds in the structure of the complex.

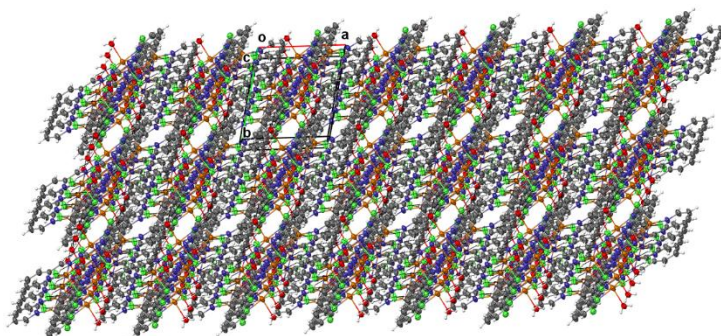


Fig. 3. Perspective view of the network constructed by hydrogen bond interactions along the ab plane.

Table-2. Selected bond distances (\AA) and angles (deg)

Fe1—O3	2.119(3)	Fe1—N3	2.103(5)
Fe1—O1	2.123(3)	S1—C29	1.613(5)
Fe1—O4	2.367(3)	S2—C30	1.636(6)
Fe1—O2	2.375(3)	N4—C29	1.172(6)
Fe1—N4	2.140(4)	N3—C30	1.142(6)
O3—Fe1—O1	157.64(10)	N4—Fe1—O4	158.73(13)
O3—Fe1—O4	71.00(9)	N4—Fe1—O2	98.64(13)
O3—Fe1—O2	91.16(10)	N3—Fe1—O3	105.50(13)
O3—Fe1—N4	89.03(12)	N3—Fe1—O1	87.69(13)
O1—Fe1—O4	91.06(10)	N3—Fe1—O4	91.45(14)
O1—Fe1—O2	70.88(10)	N3—Fe1—O2	154.20(13)
O1—Fe1—N4	106.36(13)	N3—Fe1—N4	101.16(17)

Table-3. Hydrogen-bond geometry (\AA , $^\circ$) for the Fe^{II} complex.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots O3	0.86	1.95	2.629 (4)	135.4
N1—H1 \cdots O1	0.86	1.94	2.624 (4)	135.9
C21—H21 \cdots N3 ⁱ	0.93	2.57	3.425 (6)	153.4
C7—H7 \cdots N4 ⁱⁱ	0.93	2.49	3.348 (6)	153.5
C28—H28C \cdots S2 ⁱⁱⁱ	0.96	2.87	3.769 (6)	156.3
C13—H13 \cdots S2	0.93	2.87	3.722 (6)	152.8

Symmetry codes : (i) $-x+1, -y+1, -z+1$; (ii) $-x+1, -y, -z+1$; (iii) $x+1/2, -y+1/2, z+1/2$.

IV. Conclusion

The HL organic ligand reacts with Fe(II) yielding a zwitterionic mononuclear complex **1**, which is characterized by elemental analysis, magnetic moment, molar conductivity, and IR. The structure of the mononuclear iron(II) complex is determined by single-crystal X-ray diffraction. The complex is non-ionic electrolyte in DMF solution. The ligand acts as bidentate ligand through the phenolate oxygen atom and the ethereal oxygen atom. The proton of the phenol function migrates to the azomethine nitrogen atom resulting in a zwitterion. Considering the magnetic moment, infrared spectra, and the electronic spectrum data for Fe(II) complex, octahedral geometry is proposed for the complex **1**. The structure is confirmed by the X-ray diffraction study. The structure of complex **1** is consolidated by numerous intra and intermolecular hydrogen bonds (Fig. 2) which produce a two-dimensional network (Fig. 3).

Supplementary Material:

Crystallographic data have been deposited with the Cambridge Crystallographic Data Centre. CCDC-2011033. Copies of this information may be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>, or by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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